

UDC 538.958

RESEARCH OF GRAPHITE AND ALUMINIUM PARTICLES IN A POLYMER FILM MATRIX

Makhanov K.M., Ermaganbetov K.T., Ismailov Zh.T., Chirkova L.V.,
Amochaeva G.P., Omarova Zh.T., Askerbekova A.A.

Karaganda State University named after E.A. Buketov, Karaganda, Kazakhstan makanov@inbox.ru

The paper presents the results of the development of a method for manufacturing graphite and alumina films in a polymer matrix. The difficulties arising in the formation of films on the surface of glass and aluminium substrates are determined. It is established that plastic is the best material for substrates. The results of measuring the electrical parameters of the film resistance are presented. It was found that with additional heating of the substrates, the films of graphite particles are made more homogeneous. These films are stable to mechanical influences, as they do not break down on contact with the measuring probes.

Keywords: graphite, thin films, polymer matrix, aluminum oxide, resistance, conductivity, nanoparticles

Introduction

Nowadays, carbon films are widely used in engineering and industry [1, 2]. There are various methods for obtaining carbon films: magnetron sputtering of graphite [3-5], atomization of graphite by an ion beam [6, 7], laser ablation of a target [8]. All of them require the creation of special conditions with the use of complex and expensive equipment [9].

The aim of the work is to develop a method for obtaining graphite and alumina films in a polymer matrix and to study their electro-physical parameters.

It was noted in [10, 11] that if a substance is mechanically cut to the smallest size, then in the total mass of the particles obtained, some of them may have dimensions on the order of hundreds or less than nanometers.

It is natural to assume that when obtained powder is dissolved, the rate of settling of the particles and their distribution along the thickness of the solution will depend on the mass and dimensions accordingly. First of all, the heaviest particles will drop to the bottom, the particles of medium size (and mass) will sink to the bottom of the vessel at a lower speed, and the lightest will be in the upper layers of the solvent, and may remain suspended for a longer time. Proceeding from this, we proposed a simple [12] technique for the formation of films of graphite and alumina particles on a solid surface (quartz glass).

An investigation of the absorption spectra of the initial solutions showed [13] that the optical density of the solutions increases with increasing depth of sampling. Using the method described in this paper, we obtained films of graphite and alumina particles on the surface of various substrates. The results of the investigations are presented in [14]. It was found that the films obtained from samples taken at different solution depths differ in the absorption density. The results of a study of the microstructure of films with an electron microscope showed that the particle sizes increase with increasing sampling depth.

The results presented in this paper are the next stage of the experimental work related to the production of films of graphite particles, both on the surface of the polymer film and in the matrix of polymer films (PVA).

1. Methods of forming films on surfaces of various substrates

1.1 The films formation on the surface of glass substrates

The first films in the polymer matrix were formed on the surface of glass substrates. To achieve homogeneity of graphite films, without any chips or lumens, with close packing in a row, we prepared polymeric solutions with different concentrations of graphite particles. The resulting solutions were spread on the surface of the glass substrates. Until complete drying, the film was aged for 10-12 hours. Then, using tweezers carefully removed from the surface of the substrates. Repeated experiments using glass substrates showed that PVA films are fastened to their surfaces rather tightly. And when they tried to remove them, the tapes burst. The result is shown in Figure 1. Particularly strong sticking is observed for films with a high concentration of graphite.



Fig.1. Film obtained from the surface of a glass substrate.

The next part of prepared polymer solutions with graphite particles was partially applied to the aluminium substrate and to the plastic substrate.

1.2 The films formation on the surface of aluminium substrates

Consider the situation with the use of aluminium substrates. The surface of the aluminium substrate was pre-polished, then after chemical cleaning, it was washed under a stream of distilled water. The films were applied as described above. At the end of the time to dry the time, we found that the films from the aluminium surface exfoliate unevenly. That is, if the film is saturated with graphite particles, then it does not exfoliate from the aluminium surface. In this case, the opposite effect is observed for films with a lower concentration of graphite particles. As they dry up, the films themselves exfoliate from the surface of the aluminium substrate.

