

## Influence of few-layer graphene on the complex of strength and thermophysical properties of polymer composites obtained by DLP by 3D printing

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**Abstract:** The digital light processing method (DIGITAL LIGHT PROCESSING or DLP) is one of the most affordable 3D printing methods, which allows you to obtain high-precision products, which, however, are inferior in their characteristics to products obtained by other 3D printing methods. Due to their high characteristics, graphene nanostructures can solve this problem by acting as effective modifying additives. However, despite all the promise of this approach, the introduction of graphene nanostructures into actual practice has not yet occurred due to their high cost due to the imperfection of the methods of their synthesis. The paper considers the effectiveness of using few-layer graphene synthesized according to the author's method under conditions of self-propagating high-temperature synthesis from cellulose as a modifying additive to improve the complex strength and thermophysical properties of products from photopolymer resins obtained by DLP 3D printing. It was found that adding few-layer graphene makes it possible to increase Brinell hardness up to 1.8 times, bending strength up to 1.5 times, and thermal conductivity up to 2.2 times compared to the original polymer when using no more than two wt. % few-layer graphene. The data obtained indicate the high efficiency of the synthesized few-layer graphene as a modifying additive in creating products from photopolymer resins using the DLP 3D printing method.

**Keywords:** polymer composites; digital light processing; few-layer graphene; hardness; bending strength; thermal conductivity; heat capacity.

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## Влияние малослойного графена на комплекс прочностных и теплофизических свойств полимерных композитов, полученных DLP-методом 3D-печати

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**Аннотация:** Рассмотрена эффективность применения малослойного графена (число слоев не более 5), синтезированного по авторской методике в условиях самораспространяющегося высокотемпературного синтеза из целлюлозы в качестве модифицирующей добавки для повышения комплекса прочностных и теплофизических

свойств изделий из фотополимерных смол, полученных методом DLP 3D-печати. Несмотря на высокие характеристики графеновых наноструктур, к которым относится и малослойный графен, данный класс материалов до сих пор не применяется в промышленности из-за невозможности синтеза больших объемов материала высокого качества и с приемлемой себестоимостью. Предложенный метод синтеза позволяет получать большие объемы малослойного графена, не содержащего в своей структуре дефекты Стоуна–Уэйлса. Установлено, что добавление малослойного графена позволяет добиться роста твердости по Бринеллю до 1,8 раз, прочности на изгиб до 1,5 раз и теплопроводности до 2,2 раз по сравнению с исходным полимером при использовании не более 2 масс. % малослойного графена. Дальнейшее увеличение концентрации малослойного графена не привело к дальнейшему росту свойств конечного композита. Полученные данные свидетельствуют о высокой эффективности синтезированного малослойного графена в качестве модифицирующей добавки при создании изделий из фотополимерных смол методом DLP 3D-печати.

**Ключевые слова:** полимерные композиты; DLP; малослойный графен; твердость; прочность на изгиб; теплопроводность; теплоемкость.

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## 1. Introduction

Over the past 10 years, 3D printing as a technique for creating finished polymer products has come a long way in development and has become available to a wide range of both researchers and ordinary consumers [1]. One of the varieties of 3D printing technique is the digital light processing method (DIGITAL LIGHT PROCESSING or DLP). This technique is based on the layer-by-layer polymerization of resins under the influence of light [2]. Compared to other 3D printing techniques, such as Fused Deposition Modeling (FDM), DLP produces end products with precise dimensions and shapes. Also, this method is more productive (has a higher printing speed) [3].

