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RESEARCH ARTICLE

Application of interlayer perforation and installation of transparent elements in the technology of duplicated decorative polymer films

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Abstract

Objectives. To develop technologies for producing multilayer films of transparent thermoplastic polymers; to study methods of modifying their supramolecular structure; and to determine their optical properties by means of optical-polarization methods for the use of modified films as decorative and design materials in modern architecture.

Methods. Industrial samples of polystyrene, low-density polyethylene, polypropylene, and polyvinyl chloride films from various manufacturers (*Don Polimer*, *Vektor*, and *Sibur*) were the objects of the study. The optical properties of the films were studied by means polarized-light spectrophotometry. In order to modify the supramolecular structure of the polymers, the surfaces of the films were treated under isometric conditions with volatile solvents or their aqueous solutions. Parts of the layers of multilayer films were removed by cutting with a punching knife using a press or with a manual device for perforating printing materials.

Results. The spectral characteristics of multilayer films of several transparent thermoplastic polymers in polarized light were determined. The study showed that a wide palette of colors and contrasting images can be obtained by mechanically removing part of the layers of multilayer films. The phenomenon of pseudo-disappearance of the outermost layer was discovered after treatment of a stack of films under isometric conditions with volatile solvents or their aqueous solutions.

Conclusions. Based on the example of large-scale production thermoplastics, it was shown that a combination of technological methods of stacking, perforation, and local plasticization of films of transparent thermoplastic polymers can produce pleochroic multicolor materials for a range of human activities. The possibility of hidden coding of information on multilayer packaging materials, and its visualization and instrumental reading in polarized light was confirmed by color differential and contrast of 150 and 60 units, respectively. It was also shown that several monochrome tones of different lightness and brightness can be obtained by varying the number of layers or perforating the films in multilayer materials.

Keywords

birefringence, interference image, modification, shrink films, internal stresses, supramolecular structure

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НАУЧНАЯ СТАТЬЯ

Применение межслойной перфорации и закладки прозрачных элементов в технологии дублированных декоративных полимерных пленок

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Аннотация

Цель. Исследовать технологии многослойных пленок из прозрачных термопластичных полимеров, способы модификации их надмолекулярной структуры, а также изучить их оптические свойства с помощью оптико-поляризационных методов для использования модифицированных пленок в качестве декоративных и дизайнерских материалов в современной архитектуре.

Методы. Объектами исследования являлись промышленные образцы пленок полистирола, полиэтилена низкой плотности, полипропилена и поливинилхлорида различных производителей («Дон Полимер», «Вектор» и «Сибур»). Оптические свойства пленок исследовали с помощью спектрофотомерии в поляризованном потоке света. Для модификации надмолекулярной структуры полимеров поверхности пленок обрабатывали в изометрических условиях летучими растворителями или их водными растворами. Части слоев многослойных пленок удаляли высечкой штанцевым ножом с помощью пресса или ручным устройством для перфорации полиграфических материалов.

Результаты. Получены спектральные характеристики многослойных пленок из нескольких прозрачных термопластичных полимеров в поляризованном потоке света. Показаны возможности получения широкой палитры цветов и контрастных изображений механическим удалением части слоев многослойных пленок. Обнаружен эффект «псевдоисчезновения» внешнего слоя при обработке пакета пленок в изометрических условиях летучими растворителями или их водными растворами.

Выводы. На примере крупнотоннажных термопластов показано, что сочетанием технологических приемов сборки, перфорирования и локальной пластификации пленок из прозрачных термопластичных полимеров можно решать задачи создания многоцветных материалов с эффектом плеохроизма для различных сфер деятельности человека. Возможность скрытого кодирования информации на многослойных упаковочных материалах, ее визуализации и инструментального считывания в поляризованном свете подтверждена достаточным цветовым отличием и контрастом в 150 и 60 единиц соответственно. Показана возможность получения нескольких монохромных тонов различной светлоты и яркости варьированием числа слоев или перфорацией пленок в многослойных материалах.