Thus, experiments using glass and aluminium surfaces as substrates showed that the films strongly stick. As a result the attempt to remove them leads to the destruction of the integrity of these films. In the case of an aluminium substrate, films are destroyed where the concentration of graphite particles are high. In this case, an interesting picture is observed, the graphite powder combines into local groups and forms islands, both on the surface of the polymer film and inside the film. The lower part of these islets adheres to the surface of the aluminium substrate, and as a result, it can no longer be torn off.

1.3 Formation of films on the surface and in the matrix of a polymer film

The next series of experiments was carried out using substrates made of plastic materials. It was found that graphite polymer films, irrespective of the concentration of graphite particles, peel well from the surface. In general, based on the results of the performed work, we found that the graphite films in the polymer matrix most closely meet our requirements. The thickness of the films

does not exceed 200 nanometers, under mechanical influence the film does not collapse and, finally, it is content with the flexible, which allows it to be shaped as desired. The appearance of graphite films in PVA with a matrix is shown in Fig. 2.

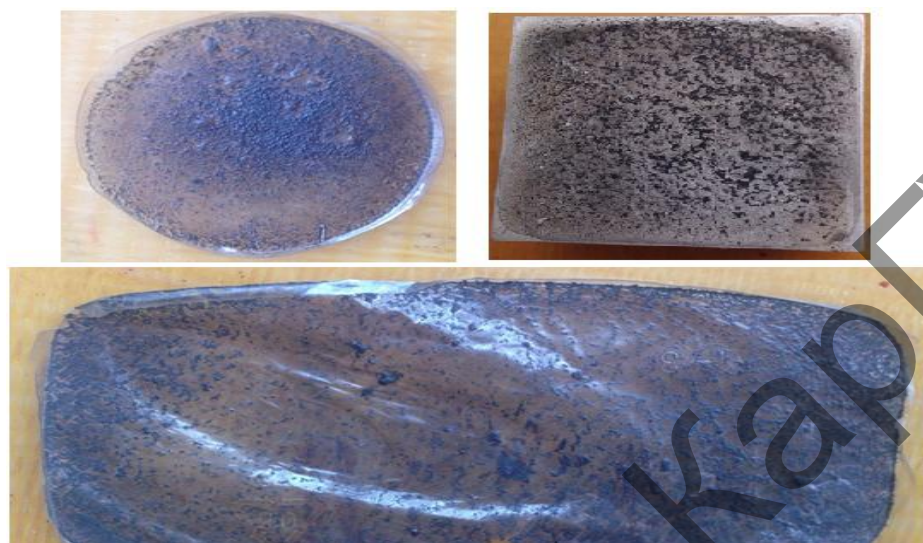


Fig.2. Appearance of graphite films in a polymer matrix prepared by the first method.

The graphite films shown in Figures 2 and 3 were obtained in two ways. Films presented in Fig. 2 were obtained by the method which consisted in the fact that the graphite particles were applied to the polymer film from the outside. Let's consider this method in more detail. The polymer film was pre-poured onto the surface of a plastic material. Then, the mechanically grinded graphite particles were transferred to the surface of the polymer film by blowing out an external air flow. After the entire mass of graphite powder settled on the surface of the polymer film, the top was covered with a glass cap and left to dry. As can be seen from Fig. 2, the graphite particles are distributed unevenly in the film. There is also the formation of local areas of accumulation of particles in islands.

The second method consisted in that the particles of the harvested graphite were mixed in a polymer solution. After thorough stirring, the solution dripped onto a pre-prepared surface. The photo of the films prepared in the second way is shown in Figure 3.



Fig.3. Appearance of a graphite film in a polymer matrix prepared by the second method.

2. Discussion of results

As we see from this figure, the films obtained by the second method turned out to be most uniform. The thickness of the films obtained also does not exceed 200 nanometres.