The main disadvantage of the DLP method is the relatively low strength of the final products compared to products obtained, for example, by the FDM method. One of the most promising ways to solve this problem is the creation of composite materials using graphene nanostructures as filler. The reason for this is their superior performance. Considering the properties of single-layer graphene, it should be noted that its thermal conductivity is up to  $5000 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$  [4], Young's modulus is up to 1 TPa [5], while its specific surface is estimated at  $2630 \text{ m}^2 \cdot \text{g}^{-1}$  [6]. However, obtaining a “true” single-layer graphene in the form of a powder, even in gram quantities, is an extremely difficult task; therefore, the so-called graphene nanostructures, which are particles consisting of 2–10 layers of graphene [7]. Despite the fact that the properties of graphene nanostructures decrease with an increase in the number of graphene layers [8], many studies, including [9–11], noted the high efficiency of graphene nanostructures when creating products from polymer composites using DLP 3D printing. At the same time, graphene

nanostructures make it possible to obtain a higher increase in properties at the same concentrations compared to classical fillers such as carbon black [12, 13] or graphite [14]. For example, in [15], the authors noted that the addition of even large volumes of carbon black (up to 50 wt. %) does not lead to a significant increase in the thermal conductivity of the polymer matrix at temperatures up to 70 °C.

However, the researchers also note that the experimental results of using graphene nanostructures do not match the theoretical estimates. The main reasons for this discrepancy include the presence of various structural defects in graphene nanostructures, which can significantly reduce their characteristics [16]. One of the types of structural defects in graphene nanostructures are the Stone-Wales defects. The Stone-Wales defect, which occurs due to a 90° rotation of adjacent carbon atoms about their center, is a connected carbon ring with five and seven carbon atoms. It was shown in [17] that such defects reduce the efficiency of using graphene nanostructures in polymer composites.

In previous papers [18, 19], we described the developed procedure for the synthesis of few-layer graphene (FLG), containing no more than 5 graphene layers in its structure, from cyclic biopolymers under conditions of self-propagating high-temperature synthesis (SHS). At the same time, such MGs were devoid of Stone–Wales defects in their structure [20]. In [21], we showed that synthesized FLG can significantly improve the complex of strength and thermal properties of nitrile butadiene rubber.

The purpose of this paper is to study the effect of FLG synthesized from cellulose under SHS conditions on the complex of strength and thermophysical properties of products made of polymer composites based on photopolymer resins obtained by DLP 3D printing.

## 2. Materials and Methods

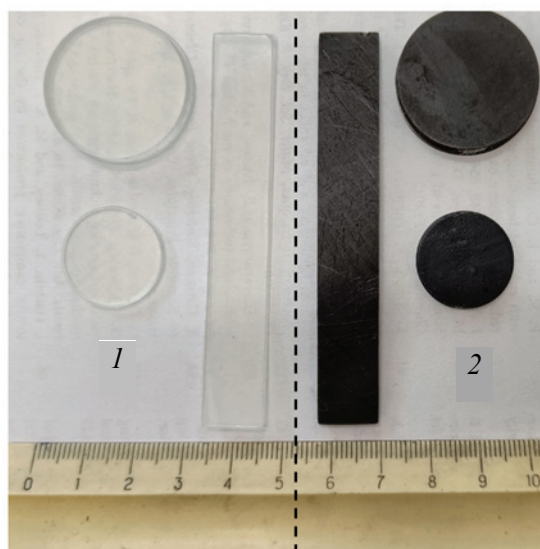
### 2.1. Materials

A commercial photopolymer resin of the Anycubic brand (405 nm, Anycubic, China) was taken as the starting material for the creation of polymer composites. FLG synthesized from cellulose under the conditions of the SHS process was taken as a modifying additive. The procedure for synthesizing FLG was described in detail in [18].

### 2.2. Methods

#### 2.2.1. Synthesis of polymer composites

Powdered portions of FLG were added to the original photopolymer resin with constant stirring (500 rpm) using an overhead stirrer. Samples were added in portions of 1/10 of the mass of the entire sample. The FLG concentration was 0.25–4.00 wt. %. After adding the entire sample, the resulting suspension was additionally stirred for 30 minutes at 1000 rpm and the resulting suspensions were treated in the ultrasonic field (ultrasonic bath, 22 kHz) for 30 minutes. From the resulting suspensions, samples were printed using the DLP 3D printing method (Anycubic Photon S printer, China). The thickness of each of the successive layers subjected to irradiation for curing was 0.1 mm, exposure time 8 seconds. Figure 1 shows various variants of the samples obtained in the study.



