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PHYSICAL SCIENCE OF MATERIALS

Method for Producing Graphite and Alumina Thin Films

K. M. Makhanov[a]*, K. T. Ermaganbetov[a], L. V. Chirkova[a], and M. A. Maukebaeva[b]

[a] *Buketov State University, Universitetskaya ul. 28, Karaganda, 100028 Republic of Kazakhstan*
[b] *Zhansugurov State Univrsity, ul. Zhansugurova 187a, Taldykorgan, 040009 Republic of Kazakhstan*
**e-mail: makanov@inbox.ru*

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Abstract—A simple comprehensible method for producing graphite and alumina films has been suggested. The optical properties of a graphite suspension in toluene and a suspension of natural clay with a high content of alumina particles in water have been studied. It has been found that the optical density of the suspensions varies from layer to layer, and the lowest optical density has been observed in upper layers. Graphite and aluminum films have been prepared by taking samples from different depths. The microstructure of the films has been examined. It has turned out that alumina particles coalesce into regularly shaped objects in the form of snowflakes. In addition, alumina films obtained from samples taken from different depths of the suspension have different thicknesses. In thin and thick films, the particle size is 0.29 and 2.81 μm or more, respectively.

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INTRODUCTION

Carbon films are finding wide applications in industry due to their unique properties [1–4]. For example, they are applied as coatings for laser disks [5] and antireflection coatings [6]. Carbon films can be obtained by different methods, such as magnetron sputtering of graphite [7–9], sputtering of graphite by a laser beam [10, 11], and laser oblation of the target [12]. They all require special conditions and expensive sophisticated equipment [13]. The aim of this work was to develop a method for producing graphite and alumina films and study their morphology and optical performance.

It was noted earlier [14, 15] that, if a material is ground to a fine-grained powder, some of the particles may be as small as several hundred nanometers or less across. It is natural to suppose that, after this powder is suspended, the sedimentation rate of particles and, correspondingly, their distribution over the depth of the suspension will depend on their weight and size. Heavy particles will reach the bottom first, lighter (finer) particles will sediment with a lower velocity, and the lightest particles will stay in upper layers and remain suspended for a long time. The main idea of the method is that one can prepare films with different thicknesses by taking samples from different depths of the suspension, since the thickness of the films is expected to depend on the geometric size of the particle.

EXPERIMENTAL

In experiments, we used single-crystalline graphite and natural clay with a high alumina content. Fine graphite and alumina particles were prepared by grinding the powdered material in a mortar. Graphite and clay were suspended in toluene and distilled water, respectively. To prepare films, we took samples from suspension layers 1.0, 2.5, and 3.5 cm deep using a glass dropper. The samples were applied on a precleaned, degreased surface of quartz substrates. Films for microstructural examination under an electron microscope were prepared by applying samples on glass substrates covered by a conducting layer of fluorine-doped tin oxide. Before being used, the glass dropper and substrates were thoroughly cleaned. To prevent foreign objects from falling on the film surface, the substrates were stored in sealed glass containers.

The absorption spectra of the as-prepared suspensions and films were taken in the spectral range 350–900 nm using an SF-46 spectrophotometer. The microstructure of the films was examined under a MIRA3 LMU electron microscope (TESCAN, Czech Republic).

To take absorption spectra at different depths of the suspensions, a special cell holder with narrow windows at a height of 1.0, 2,5, and 3.5 cm from the upper edge of the cell was designed (Fig. 1). The windows on the front wall of the holder limit the light beam diameter and also serve as an orientation point for precisely determining the depth of the suspension. Adjusting the

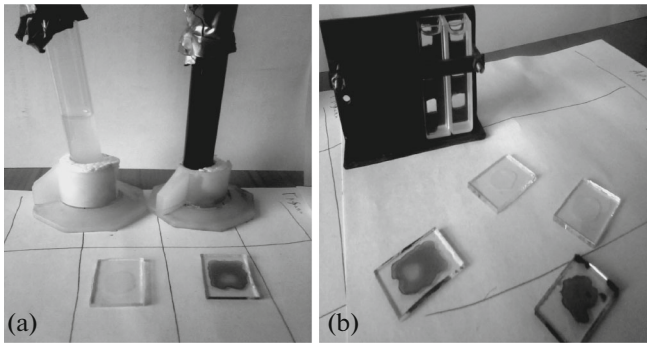

Fig. 1. (a) Containers with ready suspensions, (b) cell holder, and prepared films.

