# PHYSICS OF DEVICES, ELEMENTS AND SYSTEMS ФІЗИКА ПРИЛАДІВ, ЕЛЕМЕНТІВ І СИСТЕМ

UDC 621.793:678.073

#### DOI:10.30837/rt.2023.2.213.06

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# NANOPOLYMER OPTICALLY TRANSPARENT STRUCTURES, SYSTEMS AND DEVICES

## Introduction

Analysis of trends in the development of promising materials and technologies shows that the main efforts of researchers are currently focused on creating objects with dimensions comparable to the range length of electron nanostructures. Nanomaterials, the raw materials for the creation of which are individual atoms, molecules, molecular systems, nanoparticles no larger than 100 nm in size, have fundamentally new, often unique properties that differ from the properties of macroobjects due to a sharp increase in the reactivity of nanoparticles due to their high specific surface area. On the nanometer scale there are qualitatively new effects, properties and processes determined by quantum mechanics, dimensional quantization in small structures and other phenomena and factors. At the same time, the electronic structure is responsible for such material properties as optical absorption, electronic conductivity, chemical reactivity, and mechanical characteristics. Therefore, nanostructures reveal significantly different properties than material in volume, which can be used for practical purposes. In this regard, nanotechnology opens up new perspectives for electronics, optics, chemical industry, energy, medicine, biotechnology and many other areas of science and technology.

One of the priority research areas actively developing in recent years is the creation of transparent polymer compositions containing nanoscale fillers, which open up new prospects for optical and optoelectronic instrumentation [1].

High-tech and relatively cheap polymer optics is a means to solve technical problems associated with reducing the complexity of assembly, improving the design and reliability of various optical devices.

At the same time, new polymeric materials have confirmed their promise not only for conventional optics, but also for the purposes of laser optics and technology, where polymer lenses, deflecting plates, prisms are used, and organic glasses activated by generating organic dyes have been developed as new solid-state active media.

Luminophor containing polymers and composites are very attractive as luminescent probes, optical media for luminescent solar concentrators, electroluminescent organic LEDs, important for creating modern electronic devices, and energy-saving light sources.

Current trend is to create photochrome and other so-called "smart" materials based on optically transparent polymers. Significant scale of the latest research is aimed at the synthesis and study of polymers with nonlinear optical properties [2].

Aim of carried out work is to perform a search and analysis of data obtained from results of theoretical and experimental studies, as well as from literary sources and patents in the field of optical and optoelectronic instrumentation. Generalization of the obtained data and recommendations for the creation of optically transparent nanocomposites. Results of the work can be useful for further improvement of nano-containing transparent polymer composites and design and technological solutions not only for optical devices, but also for products of scintillation technology, photovoltaics and many other applications.

#### 1. Nanopolymer optically transparent composites

Polymer nanocomposites are polymeric materials that are filled with particles having at least one of the sizes in the nanometer range. The main differences between nanocomposites and macroand microcomposites are in the huge specific surface area of the filler-matrix interface, in large

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volume fraction of interfacial boundary, and in small average distances between the filler particles. When creating polymer nanocomposites, the main task of the polymer matrix is to ensure compatibility with nanoparticles and to ensure uniform distribution of nanoparticles. Therefore, following requirements are imposed on polymers for preparation of composites: good adhesion to surface of the filler, high strength, and a number of other properties that make it possible to carry out technological processes for preparation of polymer nanocomposites, required level of viscosity for impregnation or mixing with dispersed fillers, heat resistance under conditions of processing into products, etc. It should be noted that another important role of polymers is their stabilizing role. In composites, the filler-additives are of particular importance, since the electrical, magnetic, optical, and other properties of the material tend to depend substantially on the corresponding characteristics of the nanoparticles. Of great importance is the interaction between the filler and the polymer matrix at molecular level, which can lead to synergy of the beneficial properties of organic and inorganic components of the material.

