

## Photocurable polymer composite materials with an improved combination of strength and service properties

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This scientific paper delves into a new method of the photochemical cross-linkage of the encapsulating and adhesive materials that are based on oligoester acrylates with addition of the epoxy resin (oligomer) ER-20. The objects of research were oligocarbonate methacrylate OCM-2, epoxy oligomer ER-20 and the synthetic aluminosilicate (zeolite) filler. The additives for the photoinitiation were benzoin and its ethers. Studies the strength-, service and thermal properties of polymer composite materials (PCM) and the behavior of structural properties under different conditions. The obtained data allowed us to minimize the shrinkage of PCMs, increase their thermal oxidation stability and speed up the cross-linkage process. It was established that the combination of oligocarbonate methacrylate OCM-2 and the epoxy oligomer ER-20 has a significant effect both on the PCM cross-linkage process behavior and on its strength and service properties. The PCM composition was suggested for the encapsulation and moisture protection of the modules and assemblies of radio electronic equipment. A comparative evaluation of the suggested method of cross-linkage with the thermochemical curing allowed us to define the advantage of the photochemical method used for the material cross-linkage.

**Keywords:** photochemical cross-linkage, oligocarbonate methacrylate, epoxy oligomer, internal stresses and shrinkage.

**Фотомобильні полімерні складові матеріали з вдосконаленою комбінацією міцності і сервісних властивостей.** *В.Л.Авраменко, Л.Ф.Підгорна, О.Х.Карандашов*

Розглянуто новий метод фотохімічного структурування герметизуючих і клеючих матеріалів на основі олігоєфіракрилатів ОКМ-2 з добавкою епоксидного олігомеру ЕД-20. Об'єкти дослідження є олігокарбонатметакрилат ОКМ-2, епоксидний олігомер ЕД-20, наповнювач синтетичний алюмосилікат (цеоліт). Добавками для фотоініціації є бензоїн і його ефіри. Досліджено міцнісні, експлуатаційні, термічні властивості ПКМ і протікання процесів структурування у різних умовах. Визначено внутрішні напруги, які виникають у процесі структурування матеріалу. Встановлено, що поєднання олігокарбонатметакрилату ОКМ-2 і епоксидного олігомеру ЕД-20 істотно впливає як на перебіг процесу структурування ПКМ, так і на комплекс його міцності і експлуатаційних властивостей. Рекомендовано склад ПКМ для герметизації і вологозахисту вузлів і блоків радіоелектронної апаратури. Порівняльна оцінка запропонованого методу структурування з термохімічним отвердінням дозволила визначити перевагу фотохімічного способу структурування.

Рассмотрен новый метод фотохимического структурирования герметизирующих и клеющих материалов на основе олигоэфиракрилата ОКМ-2 с добавкой эпоксидного олигомера ЭД-20. Объектами исследования являются олигокарбонатметакрилат ОКМ-2, эпоксидный олигомер ЭД-20, наполнитель синтетический алюмосиликат (цеолит). Добавками для фотоиницирования являются бензоин и его эфиры. Исследованы прочностные, эксплуатаци-

онные, термические свойства ПКМ и протекание процессов структурирования в различных условиях. Определены внутренние напряжения, которые возникают в процессе структурирования материала. Установлено, что совмещение олигокарбонатметакрилата ОКМ-2 и эпоксидного олигомера ЭД-20 существенно влияет как на протекание процесса структурирования ПКМ, так и на комплекс его прочностных и эксплуатационных свойств. Рекомендован состав ПКМ для герметизации и влагозащиты узлов и блоков радиоэлектронной аппаратуры. Сравнительная оценка предложенного метода структурирования с термохимическим отверждением позволила определить преимущество фотохимического способа структурирования.

### 1. Introduction

The development of the basic principles for the creation of the functional photochemically cross-linked PCMs is considered to be an important achievement in the field of the chemistry of polymer material science over the last years.

The hands-on application of the photochemical technology allowed us to solve many important engineering problems; in particular we managed to create photopolymer printing plates, sealing adhesive materials with elevated adhesion, structural PCMs with specific properties, i.e. high adhesion, radiation stability, etc.