As in the case of the first films obtained on the surface of glass or quartz substrates, these films in the polymer matrix were also measured for electrical resistance. However, unlike the previous films, while measuring the resistance of these films, we found that there is no electrical connection. Upon close examination of the surface of the films under an optical microscope, it was found that most of the graphite particles are inside the polymer film, and a small part on its surface. As a result, it turned out that the film visually looks uniform with a dense packing of particles, but in reality consists of torn off from each other local areas. An attempt to eliminate this problem by reducing the thickness of the polymer matrix has not been successful. The re-manufactured films, when examined under an optical microscope, were identical to the previous ones. The thickness of the polymer film decreased, but, however this was not enough, the graphite particles had much smaller dimensions and, most importantly, they were stratified. While we assumed that homogeneous particles in the matrix will tend to line up uniform rows and form a denser packing. Thus, it turned out that the particles of graphite form a homogeneous layer, but these layers do not bind to each other. It is possible that additional energy is not enough to further combine the particles.

Based on these considerations, we decided to introduce changes in the procedure for the production of films. It was necessary to create conditions for additional thermal heating of the substrates. It was assumed that some of the heat would be transferred to the particles. With an additional supply of thermal energy, the particles will be able to line up in a row.

The attempt to warm up the aluminium substrate failed. Even with a slight increase in temperature, the film was deformed. The use of open fire resulted in immediate ignition of the films on an aluminium substrate. In this regard, it was decided to use only plastic substrates with high beads. The surface on which the polymer was buried was immersed in a container of hot water. In this case, the part on which the polymer film dripped remained naturally from the upper side. The procedure for manufacturing graphite films in a polymer matrix was as follows. A plastic box with a flat surface is installed in the container. The capacity is filled with hot water to the level of the side edges. Thus, the surface of the plastic box remains above the surface of the water. A prepared polymer solution with graphite particles is added to the open part of the plastic surface. Within two hours from the moment when the polymer film was filled, the plastic box, and therefore the polymer matrix, is maintained at a high temperature ($\sim 60^\circ\text{C}$). Thus, supply of additional, external energy is necessary for aligning graphite particles into homogeneous series. It was expected that we would obtain a homogeneous, continuous layer of graphite particles.

Conclusion

Recently the carbon, particularly, a form of it as graphite, is one of the active area of research. The interest is increased due to the discovery of nanotubes, fullerenes and monolayers of a graphite crystal. It was found that films with a thickness of only a few atomic layers of graphite (graphene), in their properties are a semimetal with a small overlap of the conduction band and the valence band. Also, a significant field effect and ambipolar Hall effect were detected, which allows using an applied external field not only to change the conductivity of the material, but also to change the main type of charge carriers. The results stimulated further investigation of graphite films. Observation of the field effect together with metallic conductivity allowed us to assume that graphite films may be of interest for microelectronics and nanoelectronics.

Modern microelectronics tends to miniaturize. As existing technologies and materials approach the limit of their capabilities, an active research of new materials and principles of operation of devices is conducted. Semiconductor materials used in modern microelectronics have some

fundamental limitations. One of the main ones is the restriction of the concentration and mobility of charge carriers. The use of all-metal transistors, that is, the use of metal as the main material of microelectronics, would undoubtedly have a positive effect on the speed and other characteristics of the devices. However, this idea encounters other obstacles: it is impossible to control the conductivity of "thick" metal films due to the fact that the field is completely screened already at a depth not exceeding a nanometer. Films of this thickness cannot be used for these purposes, as they are extremely unstable.

Our studies of thin-film graphite have shown that the stability of these materials can be achieved by forming in a polymer matrix. Investigations of the electrical resistance parameters of films have shown that, on average, this value for films varies in the range from two hundred to three hundred Ohms.