Ключевые слова

двойное лучепреломление, интерференционное изображение, модификация, термоусадочные пленки, внутренние напряжения, надмолекулярная структура

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INTRODUCTION

Established optical phenomena in polarized light in the form of a bright color of alternating stripes in transparent polymer bodies when under mechanical stresses enable their destruction to be predicted. They also enable the permissible level of load during operation to be monitored [1–2]. This practically important and effective method of non-destructive testing and mechanical testing of models constructed from transparent materials is used in the design of complex machine parts and building structures. Another important application of optical color effects in polarized light in transparent polymers is hidden packaging marking technology [3]. The creation of colored labels, multilayer labels, and other elements of original polymer products can also provide protection from counterfeiting. The advantages of such technologies include the creation of eye-catching color effects on products and packaging without the use of toxic dyes and pigments, the possibility of recycling used products and packaging made of thermoplastic polymer materials, as well as the production of feedstock (large-scale production polymers) [4].

Colors resulting from the interaction of light with spatially ordered or quasi-amorphous nanostructures or microstructures are called structural colors [5]. Such colors are widely found in nature in a range of insects, plants, and animals [6, 7]. Structural colors with a wide range of shades also appear in transparent polymer films with a high level of internal stress when polarized light passes through them. The heat sensitivity of the structural color of films of large-scale production thermoplastics is very specific. It depends on the presence of low-molecular ingredients in them and the features of the film formation technology. This may slightly increase their cost, but significantly expands the scope of application [8].

Internal stresses in polymer products and films are a natural consequence of their production technology [9]. Thin polymer films are produced by the extrusion and jet drawing of a melt. This is followed by the orientation of an elastic preform, during which internal stresses in the film are frozen. This procedure creates one-dimensional anisotropic shrink labels and biaxially oriented films of thermoplastics: polypropylene, polyethylene, polyvinyl chloride, and polyamides [10, 11]. The combination and duplication of these materials opens up new possibilities for their practical application in a range of industries and construction. A further factor is the variety of optical phenomena [12] caused by refraction, reflection, polarization, and interference of natural daylight and artificial light, as a combination of multiphase electromagnetic radiation [13].

The intensity of white light after passing through the polarizer–stressed structures–polarizer transparent system can be determined by the following expression:

$$I = \sin^2 2\alpha \sum I_\lambda \sin^2 \frac{\Phi_\lambda}{2},$$

where I is the light intensity, α is the angle between the polarization axis and the vector of the incident light, I_λ is the light intensity of the wavelength from the spectrum, and Φ_λ is the difference in the wavelength path from the radiation spectrum [1].

The phase difference of light-emitting and light-reflecting objects at the point of observation through the film depends on internal stresses and causes the phenomenon of pleochroism, i.e., the dependence of their color on their relative position relative to the radiation source and the observer. The use of pleochroism was proposed, in order to create new methods for protecting products made of transparent polymers from counterfeiting and for identifying original products by packaging, as well as to produce decorative light panels and transparent interior design elements [14, 15].

The purpose of this work was to investigate methods for controlling pleochroism in multilayer polymer materials made of thermoplastic polymers by varying the polymer used and treatment method and to justify the possibility of using them for the hidden recording of information by bar coding.

EXPERIMENTAL

Industrial samples of colorless transparent shrink films made of thermoplastic carbon-chain polymers produced in Russia were the objects of the study. They are low-density polyethylene with a thickness of 50 ± 2 μm, Biaxplen HGPL polypropylene 30 ± 1.5 μm thick (*Sibur-Biaksplen*, Russia), polyvinyl chloride 50 ± 2 μm thick (*Don-Polimer*, Russia), and polystyrene 60 ± 2 μm thick (*Vektor*, Russia).

Birefringence experiments were carried out on a laboratory setup (Fig. 1) designed for photography and optical measurements in transmitted polarized light. The radiation source was a light-emitting diode (LED) strip with a color temperature of the transmitted light flux (6500 K) and a nonlinear spectral characteristic (Fig. 2). The LED strip was mounted around the perimeter of a sealed box with a transparent window made of silicate glass, on which a film linear polarizer (*Nitto Denko*, Japan) was placed. After passing through the polarizer and the analyzer in a crossed position, the radiation spectrum can be considered to be either monotonic or linear [1, 13] (Fig. 2).