Oligoether acrylates, epoxy acrylic-, polyether and other oligomers and also the monomers of a different chemical nature and special additives [1, 2] are used as the initial components of photocured systems

General instrument-making industry and radio electronic equipment production are considered to be special branches for a wide application of PCMs and a high adhesion, a low shrinkage, a low level of residual stresses and a high curing rate under mass production conditions including a high level of moisture protection are of great importance for these branches. The use of such PCMs allows for a considerable reduction in the volume and mass of produced articles and extension of the range of permissible external effects. The priming of the assemblies and modules of the electronic equipment (EE) with photochemically cured compositions is a constructive approach to the miniaturization and creation of the unpacked moisture-resistant bonded structures.

The use of micromodules that have a high potential for the portable radio electronic equipment (REE), rockets, artificial Earth satellites allows for a considerable reduction in the sizes of the devices and equipment in comparison to those adopted for the traditional (compact) mounting [2].

A new field for the application of PCMs is microminiature holographic storage devices for computing tools. A maximum capacity of the devices that use polymer mate-

rials is higher by a factor of 2 to 3 in comparison to the magnetic tape storage and the average access time is lower by a factor of 7 [2, 3].

Electronic equipment parameters depend to a great extent on the mechanical properties of polymer materials, their strength at different loadings, including the vibration strength, hardness, elasticity, etc.

For the structures that include magnetically soft materials, a low shrinkage of encapsulating PCMs is of great importance.

The main method of encapsulation is the priming (formation) of monolithic or molded insulation.

Epoxy compounds, organic silicon polymers, polyurethanes and polyacrylates are used most frequently for these purposes.

The compounds based on the chemically reactive oligomers (CRO) of a photochemical cross-linkage (curing) are of special interest for the encapsulation and moisture protection of the assemblies and modules of different devices and these demand additional studies [4–6].

The purposes of this scientific paper are as follows: first, we were to create photochemically cured PCMs that are based on oligoether acrylates, epoxy oligomer with different initiators and additives, study the influence of different factors and system components on the process of photopolymerization and analyze many technological, physical-&-mechanical and service properties of developed PCMs and their behavior in operative conditions.

### 2. Experimental

To use the polymer skeleton of composition for the formation of the photochemically cured PCM a number of commercial oligoether acrylates was provided for the selection of appropriate oligomers. When selecting the oligomers, we took into account the dependence of the properties of polymers that are based on the given group of materials on the structure of dibasic acids and glycols or polyatomic alcohols, the number of the functional groups of hy-

droxyl- and carboxyl-containing reagents, different additives and their ratios.

However, oligoether acrylates attract special attention due to their ability to get cured photochemically.

PCM materials were developed taking into consideration the requirements set to the service conditions of the articles made of PCM and the possibility of their formation by photochemical curing. Such oligomer compositions as OCM-2, TGM-3; MGF-9 and MDF-2 were tested for their use as a PCM skeleton. These possess an easily controlled set of the properties, a high reactivity and good compatibility [7, 8].

Domestically produced lamps LUF-80 and LUF-40 were used as a source of UV-radiation.

The epoxy oligomer ER-20, the synthetic aluminosilicate filler (zeolite), different photoinitiators and photosensitizers were studied as modifying additives.

The photopolymerizing composition was prepared in the following manner: all system components were weighted, the mixture was mixed by the magnetic mixer or the paddle at room temperature at 100 to 150 rpm during 0.5 to 1.5 h depending on the mixture volume. After the mixing the mixture was settled during one hour to remove air bubbles, and then it was poured into the moulds and exposed to the action of the bank of luminescent lamps of LUF-80 and LUF-40 types for about 10 to 20 min at a lamp-to-specimen distance of 8 to 15 cm. The obtained cured compositions were studied paying attention to many parameters and the degree of cure (cross-linkage) of the PCM was defined using Soxhlet apparatus for the extraction in acetone according to the known laboratory technique.

