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Введение



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ТЕХНОЛОГИЯ УЛЬТРАФИОЛЕТОВОГО ОТВЕРЖДЕНИЯ ДЛЯ ПОЛУЧЕНИЯ ПЛЕНОК И ИЗДЕЛИЙ

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В статье представлены разработки в области создания новых UV-отверждаемых и LED-отверждаемых материалов различного назначения членом научно-педагогической школы «Технологии функциональных композитных материалов» (СПбГИКИТ, Санкт-Петербург, руководитель – д.т.н., профессор О.Э. Бабкин).

Ключевые слова: УФ-отверждение, LED-отверждение, фотополимерные материалы, технология, покрытия.

UV CURING TECHNOLOGY FOR PRODUCTION OF FILMS AND OTHER GOODS

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The article presents developments in the field of creating new UV-curable and LED-curable materials for various purposes of members of the scientific and pedagogical school «Technologies of Functional Composite Materials» (St. Petersburg State University of Cinema and Television, St. Petersburg, the head is Doctor of Technical Sciences, Professor O.E. Babkin).

Key words: UV-curing, LED-curing, photopolymer materials, technology, coatings.

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Preamble

The technology of photoinitiated polymerization, known since ancient Egypt, is widely used in various industries, both in Russia and abroad. For example, since the 60s. XX century ultraviolet curing (UV-curing) is used in the paint industry of Europe: back in 1967, a progressive industrial technology for curing by that time with ultraviolet radiation (UV radiation) of unsaturated polyesters (the most common materials at that time) was proposed [1]. The main advantage of the process then was a significant reduction in the time for obtaining the finished coating. In addition to the time gain, a significant advantage of the process was the reduction of production space due to the compactness of the equipment. In total, the availability on the market of raw materials for the synthesis of unsaturated polyester resins, the development of a technology for the industrial production of photoinitiators (at the initial stage, benzoin esters were used as photoinitiators) and the presence on the market of industrial sources of UV radiation (initially, low-pressure mercury lamps with a power of 0,5-1,0 W/cm; and since 1970 - high-pressure mercury lamps with a capacity of 20-30 W/cm) contributed to the rapid introduction of UV-cured materials on an industrial scale throughout Europe by the end of the

1970s. The first curing technologies for filled photopolymerizable compositions (FPKs) were developed. This was facilitated by the introduction of new UV sources with increased radiation intensity and the development of new photoinitiators based on thioxanthone derivatives, used in combination with synergetics (tertiary amines). Thus, a technology appeared for curing FPK containing titanium dioxide. Another effective photoinitiator system was methylthioxantone with a synergist methyldiethanolamine - these photoinitiators were proposed for the curing of unsaturated acrylate mixtures with an active diluent. Fully pigmented white UV-curing materials were prepared using acylphosphine oxides as the photoinitiator (2,4,6-trimethyl-benzoyl-diphenyl-phosphine oxide, TMPO, is the most common). The main directions of development of UV-curing technology until the end of the twentieth century, remained improving the formulation of the FPK and obtaining new photoinitiators, as well as improving the technique of emitted ultraviolet radiation. At the present stage, UV-curing technologies are developing in the direction of using light emitting diode (LED) emitters, characterized by a narrow emission spectrum. Accordingly, such emitters require the development of compositions with a specific composition and the use of specific photoinitiating systems.

Theoretical section

Today, most companies prefer UV-curable materials due to their high quality, economy, environmental friendliness and manufacturability: UV-curing industrial coatings are one of the main trends in the modern development of the paint industry. For example, in Russia the technology has already been introduced at a number of enterprises – UV-curable materials are used to create anti-corrosion coatings for metal products at the Sinarsky Pipe Plant, Volzhsky Pipe Plant, Vyksa Pipe Plant, and Taganrog Metallurgical Plant (Tagmet PJSC Research in the field of UV-polymerizable and LED-polymerizable materials is actively engaged in the scientific school «Technologies of functional composite materials» (ScSh-103-62.2016.3 of the register of leading scientific and scientific-pedagogical schools of St. Petersburg), and the implementation of the developments is carried out at the «S&H enterprise Technology» (St. Petersburg).

Fundamental and applied research is being carried out in the field of obtaining new UV-curable materials for anticorrosion protection, for moisture protection of radio electronic equipment units, for the production of decorative coatings, flexography, protective holography, cosmetology materials, for stereolithography (3D-printing), materials for coloring optical fiber cables, composite materials for repair purposes [2-17]. «S&H Technology» is an innovative enterprise in St. Petersburg that aims to produce high-tech paints and varnishes, including UV-curing compositions. The production facilities of the company allow producing up to 120 tons / month of high-tech paints and varnishes. Table.1 presents some characteristics of polymer coatings based on UV-curable coatings manufactured by «S&H Technology».