To create them, polymers are filled with nanoparticles of noble metals or semiconductors 1-20 nm in size, in which the strong spatial localization of valence electrons leads to the appearance of properties that differ from those of both a solid state and isolated molecules. The most promising for obtaining quantum size effects are particles whose size does not exceed 10 nm [2, 3].



Fig. 1. Structural diagram of a polymer nanocomposite: a – surface of the nanoparticles is not treated with surfactants, b – surface of the nanoparticles is treated with surfactants

Due to the high surface energy and presence of functional groups capable of interaction on the surface, nanoparticles are prone to agglomeration and clumping (Fig. 1, *a*). Therefore, during conventional mixing with polymer melts, the sizes of a large proportion of particles are in the micron and submicron range. Treatment with surfactants capable of being adsorbed at interphase boundaries and preventing aggregation significantly increases degree of particle dispersion in the polymer matrix (Fig. 1, *b*). Thus, surfactants and blocking of functional groups on the particle surface make it possible to partially solve the problem of agglomeration. Nanocomposites containing high concentrations of a nanocrystalline component actually become hybrid materials with a comparable content of nanocrystals and a polymer matrix.

Under favorable conditions (homogeneous distribution of nanocrystals, absence of their coagulation, monodispersity), the nanomaterial is a homogeneous optical medium whose light scattering and rheological properties are similar to those of the polymer matrix even at high nanocrystal concentrations, and optical and physical properties are a superposition of the properties of both components. Small size of nanoparticles leads to fact that polymer nanocomposites can be considered as an optical medium, and for it, as for a homogeneous medium, optical parameters can be introduced – the refractive index and the absorption index. In this regard, nanostructuring is a new way to create optical media in which the resulting set of properties cannot be achieved by other means. Highest concentration in an organic matrix can be obtained for inorganic nanocrystals coated with lowmolecular shells that stabilize them. Common property of optical nanocomposites is that introduction of high concentrations of nanocrystals into the matrix leads to change in the properties and structure of the matrix. In this case, the greater the change, the higher the concentration of introduced nanocrystals. However, the magnitude with which this effect begins to manifest itself is different for different types of nanocomposites. This effect is observed regardless of method of synthesis and composition of the nanocomposite.



Fig. 2. Light scattering in an ordered system of nanoparticles in the bulk of a polymer material

Nanoparticles embedded in original polymers do not cause light scattering if they are uniformly distributed and their size is much smaller than the radiation wavelength (Fig. 2). Refractive index of the material with introduced nanoparticles is determined by following expression:

$$n = \frac{n_1 V_1 + n_2 V_2}{V_1 + V_2} \tag{1}$$

where  $V_1$  and  $V_2$  as well as  $n_1$  and  $n_2$  are volumes of source material and nanoparticles and refractive index of source material and nanoparticles respectively.

At high concentration of nanoparticles, the index of refraction of a nanocomposite with built-in nanoparticles can be much higher than that of the source material, which increases the light output of light-emitting semiconductor light-emitting diodes or light transmission for semiconductor light receivers and optical systems [3-5].

## 2. Organosilicon composite for connecting optical elements

Components of some units of optical systems (objectives, eyepieces, wrapping systems, achromatic wedges, complex prisms, mirror reflectors, light filters, polaroids, grids, etc.) are connected together into monoblocks. As a connecting substance, various organosilicon compounds are used, which can improve the manufacturability of structures and performance of optical systems. There are many connection methods such as gluing, sintering, optical contact, welding and soldering. In this case, connecting substance must meet following requirements: not change optical properties of the connected parts, provide sufficient mechanical, chemical, thermal and light strength of the connection.

Organosilicon lubricating compositions are used as an optical contact between light source and photodetector, which provide maximum light transmission in contact, are stable in temperature range of minus 70°C plus 200°C. They are non-toxic, chemically inert with respect to structural materials, have a low dependence of viscosity on temperature, and high adhesion to contact surfaces. However, known organosilicon compositions contain functional groups that lead to crosslinking of a polymer and formation of solid compounds.