The IR-spectroscopy was used to study the process kinetics of the photochemical curing of the multicomponent system and also study in detail the photopolymerization process behavior.

The participation of functional groups in the photopolymerization reaction was defined by a change in the intensity, and also by disappearance of appropriate absorption bands and appearance of new absorption bands.

The spectra were taken using the self-acting double-beam spectrophotometer IR-20 with NaCl and LiF prisms in the domains of 700–3800  $\text{cm}^{-1}$ . The specimens were prepared in the form of the film of 10  $\mu\text{m}$  thick in the demountable liquid cell with the windows made of NaCl [9].

Residual stresses were measured by the tensometric method using the DTB-5 device. To measure the contact pressure we used the model in the form of a thin-wall cylinder with wire sensors pasted on its inner surface to measure axial and circumferential strains.

The cylinder with affixed strain sensors was placed into the split mould. Afterwards, the model was filled with the test compound so as to form a layer of 6 mm thick around the cylinder.

The residual stresses that arise during the compound setting result in the cylinder strain. The cylinder strain and, as a consequence, the effort that gives rise to it were defined by the value of a change in the resistance of strain sensors.

The contact pressure was calculated using the computation formula for the thin-wall cylinder [10].

$$P = \varepsilon_0 E_1 \Delta / r(1 - \mu/r),$$

where  $P$  is the contact pressure, MPa;  $r$  is the mean radius of cylinder, mm;  $E_1$  is the cylinder material elasticity modulus, MPa;  $\varepsilon_0$  is the relative circumferential strain;  $\Delta$  is the cylinder wall thickness, mm;  $\mu$  is the Poisson coefficient. The thermal stability of UV-cured polymers was measured using the methods of differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) on the derivatograph of a Paulik-Paulik-Erdey system [11].

A weighted quantity of the fine-grained polymer in the amount of 200 to 300 mg was heated at a rate of 5°C/min to achieve the temperature of 500°C.

The ultimate flexural strength, the impact elasticity, the water absorption, the PCM specimen shrinkage and the relative composition viscosity were measured using appropriate standards and known laboratory methods. The adhesion strength of coatings was measured using the method of cross-cuts [12]. The UV-spectra of the absorption of photoinitiators and photosensibilizers were analyzed using the UV-spectrophotometer SF-56.

### 3. Results and discussion

The studied oligomers have an absorption edge shifted to the long-wave domain of the spectrum. Oligocarbonate methacrylate OCM-2 ( $\alpha$ ,  $\omega$ -bis-methacrylosiloxyethylene-oxycarbonyloxi)-ethylene oxietylene showed the highest photochemical activity of all the

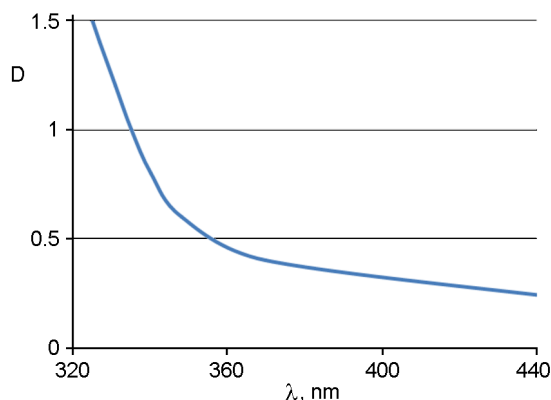


Fig. 1. The absorption spectrum of oligocarbonate methacrylate OCM-2 in the UV-domain.

tested oligomers and its absorption spectrum in the UV-domain is given in Fig. 1 and it is, evidently, conditioned by the specific features of the structure of carbonyl group.

From the photochemical point of view the carbonyl group  $C=O$  is one of the most important chromophores. Its great advantage consists in that this group is photochemically active but it is rather stable thermally. Carbonyl absorbs UV light in the near UV-domain and it can thus be excited selectively in the presence of a great number of other chemical groups.

In addition, OCM-2 has a minimum wavelength limit of the transmission in the absorption spectrum in comparison to other oligoether acrylates.