Table 1. Characteristics of UV curable coatings manufactured by «S&H Technology»

Name of indicator	Indicator value		
	Clear coat	Primer	Enamel
Adhesion, score, no more	1	1	1
Film strength upon impact with U-1 device, cm, not less than	40	50	40
The hardness of the film according to TML-2124 method A, cu, not less	0,5	0,5	0,45
Flexural film mm, no more	1	1	1
Fineness, mkm, no more	-	10	10
Coating Resistance - to static effects of water at a temperature of $20 \pm 0,5^{\circ}\text{C}$, h, at least	96	400	-
Resistance of coating to 5% salt fog, h, not less	240	400	-

A promising area of application of UV-curing technology is the painting of coil metal by Coil Coating. The process of coil-coating occurs at very high speeds, while it is possible to obtain painted products from rolled metal without destroying the paint layer. Coatings obtained by the method of coil-coating have increased corrosion resistance, which makes it possible for many years of operation without any negative changes in the structure of the coating. The use of UV-curable materials can compete with traditional coiling, due to several advantages over other types of paints and varnishes:

- high curing rate (within 0,01-0,5 s) and coating formation with maximum protective and decorative properties;

- UV-curing units are more compact than thermal curing equipment. An important issue in the implementation of the technology is the pigmentation of the composition. Pigments can absorb the energy of UV radiation by photoinitiators, which will affect the concentration of free radicals and lead to a decrease in the curing rate [11, 12, 18]. This is due to the fact that most commonly used inorganic and organic pigments absorb UV radiation in the same spectral region as photoinitiators. Using approaches to the preparation of pigmented FPKs described in [11, 18], it was possible to produce colored coatings of UV-curing according to the RAL catalog.

Results of the authors' experiments and their discussion

The UV-curing composition has been widely used in the production of optical fiber, where the speed of applying a protective coating reaches 1600 m/min. In the production of optical fiber, both protective UV curing varnishes and pigmented varnishes – UV-curing inks necessary for marking optical fibers – are used [19-21]. «S&H Technology» produces UV-curing varnishes for dyeing optical fibers and inks for marking them. The production of optical fiber cables is currently being launched in the Russian Federation (Saransk, AO «Optical Fiber Systems»), and given the current import substitution strategy in the manufacturing sector of the economy, the domestic production of optical cable has every reason for development. A feature of obtaining a two-layer sheath on a fiber is the use of technology «wet on wet», and the curing process itself must occur in a nitrogen environment. Coatings using two-layer technology were applied to a quartz glass thread on an industrial line of Optic Fiber Systems CJSC at a speed of 1500 m/min at a temperature of 50°C . The characteristics of the primer and topcoat varnish are given in tabl. 2.

Table 2. Properties of the primer and topcoat varnish

Name of indicator	Indicator value	
	S&H Techno Primer 056	UV OF Lac 155 Clear coat
FPK characteristics		
Dynamic viscosity, MPa · s Brookfield RVDV-E, sp. 4/20 rpm at 25°C	12120	9110
Dynamic viscosity, MPa · s Brookfield RVDV-E, sp. 4/20 rpm, at 50°C	1936	1012
Refractive index of liquid FPK, at 20°C	1,5550	1,5202
Properties		
Appearance of film	forms a transparent uniform coating, without mechanical impurities	forms a transparent uniform coating, without mechanical impurities
Colour	clear	clear
The hardness of the film according to TML-2124 method A, cu, not less	-	0,48
Relative elongation (elasticity), %, not less than	50,7	3,6
Tensile Strength, MPa	0,7	39,0
The refractive index of the cured coating, at 20°C	1,6230	1,5180

Fig.1 shows microphotograph of section of optical fiber obtained with a TESCAN VEGA3 SBH scanning electron microscope at the SPbGTI engineering center (TU): in the figure, the primer and coating varnish layers are distinguishable and their thicknesses are marked.