To connect and seal optical elements using a plastic base and a thickener, a new composition was proposed in [6], which consists of a mixture of polydimethylsiloxane and polymethylphenylsiloxane liquids with a viscosity of 3000 to 40000 mm<sup>2</sup>/s at 20°C temperature and a silicon dioxide thickener. The composition has refractive index of 1.41 - 1.43 and penetration

value (density) of 160 - 280 units. Moreover, it operates in temperature range from minus 70°C to plus 300°C, with following composition (Table 1):

	Table 1
Mixture of the optical composition	
Base (mixture of polydimethylsiloxane and polymethylphenylsiloxane liquid), wt. %	90 - 96
Thickener – silicon dioxide ( $SiO_2$ ), wt. %	10 - 4

The base of the silicon organic composition is mixture of polymethylsiloxane liquid (PMS), general formula:  $(CH_3)_3SiO[(CH_3)_2SiO]_n Si(CH_3)_3$ , with viscosity between 1000 and 50,000 mm<sup>2</sup>/sec and a polymethylphenylsiloxane liquid (PFMS), general formula:  $(CH_3)_3 SiO[(CH_3)_2SiO]_k [CH_3(C_6H_5)SiO]_m Si(CH_3)_3$ , where k/m = 10/1, with viscosity from 10,000 to 20,000 mm<sup>2</sup>/s in ratio PMS-60 – 40%, PFMS – 40 – 60%.

Manufacturing process of the organosilicon composition is as follows: in container equipped with heater, mixing device and thermometer, loaded with 180-270 g PMS fluid with viscosity of 1000-20000 mm<sup>2</sup> / s and 270 – 180 g PFMS fluid with viscosity of 10000 – 20000 mm<sup>2</sup> / s, the contents of the container stirred and obtained 450 g mixture with viscosity 3000 – 20000 mm<sup>2</sup> / s, which is basis of the composition, then added 50 – 20 g of silicon dioxide powder, the mass is heated to temperature of 40-60°C and stirred for 3 – 4 hours. After that the resulting mass is cooled to room temperature, unloaded and analyzed in terms of quality indicators:

1) Appearance – colorless transparent ointment plastic mass.

2) Penetration at  $20^{\circ}C - 160 - 280$  units.

3) Refractive index  $(n_d^{20})$  at  $20^{\circ}$ C - 1.41 - 1.43.

4) Frost resistance (pour point), °C – minus 70.

5) Thermal stability, % weight loss at  $300^{\circ}$ C for 2 hours – 1.0.

The organosilicon composition is non-toxic, chemically inert with respect to structural materials, has low dependence of viscosity on temperature, and high adhesion to contact surfaces. High viscosity of the base composition  $(3000 - 40000 \text{ mm}^2/\text{s})$  and small change in its value with temperature ensures normal operation of optical devices, smooth running and clear fixation of moving parts in winter and summer.

# 3. Plastic scintillator with nanostructured phosphors

Plastic scintillators (PS) are solid solutions of activating additives in polymer bases. History of their development begins in 1950, when the first scintillation composition based on polystyrene with pterphenyl was obtained. Creation of PS has become a new direction in development of scintillation method due to their unique properties. These include the following properties:

- high speed (0.5 - 3 ns);

- high transparency to its own radiation;

- manufacturability, ease of production and processing (possibility of obtaining PS of almost unlimited size and given shape);

- resistance to moisture, atmospheric and mechanical influences;

- relatively high radiation resistance;

- sufficient stability of scintillation characteristics in wide temperature range;

- relative cheapness;

- high fire safety and non-toxicity.