Hence, to carry out follow-up studies we selected the oligomer OCM-2 that defines a high level of such properties of the cured material as water resistance, hardness, bent strength, low shrinkage, etc.

It turned out that the addition of chromophore groups to the system in the form photoinitiators and photosensibilizers results in an increased photoactivity of OCM-2 and, as a result, the cross-linkage rate is also increased.

We tested a great number of the different classes of compounds, in particular amides, phenons, etc to check them as the photosensibilizers and photoinitiators.

The best results were obtained using benzoin photoinitiators and its derivatives. i.e. benzoin methyl ether and ketal. Fig. 2 gives the UV-spectra of mentioned photosensibilizers and photoinitiators.

We carried out a series of the experiments on the introduction of thermoinitiators to the composition for a more total activation of the double bonds of oligomer that failed to enter into reaction. Benzoyl

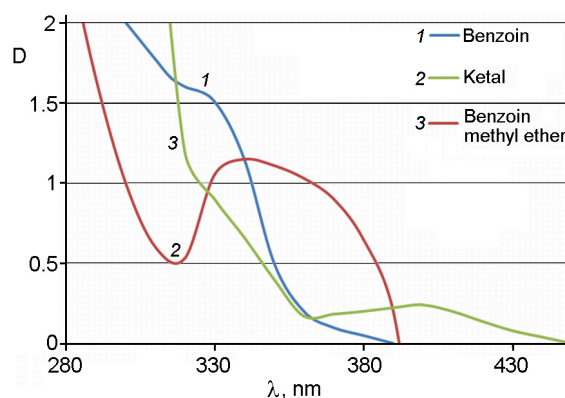


Fig. 2. UV-spectra of the absorption of photoinitiators and photosensibilizers.

peroxide, cumene peroxide, tertiary butyl maleate and peroxide urea formaldehyde resin have been investigated.

The studies of the influence of peroxide compounds by the example of the use of benzoyl peroxide allowed us to establish that it is more reasonable to introduce particularly benzoyl peroxide that allows for an enhanced curing degree and improved strength performances.

Thermal treatment of the composition at  $90^{\circ}C$  during 60 min turned out to be efficient. At the same time, it was established that the addition of thermoinitiator results in an increased induction period, a decreased photopolymerization rate and reduced photochemical in-depth transformations. Evidently, it is related to the filtering action of benzoyl peroxide that contains the residues of carboxyl groups conjugated with benzene nucleus.

A change in the quantity of added photosensibilizer (benzoin) was studied by a change in the value of gel-fraction (Fig. 3). By selecting the promoters and adding them to the composition formulation we expected to change the system photosensitivity and also the rate of photochemical reaction and the set of physical and mechanical properties.

We studied the influence of different amines and the amines that contain hydroxyl groups in their structure evoke special interest.

The analysis of obtained data allows us to draw a conclusion that amine derivatives have a significant effect on the light sensitivity of chemically cured compositions.

We can assume that the interaction of amine derivative and photoinitiator results in a partial neutralization of the number and options of free radicals both of amine and benzoin types in the system in the pres-

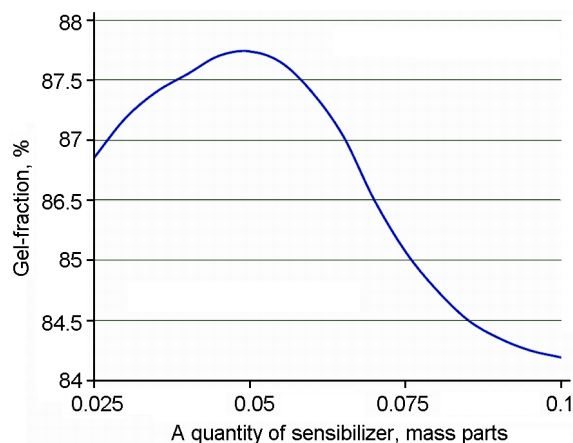


Fig. 3. Dependence of the output of gel-fraction on the quantity of sensitizer.

ence of air oxygen. In the absence of the synergetic action of amines the activity of photosensibilizers is slightly decreased.