**Fig. 1.** The appearance of products made of polymer composites based on photopolymer resins obtained by DLP by 3D printing: 1 – obtained from unmodified resin (transparent samples); 2 – from modified resin at an FLG concentration of 2 wt. % (black samples)

The resulting samples after 3D printing were additionally processed with a UV lamp with a wavelength of 405 nm for 30 minutes (UV radiation power 50 W). This exposure time was due to the cessation of changes in the properties of the samples at a longer exposure.

#### 2.2.2. Study of few-layer graphene parameters

Electronic images of FLG were obtained with a Tescan Mira 3M scanning electron microscope (SEM) (Tescan, Brno, Czech Republic). The accelerating voltage was 20 kV. The diffraction pattern of the FLG sample was obtained on an XRD-7000 X-ray diffractometer (Shimadzu, Kyoto, Japan). The shooting speed was 0.5 degrees/min,  $\text{CuK}\alpha = 0.154051$  nm. The Raman spectrum (RSS) of FLG was obtained on a Confotec NR500 instrument (SOL Instruments, Minsk, Belarus). The laser length was 532 nm. The dispersion of FLG particles was measured by laser diffraction using a Malvern Mastersizer 2000 instrument (Malvern Instruments, Malvern, UK). For measurements, a sample of FLG (0.25 wt. %) was dispersed in water by ultrasonic treatment (ultrasonic bath, 22 kHz) for 30 minutes.

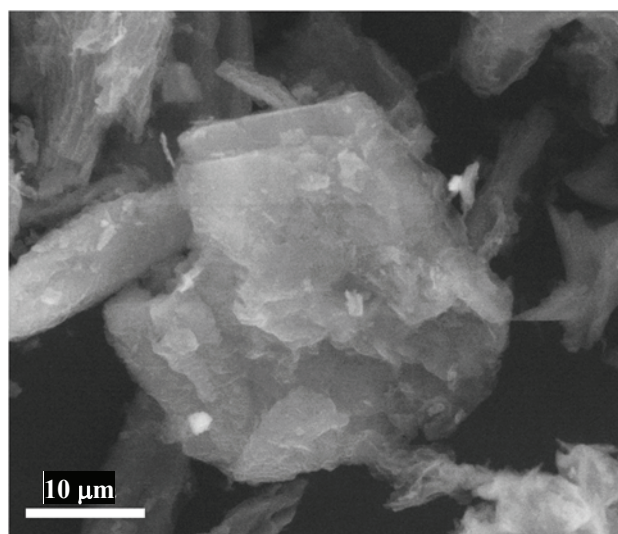
#### 2.2.3. Study of the properties of polymer composites

Hardness measurements were carried out by the Brinell method on an ITB 3000 AM hardness tester (Metrotest, Neftekamsk, Russia). For measurements, a ball of 2.5 mm was used; the load was 62.5 kgf, in accordance with Russian Standard 23677-79. The samples were disks 5 mm thick and 20 mm in diameter. The measurement error was 5%. Measurements of bending strength were carried out on a hydraulic press PM-FLG4 (Stroypribor, Chelyabinsk, Russia) in accordance with Russian Standard 4648-2014. The samples were plates 80 mm long, 10 mm wide and 3 mm thick. The measurement error was 6%. The thermal conductivity and heat capacity were measured by the monotonic cooling method on a KITT Polymer instrument (Teplofon Design Bureau, Novomoskovsk, Russia) in accordance with Russian Standard 23630.1-79. The samples were disks 10 mm in diameter and 2 mm thick.