To assess the change in the mechanical characteristics of the coating, we used the dynamic mechanical analysis (DMA) method, which allows us to evaluate the dynamics of changes in parameters over a wide temperature range. The study was performed on a DMA / SDTA 861e Mettlen Toledo instrument. The experiment was carried out at a frequency of 1 Hz in the temperature range from -150 °C to +100 °C with a heating rate (and preliminary cooling) of 2 °C/min. Fig. 2 shows the DMA curves for the primer. The dropping curves correspond to the elastic modulus curves (for two parallel soil samples 1 and 1'). The determined coordinates of the inflection points of the elastic modulus (E') curve show the start and end temperatures of the relaxation transition: -16,12 °C and -36,75 °C, respectively. Fig. 2 also shows the experimental data of the loss modulus (E'') for the same samples (curves with a maximum at -34,54 °C and -24,60 °C, respectively, for two parallel samples) and simulated tangent curves angle of mechanical losses according to the calculated data for the ratio E''/E' .

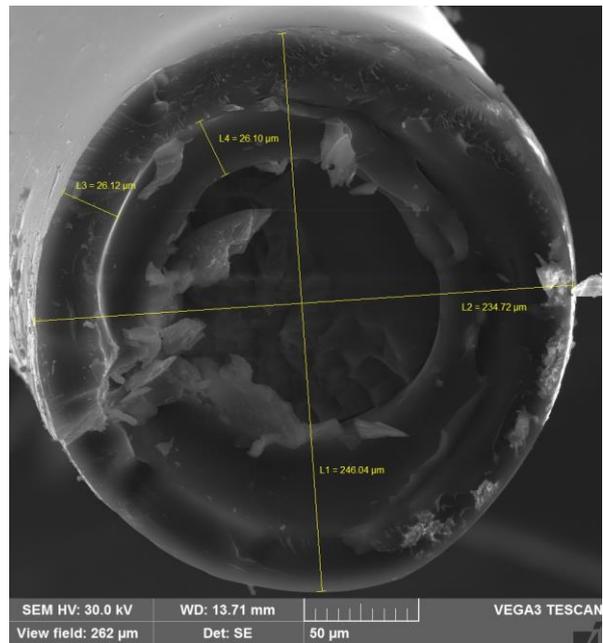


Fig. 1. CUT of optical fiber with a two-layer protective coating

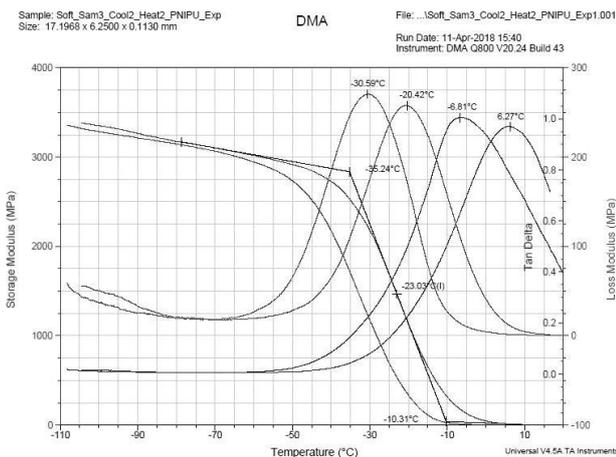


Fig. 2. DMA curves of primer coating

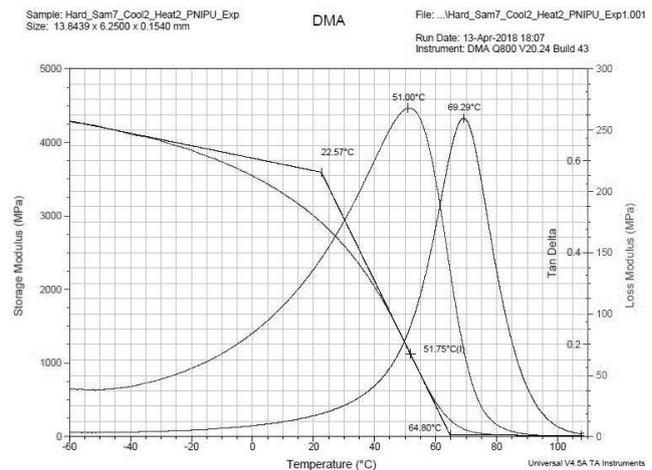


Fig. 3. DMA curves of varnish

Fig.3 shows the DMA curves of the varnish coating. Similarly to Fig. 2, the falling curve in Fig. 3 corresponds to the elastic modulus curve. The experimental value of the transition temperature, determined in Fig. 3, corresponds to +51,75 °C.

Conclusion. The experimental studies showed the dependence of the working temperature range, on the glass transition temperature to the temperature of transition to the viscoelastic state, on the formulation of the UV curing compositions.