The best results were obtained for the introduction of the mixture of diethylene triamine and imidazole to the composition in the ratio of 70:30 in the amount within 2.5 %.

The dependence of the output of the gel-fraction of the chemically-cured PCM (OCM-2-99. 95 %, ketal – 0.05 %) on the type and amount of the promoter is given in Table 1. The epoxy oligomer ER-20 served as a PCM modifying additive. An optimal quantity of this additive was equal to 10 wt. %.

The synthetic aluminosilicate (zeolite) with its general formula of  $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 1.8\text{H}_2\text{O}$  was tested by us as a filler.

Addition of the filler allowed for an increase in the viscosity of the poured composition and the photochemical transformation depth (up to 3 wt. %) was also slightly increased. It can be explained by an increase in the mobility of the polymer binder for the mean and extensive levels of transformation due to the retarded intermolecular interaction and the collapse of associated formations.

The main requirements to the developed compositions were a decrease (minimization) in the level of residual stresses that arise

during the cross-linkage, and also an increase in the adhesive properties of PCM and a decreased shrinkage.

The tensometric method was used for the estimation of the value of residual stresses that arise during the composition cross-linkage.

This method enables the estimation of the level of residual stresses during the cross-linkage process in a wide temperature range and it is an essential advantage of it.

Since a change in the system volume due to different structural transformations during the photochemical polarization is one of the main reasons for the origination of residual stresses we studied the kinetics and relaxation of residual stresses depending on the oligomer nature, modifying additives, the filler nature and the polymerization process behavior.

The spreading of composition plays an important role in the origination of residual stresses during the encapsulation of the modules and assemblies.

The research data are given in Fig. 4 and 5.

The given diagrams show that the value and behavior of residual stresses that arise in the studied compositions are not similar.

The highest level of residual stresses is observed in the thermally cured ER-20 oligomer-based composition (Fig. 4) and it is conditioned by the formation of polymer with a rigid cross-linked structure and this polymer is incapable of significant relaxation due to the insufficient flexibility of macromolecules.

A high value of the level of residual stresses and their positive polarity result in a considerable pressure exerted by the epoxy polymer material layer on the substrate and it results in the relaxation of the adhesive interaction between the substrate and the PCM.

All the photochemically cured PCMs, including the initial OCM2-based material (Fig. 5) and the composition modified by the epoxy oligomer ER-20 have a very low level of residual stresses and a negative polarity.

Such a behavior of residual stresses is conditioned by that the addition of epoxy

Table 1. The dependences of the output of the gel-fraction of the chemically-cured composition (OCM-2-99 – 95 %, ketal – 0.05 %) on the type and amount of the promoter

Promoter	Content, wt. %	Gel-fraction output, %	Gelatinization time at daylight, min
With no promoter	–	87,40	No gelatinization
Diethylene triamine with imidazol (70:30)	2,5	82,63	20
Diethylene triamine with triethanol amine (70:30)	2,0	83,71	20

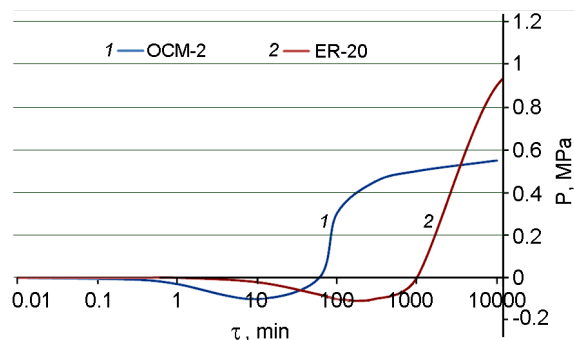


Fig. 4. Dependences of the value of residual stresses of the thermochemically cured compositions on the time of cure.

oligomer to OCM-2 results in the formation of polymer with the "snake-in-the-cell" structure that considerably suppresses shrinkage processes.

Similar effect is observed in the case of the addition of zeolite filler to the composition and as a result the conditions are created in the photochemically cured polymer under which the stress relaxation time becomes commensurable with the polymer formation time (Fig. 5).