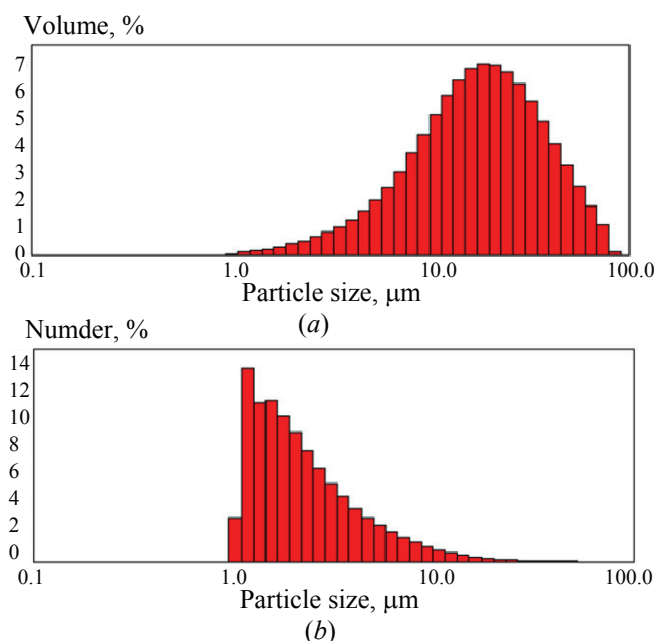
## 3. Results and Discussion

Figure 2 shows a micrograph of the FLG surface obtained by SEM. As can be seen from Fig. 2, the few-layer graphene used as a modifying additive consisted of aggregates of flat particles with linear sizes up to several tens of microns, which is confirmed by the results of measuring the dispersion presented in Fig. 3.





**Fig. 2.** Electronic image of few-layer graphene.  
Linearscale – 10 μm

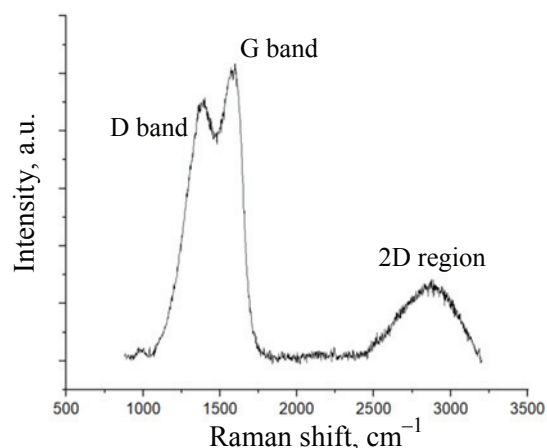


**Fig. 3.** Results of particle dispersion measurement:  
*a* particle dispersion distribution by volume;  
*b* particle dispersion distribution by the number of particles

As can be seen from Fig. 3, despite the fact that the sample contains particles with a dispersion of up to several tens of microns, the number of such particles is small, and most of the particles have a dispersion of 1–5 microns.

Figure 4 shows the results of the FLG study by Raman spectroscopy.

As can be seen from Fig. 4, the FLG sample spectrum contains peaks D  $1391\text{ cm}^{-1}$ , G  $1586\text{ cm}^{-1}$  typical for graphene nanostructures and a region  $2250\text{--}3250\text{ cm}^{-1}$  (2D region) with a superposition of

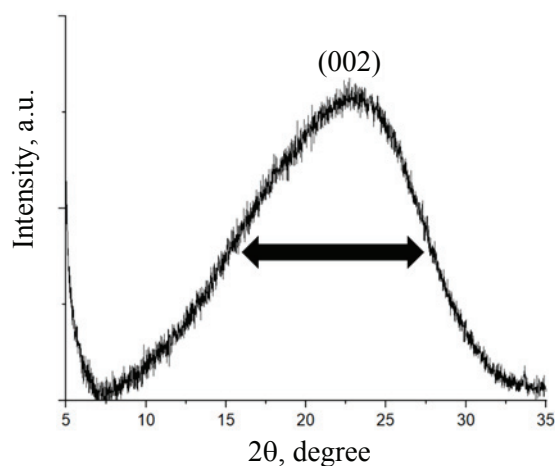


**Fig. 4.** Raman spectrum of FLG sample

many peaks characteristic of graphene structures ( $D^*$ , 2D,  $D+D'$  and  $2D'$ ) [22]. As noted in [23], such Raman spectra are characteristic of graphene nanostructures with various folds, which are not point defects. Since, due to the broadening of the 2D region, it is impossible to determine the number of layers in the sample based on the position and shape of the 2D peak [24], we additionally conducted FLG studies using the X-ray diffraction method.