To date, polystyrene and vinyl toluene are widely used as the polymer base of PS. The main scintillation characteristics of the plastic scintillator based on vinyl aromatic polymer:

light output ~ 8500 photon / MeV;

- main emission band – 420 nm;

- scintillation duration -0.9 ns rise and 2-3 ns fall of the scintillation pulse.

Modern development of physical experiment requires creation of new advanced devices based on scintillators with improved time characteristics and light output values. But, at the same time, they must be cheap enough to be able to create a large-scale production of detector devices for experiments in high energy physics. Thus, there is need to improve the properties of PS, to create faster and more efficient plastic scintillators compared to already existing traditional PS [7 - 9].

PS proposed in [10] can be used in nuclear physics, high-energy physics, radiation chemistry, nuclear industry, radiation medicine, X-ray and gamma-ray astronomy. In diagnostics of fusion, in determining the lifetime of positrons and in a number of other tasks, in which are widely used fast-acting plastic scintillators with short luminescence time.

Technical solution considered in this work belongs to field of creating materials for scintillation technology, namely to plastic scintillators with nanostructured luminophores.

Since 60s of 20th century, not single fundamental technical solution has been proposed that would significantly increase light output of PS. The light output of three-component PS (polymer base, primary phosphor, secondary phosphor) primarily depends on the efficiency of transfer of electronic excitation energy from polymer matrix to the primary and, further, to the secondary phosphor. Due to low concentration of the secondary phosphor, energy transfer to it occurs due to photon mechanism, which leads to increase in duration of scintillation and decrease in the light output of the scintillator. Low concentration of the secondary phosphor in PS allows the self-absorption to be reduced, so that higher light output can be obtained. Attempt to increase efficiency of energy transfer by increasing concentration of the secondary phosphor leads to an increase in self-absorption and, consequently, to decrease in the light output of PS, therefore, this technical solution is used only in case of thin-film (0.001 - 0.01 cm) PS, which have very narrow scope.

Task of technical solution proposed in [10] is to obtain a new PS with a fundamentally new distribution of primary and secondary phosphors in the polymer matrix, due to which efficiency of nonradiative energy transfer from the primary to the secondary phosphor is close to 100%.

Technical result that can be obtained by implementing this solution:

1) the light output of the new PS relative to anthracene is up to 100 - 110%;

2) light attenuation coefficient at wavelength corresponding to maximum in fluorescence spectrum of the secondary phosphor 0.0015 - 0.0025 cm<sup>-1</sup>.

3) duration of scintillation 1 - 3 ns.

This problem is solved by creating a new PS consisting of a polymeric base, which contains primary and secondary phosphors connected by silicon atoms in nanoscale branched macromolecules. Total number of primary and secondary phosphor links in a macromolecule is from 3 to 45. Ratio of number of primary phosphor links to number of secondary phosphor links:

$$2 \le \frac{N_{L1}}{N_{L2}} \le 14$$
 (2)

where  $N_{L1}$  is number of primary phosphor links in the macromolecule and  $N_{L2}$  is number of secondary phosphor links in the macromolecule. Distance between the centers of any two adjacent links is no more than 1,2 nm.

As a polymer base, any polymer from group of vinyl aromatic polymers can be used. In this case, the primary phosphor is selected from the group of compounds in which the maximum of the long-wavelength absorption spectrum band is in range from 270 to 350 nm. Quantum yield of fluorescence is not less than 5 %. The secondary phosphor is selected from the group of compounds in which the maximum of long-wavelength band of absorption spectrum is in range from 330 to 400 nm. Quantum yield of fluorescence is not less than 30 %. An increase in the light output of the scintillator and reduction in duration of scintillation is achieved due to fact that in a nanosized branched macromolecule with claimed parameters, efficiency of nonradiative transfer of electronic excitation energy from units of the primary to the units of the secondary phosphor can reach 100 %.