The spectral characteristics of the radiation source, polarizing films, and stacks of shrink films before and after external influences were determined using an X-Rite i1Pro spectrophotometer (*X-Rite Inc*., USA). The spotread command line tool from the ArgyllCMS software[1], version 2.3.0, was used. The measurements were made in the "high-resolution" mode (measurement step 3.333 nm). The radiation characteristics and the color of the transmitted light were recorded in units of spectral radiance: watt per steradian per square meter per nanometer (mW/(sr·m^2·nm)). The spectrophotometer was also used to evaluate the contrast of images visible in polarized light and to determine the CIELab[2] color coordinates used to calculate the color difference ΔE_{ab}:

$$\Delta E_{ab} = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2},$$

where L^* is lightness, a^* are red–green coordinates, and b^* are yellow–blue coordinates.

Based on the spectral transmittances of different areas of the multilayer films, color coordinates were calculated

[1] https://www.argyllcms.com/. Accessed February 8, 2023.

[2] CIELab is a color space defined by the International Commission on Illumination (CIE) in 1976.

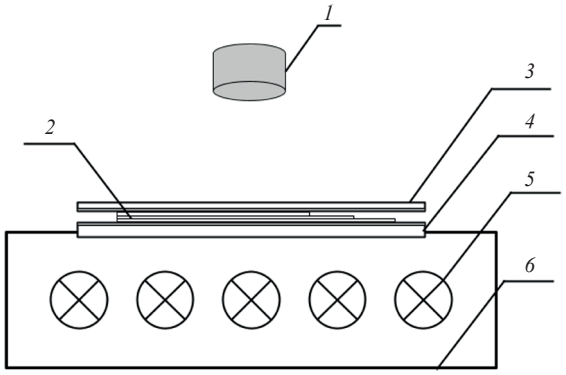


Fig. 1. Schematic of a setup for measuring the spectral characteristics of film samples: (*1*) spectrophotometer, (*2*) a film sample or a stack of films (Stoletov's stack), (*3*) movable polarizer, (*4*) fixed polarizer, (*5*) LED strip around the perimeter of the camera, and (*6*) sealed chamber

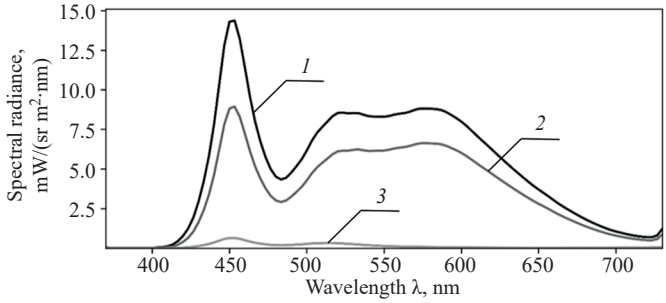


Fig. 2. Spectral radiances of the LED strip: (*1*) light spectrum of the radiation source, (*2*) light spectrum through two linear polarizers with mutually parallel polarization axes, and (*3*) light spectrum through two linear polarizers with mutually perpendicular polarization axes

in the *XYZ* space, where *Y* is the luminance component. Then, from one Y_1 value, the Y_2 value for the adjacent area was subtracted, in order to give the symbol contrast.

The internal stresses in samples of polymer films were altered by plasticization through local treatment with active solvents and their aqueous solutions (tetrahydrofuran, acetone, methylformamide, Russia). Heat treatment was performed in laboratory-scale apparatuses for welding polymer films of various designs [16].

The color effects were visually recorded using a Nikon D7000 camera (*Nikon*, Japan).

From the selected polymer materials, films were arranged in Stoletov's stacks. This also involved the cutting of part of the layers or the addition of flat transparent elements using both monopolymer films or a combination of films of different thermoplastics.