Figure 5 shows the diffraction pattern of the FLG sample in the  $2\theta$  range of angles from 10 to 35 degrees.

Based on the position of the 002 peak, as well as the FWHM of the 002 peak, the crystallite size ( $L$ ) was calculated using the Scherrer formula [25], which, together with the data on the interplanar distance ( $d$ ), made it possible to determine the number of layers in the synthesized sample ( $N$ ) using the formula  $L = N/d$  (Table 1).



**Fig. 5.** Diffraction pattern of the few-layer graphene

**Table 1.** Results of calculating the number of layers

Crystallite size $L$ , nm	Interplanar spacing $d$ , nm	Number of layers $N$
1.50	0.38	4

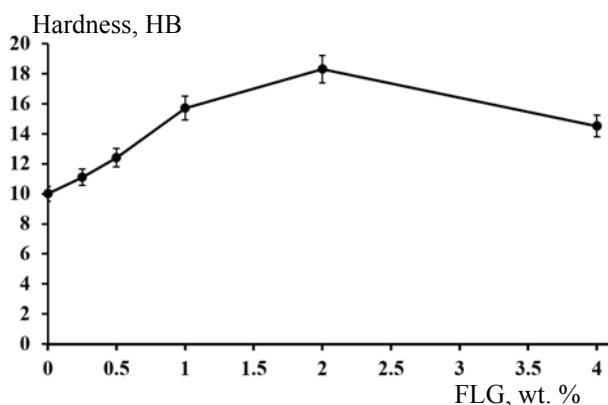
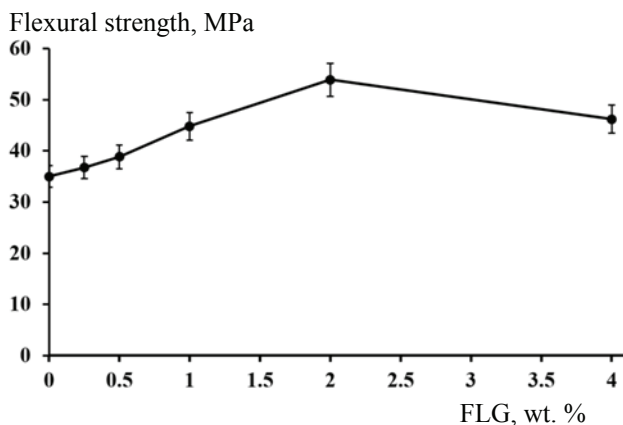
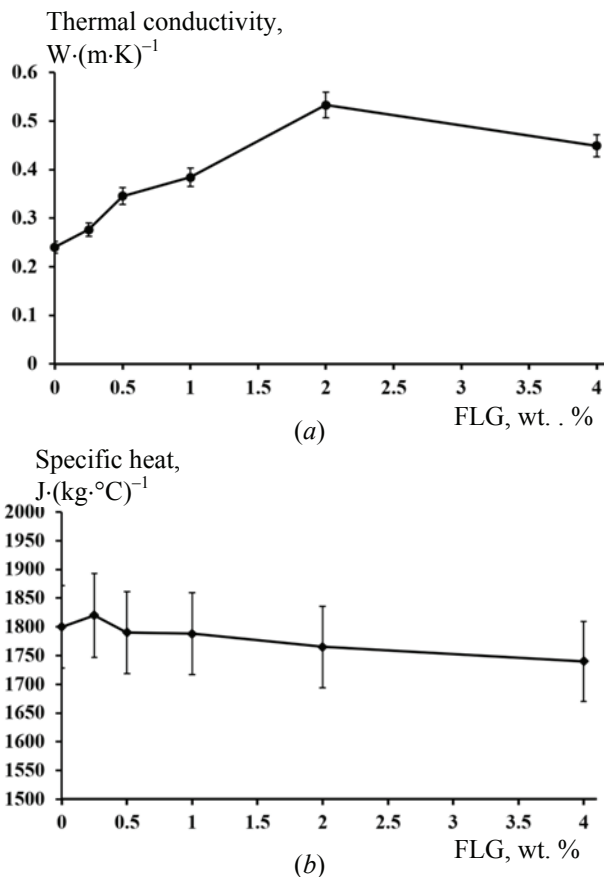
As can be seen from Table 4, the synthesized FLG particles consist of no more than 4 layers of graphene.