The thermochemical cure of OCM-2 with benzoyl peroxide (Fig. 4) shows a higher level of residual stresses due to the origination of the nonuniform force field in the system and the origination of it is conditioned by the action of the temperature gradient and these internal stresses are lower by one order of magnitude in comparison to those peculiar for the PCM based on thermally-cured epoxy compositions, because OCM-2 is polymerized due to the availability of finite or regularly arranged unsaturated groups in the oligoether module. It conditions a high mobility of the circuits and, as a consequence, a possibility of the relaxation of originated residual stresses.

Hence, the research done allowed us to study the cross-linkage process of photochemically cured systems, select photochemical polarization parameters and PCM formulations with minimum residual stresses.

Due to the fact that the reliability and service-life of the polymer material-containing articles depend on many service properties and physical-mechanical properties of PCMs and an extent of the completeness of the setting process we also studied the processes of the photochemical cure of OCM-2 using IR spectra.

The initial OCM-2 spectrum shows the following absorption bands: the most inten-

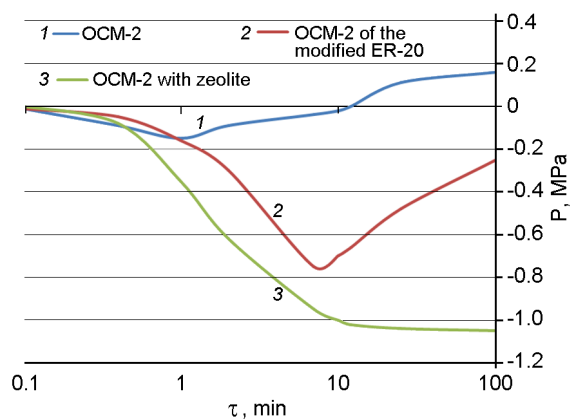


Fig. 5. Dependences of the value of residual stresses of the UV-cured compositions on the time of cure.

sive bands that are observed at  $1728\text{ cm}^{-1}$  correspond to the valent vibrations of  $\text{CH}_2$ -groups, the absorption band with a maximum of  $1635\text{ cm}^{-1}$  corresponds to the valent vibrations of  $>\text{C}=\text{C}-$ bonds and the absorption band with a maximum of  $1448\text{ cm}^{-1}$  corresponds to the strain vibrations of  $\text{CH}_2$ -group.

A comparison of the IR-spectrum of the initial OCM-2 and that of the obtained PCM shows that the band intensity at  $1448\text{ cm}^{-1}$  endures no changes during the photo-cure process. The band is subjected to the most intensive changes at  $1638\text{ cm}^{-1}$ , especially during the first period of the photo-polymerization process and afterwards it remains actually constant. Its intensity remains unchanged even after a long-time exposure. Evidently, it is conditioned by the availability of unreacted  $>\text{C}=\text{C}-$ bonds in the photo-cured material and on the termination of radiation these bonds condition the "post-effect" behavior, i.e. the possibility of a further material cross-linkage using spatially-screened radicals and nonreacted double bonds. These data are correlated with the data obtained during the measurement of residual stresses that were described earlier.

It is known that the stability of PCM properties is achieved at a maximum depth of the oligomer cross-linkage.

Since the developed PCMs and the articles made of them are subjected to the action of air oxygen and the temperature difference during their operation it was of great interest to study the process of the thermooxidation destruction of developed compositions and also the stability of physical and mechanical properties. For this purpose we used DTA and TGA methods.

The TG data show the difference in the behavior of the polymers depending on the added modifying additives and the polymerization method.

The reactions that progress during the thermooxidation destruction are accompanied by the mass loss and many thermal effects. The analysis of TG curves (Fig. 6) shows that the temperature of the onset in the loss of mass of tested polymers varies in the range of 60 to 210°C. The initial specimen of the OCM-2 with the sensitizer has the mass loss onset temperature of 100°C. For the composition with the epoxy oligomer this temperature is equal to 110°C, and it is indicative of the formation of a more cross-linked and thermally stable polymer.