RESULTS AND DISCUSSION

In order to obtain and quantify pleochroism, film samples were cut into strips of various lengths and 30 mm wide along (or across) the winding direction of the roll and stacked on top of each other in Stoletov stacks. From 2 to 14 layers were used, in order to prepare a stack of films. The stacking of more layers of film significantly reduces the intensity of transmitted light and the accuracy of measurements [12].

The maximum rainbow effect was seen on films of amorphous glassy polymers: polyvinyl chloride and polystyrene. Films of elastic crystallizable polymers produced color palettes with were less bright and less multicolor. The lower level of brightness of the colors in polyethylene stacks is apparently due to decreasing the level of internal stress in the materials because of relaxation during storage. Table 1 indicates the original color when observed at a right angle, the order of color changes and changes in the lightness of shades, the frequency of occurrence and alternation of the color gamut for each polymer and film stack. For polyvinyl chloride and polystyrene films, color and lightness are repeated every 2–3 layers. In low-density polyethylene and polypropylene film stacks, color is repeated in every other layer.

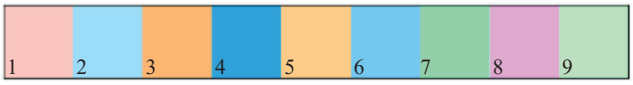
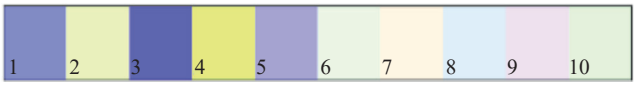
For the purposes of the practical application of the results of the spectrophotometry of multilayer films of all the polymers studied, Fig. 3 indicates points with color coordinates in the color diagram (Fig. 3).

Active solvents which cause plasticization of polymers and a decrease in internal stresses in films affect the color palette of a stack of films of amorphous glassy polymers: polyvinyl chloride and polystyrene. The reason for the change in color of the film stack in polarized light is due to a change in the internal stresses of these polymers due to plasticization [17]. Even short-term (several minutes) exposure of the film surface to a solvent led to a noticeable local change in the color of the film stack (Fig. 4). In the case of multilayer samples, in the areas of contact between the film and the liquid, the colors of adjacent internal layers appear under the outermost layer which absorbs a certain amount of solvent. This phenomenon can be called pseudo-disappearance of the outermost layer of the multilayer film. After the solvent evaporates, this local optical effect persists for a long time, indicating an irreversible decrease in internal stresses.

The effect of solvents on the film color can be used in the technology of special liquid-sensitive materials for security printing. One of the methods of operational visual control is wetting the surface of prints on special water and fluid-sensitive materials with test liquids or solutions. This can be used to verify the authenticity of banknotes and documents [18].

It is reasonable to assume that the transillumination of a film stack causes no changes in the outermost layer

Table 1. Film colors when observed at a right angle

Polymer	Color palette
Polyvinyl chloride	
Polystyrene	
Low-density polyethylene	
Polypropylene	

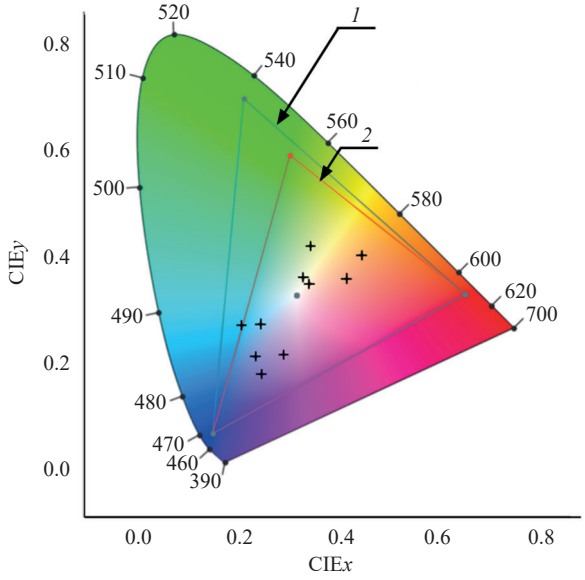


Fig. 3. Positions of colors of a polyvinyl chloride film stack on the CIE*xy* chromaticity diagram: (*1*) Adobe RGB (1998) color gamut and (2) sRGB color gamut