We consider the FLG influence on the complex of strength and thermophysical properties of final polymer composites.

Figures 6 and 7 show the results of a study of Brinell hardness and bending strength of synthesized polymer resin samples.

As can be seen from Figs. 6 and 7, the introduction of FLG in small amounts makes it possible to increase the Brinell hardness up to 1.8 times and the bending strength up to 1.5 times at a FLG concentration of not more than 2 wt. %.

Figure 8 shows the thermal conductivity measurements and specific heat capacity of polymer samples.

**Fig. 6.** Dependence of sample hardness on FLG concentration**Fig. 7.** Dependence of the flexural strength of samples on the concentration of FLG**Fig. 8.** Dependence of thermal conductivity (a) and specific heat (b) of samples on the concentration of FLG

As can be seen from Fig. 8, the introduction of FLG in small amounts makes it possible to increase the thermal conductivity of the samples up to 2.2 times compared to the initial polymer.

At the same time, the specific heat capacity of the samples is practically independent of the FLG concentration (change within the measurement error).

The increase in the strength and thermophysical properties of polymer composites with the addition of graphene nanostructures is primarily due to the high properties of graphene nanostructures, which is clearly demonstrated by the results of measuring the properties of the composite. As noted earlier, the thermal conductivity of graphene can reach up to  $5000 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ , which leads to an increase in thermal conductivity (Fig. 8a). However, the specific heat capacity of graphene is about  $700 \text{ J} \cdot (\text{kg} \cdot ^\circ\text{C})^{-1}$  [26], so that the addition of FLG has almost no effect on the specific heat capacity of the polymer composite at relatively low (up to 4 wt. %) concentrations of the additive (Fig. 8b).

However, despite a significant increase in the properties of the final composites, the obtained values are still significantly inferior to the theoretically

expected results. In review [27], the authors note that the efficiency of using graphene nanostructures is greatly reduced when they are used in the form of powders (i.e., aggregates of particles), as well as under the influence of other factors such as the defectiveness of particles, the degree of their interaction with the polymer matrix, etc. Since FLG particles are actually distributed throughout the polymer in the form of aggregates (see Figs. 2 and 3), their efficiency is much lower than the expected theoretical assumptions. A similar mechanism was observed in [20].

It should also be noted that with an increase in the FLG concentration in the composite from 2 to 4 wt. %, no further increase in the properties of the composite is observed, and even a slight drop in characteristics occurs. This fact may be due to the fact that at 4 wt. % due to the large number of FLG particles, they interact with each other with the formation of secondary aggregates, which negatively affects the properties of the composite [28].

#### 4. Conclusion

A few-layer graphene synthesized from cellulose under the conditions of the SHS process has shown its high efficiency as a filler for increasing the complex of strength and thermophysical properties when creating composite products from polymer resins using the DLP 3D printing method. It was found that the addition of low-layer graphene makes it possible to achieve an increase in Brinell hardness up to 1.8 times, bending strength up to 1.5 times and thermal conductivity up to 2.2 times compared to the original polymer when using no more than 2 wt. % low-layer graphene. A further increase in the concentration of few-layer graphene did not lead to a further increase in the properties of the final composite, which may be due to the formation of secondary aggregates. However, in order to fully exploit the potential of using FLG as a builder in polymer resins, it is necessary to further improve the methods for dispersing FLG in the original polymer resin in order to avoid secondary aggregation of FLG particles.

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#### 6. Conflict of interests

The authors declare no conflict of interest.

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