In a conventional three-component scintillator (with uniform distribution of primary and secondary phosphor molecules in volume of the polymer matrix) efficiency of irradiation-free energy transfer does not exceed 0.1 %. In a conventional scintillator, there is a radiative transfer of electronic excitation energy from the primary to the secondary phosphor, efficiency of which cannot be greater than the quantum yield of the primary phosphor. For the main primary and secondary phosphors used in creation of PS, the efficiency of radiative transfer does not exceed 50 - 60%, while the light yield of PS relative to the light yield of anthracene is 60 - 65%. Consequently, increasing efficiency of irradiative energy transfer to 100 % will increase the luminous yield of PS up to 100 - 110% relative to anthracene.

Decrease in light attenuation coefficient at wavelength corresponding to maximum in the PS fluorescence spectrum (increase in transparency) is achieved by choosing maximal ratio between the units of the primary and secondary phosphors:

$$k = \frac{N_{L1}}{N_{L2}} \tag{3}$$

Choice of maximal ratio is due to need to minimize absorption of the secondary phosphor at a wavelength corresponding to maximum of its fluorescence. Increase in the ratio leads to an increase in transparency and, at the same time, to decrease in efficiency of nonradiative energy transfer as a result of an increase in distance between units of the primary and secondary phosphors. To avoid this, the phosphors are distributed in

$$n_{L1} = \frac{N_{L1}}{k} \tag{4}$$

the macromolecule in such way that the distance between the centers of each unit of one secondary phosphor and group consisting of units of the primary phosphor is minimal, as shown in Fig. 3.



Fig. 3. Schemes of structure of branched nanoscale macromolecules

On Fig. 3 schematically shows structure of branched nanosized macromolecules with different ratios of number of units of the primary and secondary phosphors:

$$GI: \frac{N_{L1}}{N_{L2}} = \frac{12}{1} , \qquad (5)$$

$$GII: \frac{N_{L1}}{N_{L2}} = \frac{4}{1} \quad , \tag{6}$$

$$GIII: \frac{N_{L1}}{N_{L2}} = \frac{6}{3}.$$
 (7)

Index (a) marks the primary phosphor links, and index (b) marks the secondary phosphor links. Nanostructured filler – nanosized branched macromolecules consisting of links corresponding to

primary and secondary phosphors are produced using one of the reactions of organometallic synthesis (Suzuki, Kumada, Stille, Ullmann), as well as the interaction of chlorine- or alkoxysilanes with lithium- or magnesiumorganic derivatives.

Scintillator blank is obtained by mixing the nanostructured filler with polymer selected as a base in twin-screw mixer with return channel (at 180°C temperature and screw rotation frequency of 600 rpm). Further, pressing (at temperature of 180°C) get samples of PS with diameter of 25 mm, height of 10 mm. Surface of the samples is carefully polished.

Measurement of light output of PS carried out on calibrated amplitude spectrometer. Duration of scintillation is measured with oscilloscope with bandwidth of 1000 MHz. Value of light attenuation coefficient at wavelength corresponding to maximum intrinsic fluorescence is determined using a spectrophotometer.

#### 4. New nanopolymer materials and nanoparticle dispersion methods

Nanoparticles such as quantum dots (QDs) are of high interest for use in transformative devices. They can, for example, serve as an non-organic luminophore in transformation of blue light into other colors with narrow bandwidth and adjustable frequency of radiation using QDs, in order to be able to obtain high quality white color.

However, introduction of nanoparticles in many types of polymers leads to clumping nanoparticles. Therefore, relevant technical task is to create an alternative system of nanoparticle – polymer, especially a polymer system with quantum dots, in which causes of aggregation of nanoparticles would be eliminated, and polymer matrices had high values of glass transition temperature Tg to 150 - 200 ° C, for example, photochemically stable silicon-containing polymers.

Silicon-containing polymers can have much higher thermal stability and acceptable light transmission ratio than other organic polymers. However, QDs with conventional surface protective molecules are not dispersed in silicones and show aggregation resulting in reduced light transmission.