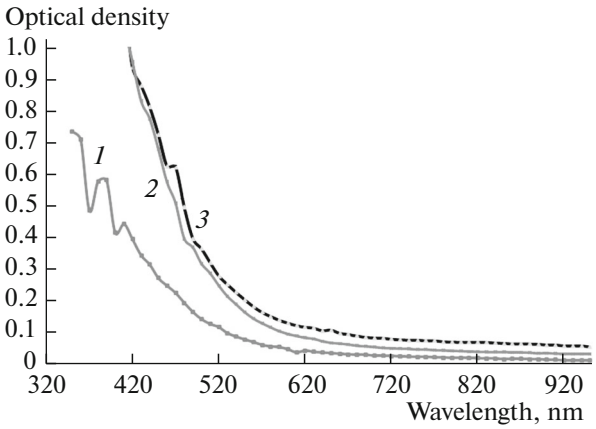

Fig. 3. Absorption spectra of the graphite films. Samples used to prepare the films were taken from suspension depths of (*1*) 1.0, (*2*) 2.5, and (*3*) 3.5 cm.

cells for height was made using a step motor with a gear-wheel drive. This drive provides smooth ascent and descent of the holder to a desired height. The prepared suspensions were preliminary kept in the cell section for 6 h. It was assumed that only the lightest particles will remain on the surface within this time.

RESULTS AND DISCUSSION

The absorption spectra of the suspensions are shown in Fig. 2. It can be seen that optical density D of the clay suspension at a depth of 1 cm from the surface is roughly equal to 0.04 (curve *1*). At a depth of 2.5 cm (curve *2*), the absorbed energy slightly grows (D = 0.2), and, at a depth of 3.5 cm, the optical density sharply rises, reaching 0.2 at a wavelength of 740 nm (curve *3*). The optical density is maximal (D = 0.6) at a wavelength of about 380 nm. It should be noted here that the absorption spectra of the clay suspension taken in the spectral interval 350–900 nm do not exhibit distinct vibrational absorption bands.

Similar results were obtained for the graphite suspension. It was found that the optical density at a

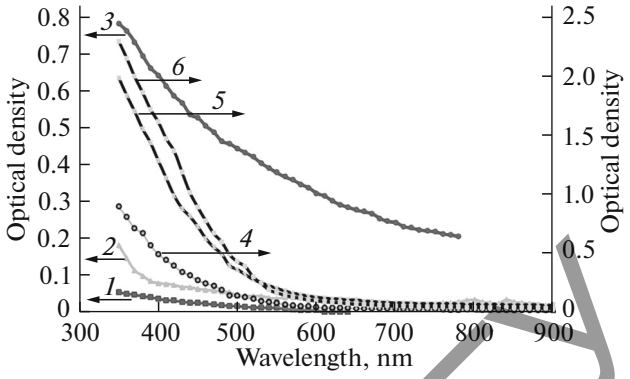

Fig. 2. Absorption spectra of the suspensions of (*1–3*) clay with a high alumina content in water and (*4–6*) graphite in toluene.

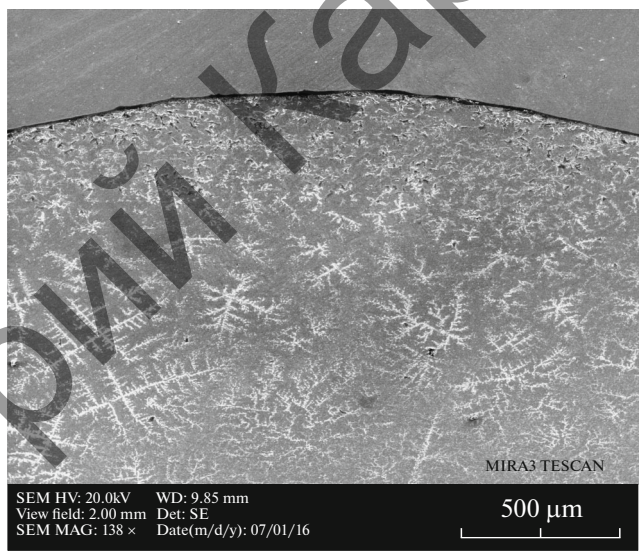

Fig. 4. Micrograph of the alumina film structure. Interface is shown for illustration properties.

depth of 1 cm is D = 0.29 (Fig. 2, curve *4*) in the short-wave part of the spectrum and falls to zero in the long-wave part. It can be seen in Fig. 2 that the spectrum of the graphite suspension does not exhibit distinct absorption bands either. The above dependences suggest that the optical density grows with depth of sample taking. This may take place if the number of absorption centers is small or they are completely absent. This finding can also be viewed as an indirect validation of the idea underlying the method suggested.