REFERENCES

- 1 Yastrebov S.G., Ivanov-Omskiy V.I., Richter A. Fotoluminescenttsiia amorfnoogo ugleroda, vyrashchennogo lazernoi abliatciei grafita. *Fizika i Tekhnika Poluprovodnikov*, St. Petersburg «Science», 2003, Vol. 37, Issue 10, pp. 1193 -1196 [in Russian]
- 2 Gadomsky H.E., Altunin K.K., Ushakov N.M., Kosobudsky I.D., Podvigalkin V.Ya., Kulbatsky D.M. Vysokoeffektivnye prosvetliaiushchie nanostrukturnye opticheskie pokrytiia dlia solnechnykh elementov. *Zhurnal Tekhnicheskoi Fiziki*, St. Petersburg «Science», 2010, Vol. 80, Issue. 7, pp. 83 - 89. [in Russian]
- 3 Oskomov K.W., Soloviev A.A., Rabotkin S.V. Tverdye uglerodnye pokrytiia, nanosimye metodom impulsnogo silnotochnogo magnetronnogo raspyleniia. *Zhurnal Tekhnicheskoi Fiziki*. St. Petersburg «Science», 2014, Vol. 84, Issue. 12, pp. 73 - 76. [in Russian]
- 4 Kostanovsky A.V., Zhilyakov L.A., Pronkin A.A., Kirillin A.V. Poluchenie tonkikhalmaznykh plenok pri magnetronnom raspylenii grafitovoi misheni. *Nanosystems, Nanomaterials, Nanotechnologies*. Institute of Metal Physics G.V. Kurdyumov, the National Academy of Sciences of Ukraine. 2008, Vol. 6, Issue 3, p. 911 -917.(2008).
- 5 Zvonareva T.K., Lebedev V.M., Polyanskaya T.A. et al. Elementnyi sostav i elektricheskie svoistva plenok a-C: HhCui, poluchennykh magnetronnym raspyleniem. *Fizika i Tekhnika Poluprovodnikov*. St. Petersburg «Science», 2000, Vol. 34, Issue 9, pp. 1135 -1141. [in Russian]
- 6 Borisov A.M., Mashkova E.S., Ekshayn V. Zakonomernosti raspyleniia i elektronnoi emissii grafita pri vysokodozovom obluchenii ionami azota. *Problems of Atomic Science and Technology*. Ser. Thermonuclear fusion. Moscow: National Research Center "Kurchatov Institute".2002, Vol. 1-2, pp.122 - 125. [in Russian]
- 7 Semenov A.P., Belyanin A.F., Semenov I.A. et al. Tonkie plenki ugleroda. II. Stroenie i svoistva. *Zhurnal Tekhnicheskoi Fiziki*. St. Petersburg «Science», 2004, Vol. 74, Issue 5, pp.101 - 104. [in Russian]
- 8 Shatokhin A.N., Putilin F.N., Roumiantseva M.N., Gas'kov A.M. Issledovaniia energeticheskikh kharakteristik i kinetiki osazhdeniia metallov iz vakuumnoi lazernoi plazmy na dielektricheskie podlozhki. *Bulletin of Moscow University. Series 2. Chemistry*. 2007, Vol. 48, No. 4, pp. 271 – 276. [in Russian]
- 9 Suzdalev I.P. *Nanotechnology: physical chemistry of nanoclusters, nanostructures and nanomaterials*. Moscow: Kom Kniga, 2006, 592 p. [in Russian]
- 10 Kovtun G.P., Verevkin A.A. *Nanomaterials: Technology and Material Science: A Review*. Kharkov, NSC KIPT, 2010, 73 p. [in Russian]
- 11 Rusanov A.I. *Thermodynamic basis of mechanochemistry*. St. Petersburg, Science, 2006, 221p.
- 12 Makhanov K.M., Ermaganbetov K.T., Chirkova L.V., Maukebaeva M.A. Sposob polucheniia tonkikh plenok grafita i oksida aliuminiia. *Zhurnal Tekhnicheskoi Fiziki*. St. Petersburg «Science», 2017, 87, Vol. 7, pp. 1057 – 1060. [in Russian]
- 13 Makhanov K.M., Ermaganbetov K.T., Chirkova L.V., Maukebaeva M.A. Method for Producing Graphite and Aluminium Thin Films. *Technical Physics*. Pleiades Publishing, Ltd. 2017, Vol. 62, No. 7, pp. 1073 -1076.
- 14 Ermaganbetov K.T., Chirkova L.V., Makhanov K.M. Method of Reserving of Graphite Films and Oxide of Aluminium in a Polymeric Matrix. *Proceedings of the 2017 IEEE 7th Intern.Conference on Nanomaterials: Applications & Properties (NAP-2017)*. Zatoka, Ukraine, 2017, Vol. 2. pp. 111 – 114.