Since this indicator is decisive for a number of production situations, the choice of copolymerizable ingredients of compositions is highly significant.

In the case of UV-curing protective coatings by optical fibers, it is worth considering the function of the coating (elastic primer or non-abrasive hard varnish), and, accordingly, choose active thinners with low, below -30°C (for primer formulations) or high, above + 50°C (for var-

nish formulations) with the intrinsic glass transition temperature of the homopolymer.

Currently, UV-curable coatings are used for painting plastics, composites, building panels.

A special niche of photopolymerizable coatings is occupied by products for moisture protection of boards. In view of the complexity of the coating and the presence of shadow zones, the production of UV-curable coatings with a double-curing mechanism was developed and launched. Among them, of particular interest are materials in which spatially crosslinked structures are formed by two mechanisms: polycondensation and radical polymerization during UV-curing. Their advantage is that, on the one hand, it is possible to control the process inexpensively and efficiently, and on the other, the coating is chemically cured in parts of the surface that are hidden from exposure to radiation. The development of such coatings, cured within a few seconds under the influence of UV radiation and for several hours in shaded areas due to interaction with the

hardener, significantly expanded the possibilities of using UV technology. Double curing systems are usually cross-linked first with UV radiation due to radical polymerization, and then through the reaction of isocyanate and hydroxyl groups to form a urethane group. The study of the physico-mechanical properties of coatings based on double cured varnishes, their protective and dielectric characteristics (specific, volume and surface resistance, electric strength, dielectric constant, dielectric loss tangent) made it possible to recommend these materials for moisture protection of printed circuit boards. The time required for their application and curing is from 0,5 to 1,5 minutes. It should be noted that the total duration of technological operations for applying and curing all layers of the previously applied varnish was at least 25 hours, which is not technologically advanced for serial production of products [14,22]. Tab. 3 shows the technical characteristics of a two-component UV curing varnish for moisture protection boards.

Table 3. Technical characteristics of a two-component UV curing varnish for moisture protection of boards

№ п/п	Name of indicators	Test method	Indicator value
1	Appearance	GOST 293-92	After curing it should form a uniform glossy film without mechanical impurities
2	Adhesion, score, no more	GOST 15140 part.2	1
3	Film strength upon impact with U-1 device, cm, not less than	GOST 4765	40
4	The hardness of the film according to TML-2124 method A, cu, not less	GOST 5233	0,45
5	Film elasticity in bending, mm, not more than	GOST 6806	1
6	Resistance to 5% salt fog with a coating thickness of 50 microns, h, at least	GOST 20.57.406-81 Method 215-3	500
7	Volumetric electrical resistivity, Ohm × s, not less	GOST 50499-93	1×10 ¹⁴
8	Dielectric constant, no more	GOST 6433.4-71	4,5
9	Dielectric loss tangent, not more than	GOST 6433.4-71	0,03
10	Electric strength, kV / mm, not less	GOST 27427-87	60

Conclusion. The developed formulation of UV double -curable varnish made it possible to obtain moisture-proof coatings for electronic equipment with protective and dielectric properties, both in the initial state and after simulating the effects of long-term storage.

The achieved parameter of the electric strength of the coating (not less than 60 kV/mm) is especially important, which allows us to recommend the developed material in the manufacture of both low-voltage and high-voltage equipment, as well as the high resistance of the coating to fog (at least 500 hours), characterizing its effective protective properties.

High strength indicators, adhesion and cohesion properties, as well as the stability of these properties in the conditions of sharp temperature changes, expand the possibilities of using UV-cured composite materials, gradually replacing traditional repair compounds. Today, the field of application of UV-curable composite coatings extends from medicine and housing, where they are already actively used, to space technology. By varying the composition and properties of the components of composite materials (matrix and filler, their ratio, filler orientation), you can get almost any product with a predetermined combination of operational and technological properties. The most promising polymer composite materials as repair compositions are compositions based on photocurable binders due to the already mentioned advantages of the technology [1, 5, 23, 24]. First of all, a short coating formation cycle; increased installation speed; the absence of significant energy costs and expensive equipment, as well as the absence of solvents in the system and the ability to ignite, and this is with the real possibility of obtaining a high-quality product.