The 50 % composition disintegration temperature varies in the range of 260 to 370°C.

The most efficient additive from the standpoint of a rise in the decay temperature is considered to be the epoxy oligomer ED-20 (its 50 % disintegration temperature is equal to 370°C), while for initial composition it is equal to 270°C. A total disintegration of all the tested compositions is observed in the temperature range of 370 to 470°C, and for the zeolite-containing com-

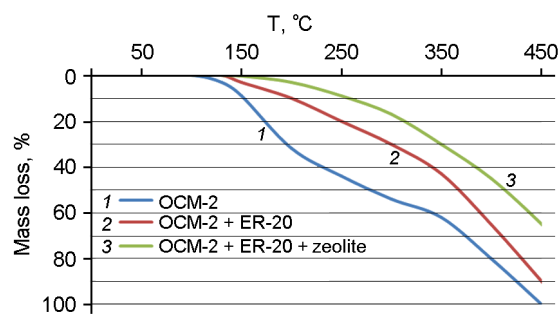


Fig. 6. Thermogravimetric curves for photochemically cured polymers.

positions it is equal to 500°C. Proceeding from the obtained data we can draw a conclusion that the OCM-2 composition modified by the epoxy oligomer is the most important as for the thermal stability.

Table 2 gives physical-mechanical and service properties of cured compositions.

Based on the research data obtained for the photocured polymer composite materials given in Tables 2 and 3 we can state that compositions 1, 2 and 3 are optimal from the standpoint of photochemical technology and these were used as a basis for the formation of the specimens with the unlimited

Table 2. Physical-mechanical and service properties of the photochemically cured compositions based on the OCM-2 and the epoxy oligomer ER-20

Indices of the properties	Composition			
	1	2	3	4
Density, kg/m <sup>3</sup>	1399	1337	1321	1604
VS-4 viscosity, s, at 20°C	46	44	70	100
Vital capacity, h, at 20°C	Unlimited	Unlimited	Unlimited	3
Water absorption, %, 1 day/30 days	-0.78/0.1	-0.75/0.1	-0.10/0.08	0.035/0.112
Shrinkage, %, 1 day/30 days	0.076/0.155	0.068/0.15	0.035/0.112	0.46/0.64
Impact elasticity, kJ/m <sup>2</sup> , 1 day/30 days	7.0/8.4	14.1/14.6	15.6/16.1	22.4/23.0
Ultimate flexural strength, MPa, 1 day/30 days	65/71	60/67	90/94	110/225
Adhesion, points	1.0	1.0	1.0	1.0

Table 3. Remark: the composition formulation, mass parts

Component	Content			
	Composition 1	Composition 2	Composition 3	Composition 4
OCM-2	85.95	90.95	90	6
Benzoin	0.05	0.05	1.0	1.0
ER-20	10	5	5	88
Zeolite	3	3	3	3
Benzoyl peroxide	1	1	1	2

life capacity, a low water absorption and shrinkage and rather high strength performances. However, many fields of the application of the articles that are subject to the encapsulation and moisture insulation require on the contrary a low life capacity and an increased strength (printed boards that contain different non-ferrous metals) and in these cases it is recommended to use composition 4 that will be optimal for such conditions.

#### 4. Conclusions

Photochemically cured polymer composite materials were studied to define their appropriateness for the priming, encapsulation and moisture protection of the articles of radio electronic equipment.

It was established that a decrease in the shrinkage and residual stresses of the photochemically cured PCM has a positive effect on the set of physical-&-chemical and service properties of PCMs and provides a reliable compound operation under elevated temperatures.

It was established that a combination of oligocarbonate methacrylate and the epoxy oligomer ER-20 has a significant effect on the PCM cross-linkage process and also on the set of its strength and service properties.

The research done showed that the developed composition formulations can be recommended for the encapsulation and moisture protection of the modules and assemblies of the electronic equipment.

The comparative estimation of the data obtained for photo- and chemically cured

PCMs has been carried out. It was established that photochemically cured PCMs have a higher level of service and process performances and these are suitable for the use in different electronic devices.

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