Thus, there is a problem of mixing nanoparticles into silicon-containing polymers. Phase separation between nanoparticles and polymers causes QDs agglomeration and drastically reduces quantum yield and light transmission ratio through nanoparticle/polymer mixtures.

In [11], technical solution was proposed that makes it possible to obtain well-dispersed QDs layers in silicones using protective molecules that can themselves attach to the surface of QDs. Group of protective molecules compatible with silicones has been developed. These protective molecules can easily coat the QDs and ensure the formation of uniform QDs/silicone polymer composites. These protective molecules are made up of two parts; one part connects to outer unprotected atoms on crystal surface of the QDs, and other part is compatible with silicone matrix. By surface modification, the nanoparticles can be easily mixed with the silicone matrix. New matrices can form very thin transparent films. The films have high thermal stability and can be used as new light-converting phosphors. With choice of matching silicone polymers and surface protective molecules in any given silicone matrix. The formed thin films of the nanoparticle/silicone matrix have high light transmission coefficient and are not inferior in stability compared to nanoparticles in purely inorganic matrices.

This kind of nanopolymer produced by this method can be used either alone or in a polymer product, and it seems possible to provide luminescence with high quantum yield and stability. In addition, the polymer can be stable with respect to effects of temperature and photochemistry. In this method, nanoparticles can be dispersed uniformly in the polymer, and agglomeration processes can be eliminated.

Such luminescent materials can also be used successfully in various devices. The present technical solution can be applied to devices including light sources and light converters designed to convert part of radiation of light source into light of converter, which is a solid polymer obtained according to the proposed technical solution. Luminescent nanoparticles can be, for example, include compounds of semiconductor nanoparticles of groups II – VI selected from group consisting of CdS, CdSe, CdTe, ZnS, ZnSe, ZnTe, HgS, HgSe, HgTe, CdSeS, CdSeTe, CdSTe, ZnSeS, ZnSeTe, ZnSTe, HgSeS, HgSeTe, HgSTe, CdZnS, CdZnSe, CdZnTe, CdHgS, CdHgSe, CdHgTe, HgZnS, HgZnSe, HgZnTe, CdZnSeS, CdZnSeTe, CdZnSTe, CdHgSeS, CdHgSeTe, CdHgSTe, HgZnSeS, HgZnSeTe and HgZnSTe.

In another variant of applications of luminescent nanoparticles can be, for example, compounds of semiconductor nanoparticles of III-V groups, selected from group consisting of GaN, GaP, GaAs, A1N, A1P, AlAs, InN, InP, InAs, GaNP, GaNAs, GaPAs, A1NP, AlNAs, AlPAs, InNP, InNAs, InPAs, GaAlNAs, GaAlNAs, GaAlPAs, GaInNAs, GaInPAs, InAlNAs and InAlPAs.

Thus, the silicone nanocomposite to the proposed technical solution is able to transmit light radiation with high efficiency with wavelength selected from the range of 420-750 nm at temperatures up to  $100^{\circ}C - 200^{\circ}C$ .

## Conclusions

Analysis of some currently existing polymer and nanopolymer optical systems and their applications showed that complexity of structures and micro-dimensions of such optical systems for their wide application requires new easy-to-use and inexpensive optical materials. New types of polymer materials are replacing the traditional optical material (glass). In addition to fact that they make it possible to obtain structures of micro and nanosizes, there is already real opportunity to select their physical and optical properties – index of refraction, optical uniformity, light transmission, light scattering, stiffness and other ones, depending on specific task. Nanostructured optical polymer materials are increasingly being used to further improve and enhance efficiency of not only optical devices, but also products of scintillation technology, lighting engineering, photovoltaic, as well as applications in other fields of science and technology.

In this regard, the research aimed at finding new approaches to creation of nanocomposites on the basis of wide range of polymers and nanoparticles becomes relevant.

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Received 07.06.2023

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