Next, we studied the optical performance of films containing graphite and alumina particles. It was found that dependences obtained for films made of the clay suspension are generally similar to the dependence obtained for the clay suspension. Figure 3 shows the absorption spectra of graphite films made of samples taken at the three depths mentioned above.

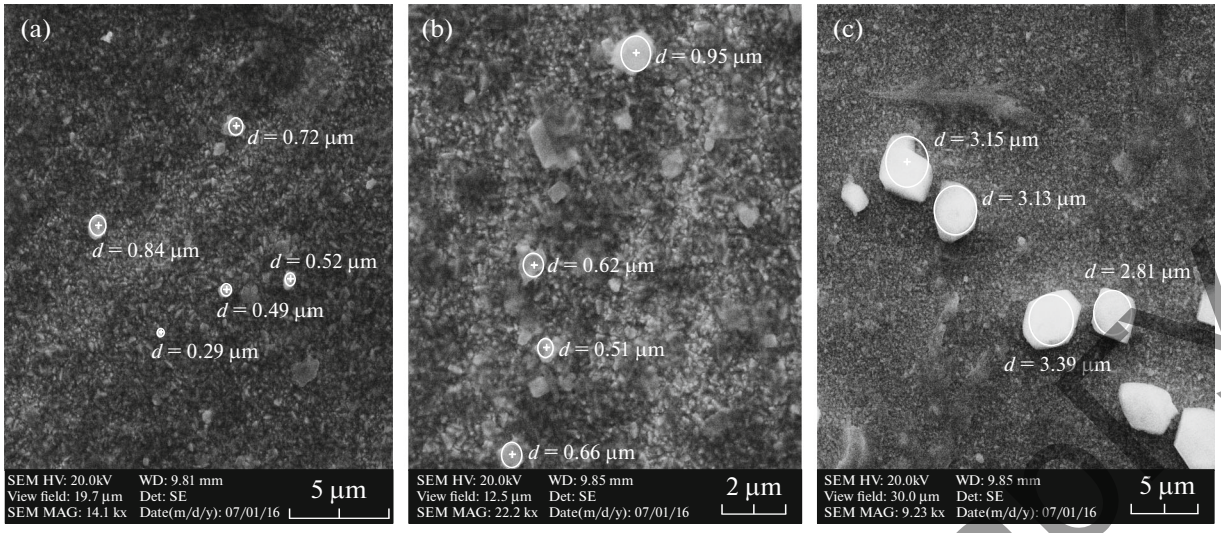

Fig. 5. Micrographs of alumina particles in the films prepared from the samples taken from a depth of (a) 1.0, (b) 2.5, and (c) 3.5 cm.

As follows from these data, the optical density of the film made of the sample that was taken from a depth of 1 cm (curve *1*) is much smaller than that of films made of samples taken from the other depths. In addition, the spectra contain bands characteristic of vibrational transitions. The nature of these bands is still unclear. One can suppose that they may be attributed to the presence of impurities in the clay suspension. In general, the run of the absorption curves indicates the presence of absorption centers in the short-wave part of the spectrum. Thus, the absorption spectra show that films prepared from samples taken from various depths differ in absorption density. The weakest absorption is observed for films prepared from samples taken at a depth of 1 cm.

An examination of the film microstructure under the electron microscope provided more information about the size of graphite and alumina particles in the films. Micrographs of the films with alumina particles are shown in Fig. 4. The micrographs were taken of the film–substrate interface. It can be seen that the alumina particles coalesce into regularly shaped objects in the form of snowflakes; that is, they demonstrate an ordered arrangement instead of being distributed randomly.

Figure 5 shows micrographs of alumina particles from three different films. The particles in Fig. 5a belong to the sample taken from a depth of 1 cm. Those shown in Figs. 5b and 5c belong to samples taken from depths of 2.5 and 3.5 cm, respectively. It follows from the micrographs that particles grow in size with increasing depth. A minimum particle size of 0.29 μm is observed at a depth of 1 cm (Fig. 5a) and the maximum particle size (3.39 μm) was at a depth of 3.5 cm (Fig. 5c).

CONCLUSIONS

At the given stage of the investigation, we demonstrated the feasibility of producing graphite and alumina films using a simple vacuum-free method. By taking samples from different depths of the suspension, we succeeded in preparing films with different thickness on quartz substrates. The minimal size of alumina particles depends on the depth at which the sample was taken; this equals 0.29 μm in the samples taken from upper (1 cm deep) layers and brows with depths of 2.81 μm or more. A morphological examination of the films revealed that alumina particles coalesce into regularly shaped objects in the form of snowflakes.

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