To fully undergo the polymerization reaction, the reinforcing material used in the composite must have a high throughput of UV radiation. Traditionally, high-pressure mercury emitters emitting in the range 254-456 nm were mainly used in industrial UV-curing technologies. Such an emitter has a number of disadvantages: low efficiency, surface heatability, environmental damage. Mercury emitters are being replaced by LED technologies (UV LED technologies) with LED sources, which have several advantages: low power consumption, long life, increased durability, environmental safety and much higher efficiency [5]. The role of the photoinitiator in the FPK is difficult to overestimate [25]. One of the main parameters of UV-curable compositions is the region of its maximum spectral sensitivity, which should coincide with the most intense lines of the UV spectrum of the radiation source. The curing technology of LED sources has many advantages, but a distinctive feature is that LED sources have a narrow emission range of 390-410 nm. To cure the FPK layer using LED sources, it is necessary to use photoinitiators with energy absorption in this narrow range. As a rule, such photoinitiators are thick, yellow components, which is inconvenient in the technological process, since it is not possible to obtain transparent and pastel shades of coatings.

At present, the use of a photoinitiating system consisting of two or more photoinitiators is an effective technique for increasing the efficiency of initiating polymerization by LED radiation. Such a system is characterized by higher efficiency due to the manifestation of a synergistic effect. The use of a combination of photoinitiators of various types in FPK makes it possible to obtain a fully cured coating, while curing is equally effective both on the surface and in the depth of the layer [2, 20, 24].

Preparation of prepregs (combining a binder and a filler) can be carried out using the calendaring method (Fig. 4) and the s-wrap method (Fig. 5). The finished composite (prepreg) can be presented for use in the form of «double-sided tape» or in the form of various geometric dimensions packed in airtight packaging.

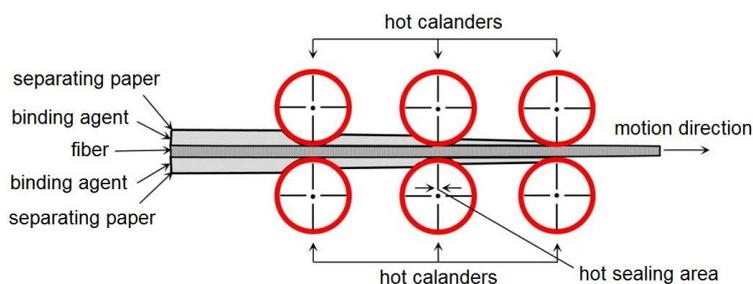


Fig. 4. Preparation of the prepreg by calendaring

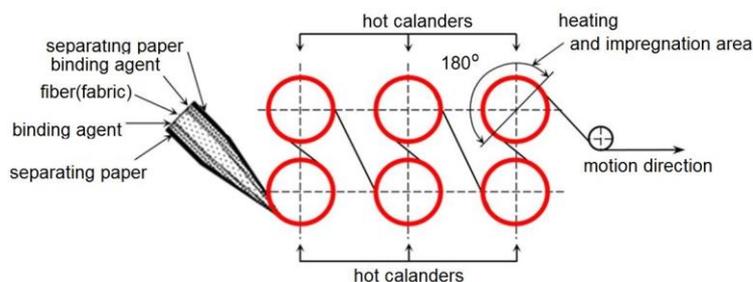


Fig. 5. Getting the s-wrap prepreg

The finished composite (prepreg) can be presented for use in the form of «double-sided tape» or packed in airtight packaging of various geometric sizes. The use of composite repair composite material consists of simple actions:

- opening the packaging of LED-cured composite prepreg;
- the imposition of damage to the cutting of fabric LED-cured composite prepreg of the required size, in the subsequent irradiation of LED sources. Under the influence of LED radiation or visible radiation, the binder polymer cures;
- LED technology allows you to cure composite material on products of various geometric shapes in a few minutes, which is also characterized by high strength and adhesion to the surface, which makes such a material an integral part of the repaired structure. Prospects for the development of this technology and widespread adoption in all spheres of human activity are inevitable in the foreseeable future.

Conclusion

The technology for producing coatings and products using UV-curing is a knowledge-based and promising direction: the coating can be formed in fractions of a second, the product – depending on the chosen method of forming the product, from several seconds to several minutes. UV-curing technology has been actively implemented in 3D-printing, in construction, in medicine, in design, in cosmetology, in the production of optical cables, in the production of rolled tubes, in the automotive industry and in the manufacture of printed circuit boards. The field of application of UV-curable materials is constantly expanding and developments in the field of improving their formulations are in demand and are actively carried out in Russia and abroad.

Unfortunately, the share of domestic production of UV-curable and LED-curable materials is still very small in comparison with imports. Now in Russia a fairly wide range of UV-curable and LED-curable materials is offered by S&H Technology (St. Petersburg), which carries out its own development and has industrial production.

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