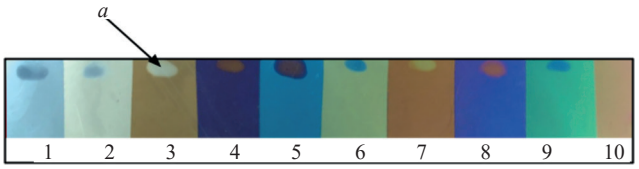


Fig. 4. Exposure of a PVC film to the solvent in areas *a*. The numbers 1–10 show the number of layers in ascending order

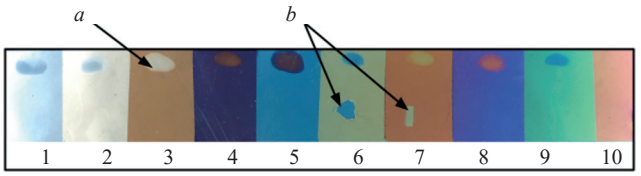


Fig. 5. Short-term local exposure of a film to solvent in areas *a* and cutting of one layer of film in areas *b*

of the film in the direction and phases of the light beam. This is due to the absence of film in this place. This phenomenon is known as pseudo-disappearance of the outermost layer from the stack of transparent films. Since the transmission of polarized light through a medium with internal stresses gives rise to an interference pattern, after contact with the solvent, there are either no internal stresses in the film, or their level is significantly reduced to such an extent that it is insufficient to change the characteristics of the transmitted light. In order to confirm this assumption about the physical essence of the phenomenon of pseudo-disappearance, fragments of films inside and on the surface in different layers were specially cut out in a multilayer stack near the place where a drop of plasticizing liquid was applied. Transmitted polarized light from the film stack with perforated layers led to the same effect as exposure of the film to a volatile solvent (plasticizer).

The perforation of films has an important technological and operational advantage over treatment with solvent. It is not only the outer layers of films of multilayer materials that can be perforated (or not to any significant

Table 2. Color difference between adjacent layers

Material of polymer film	Color difference$_{max}$	Layers
Polystyrene	150	Between layer 10 and layer 5
Polyvinyl chloride	126	Between layer 8 and layer 6 Between layer 10 and layer 8
Polypropylene	125	Between layer 4 and layer 3
Low-density polyethylene	117	Between layer 9 and layer 8

Table 3. Contrasts of gradient color areas of a polyethylene film

Compared areas of multilayer stack	1–3	1–5	3–5	2–4	2–6	4–6	7–9
Contrast	44	48	12	29	23	9	11

extent), but also any inner ones. When the inner layers are perforated, the multilayer material with an image or text hidden from the naked eye will have a higher degree of strength and a higher resistance to external influences when used, e.g., as commercial packaging.

For the practical application of this effect, it is necessary to determine between which layers part of the film should be removed, in order to obtain the maximum color difference between adjacent areas. For quantitative assessment, the CIE76 color difference formula was used [3]. For each of the films, the maximum values of color difference and the number of layers at which it occurs were identified (Table 2).

Such indicators of color difference are many times greater than the threshold value distinguishable by the human eye: $\Delta E \approx 2–3$ [12]. These quantitative estimates are confirmed visually, since the lightness or brightness curves of these colors are different. In order to resolve the problems of hidden marking of multilayer films intended for packaging and protection against counterfeiting of expensive original products [12, 18], the most important factor is to compare the color indicators between shades of the same color. For example, in the case of low-density polyethylene films with repeating colors, a table of contrasts of areas of a multilayer film of the same spectral color, but different tones (gradient color), was compiled (Table 3).

The colors of such areas of the film appear identical, but their color difference ΔE is many times greater than the resolution of the human eye. This color difference was determined for all polymer materials studied in this work. The presence of several shades of the same color in materials made of one polymer, but with a different number of layers, expands the artistic possibilities for creating a tone image.

Another important characteristic of multilayer films intended for marking is the contrast of adjacent areas of the film with different numbers of layers or cut locations. Image contrast is a quantity different from the color difference which shows the difference between two parameters [19].

For the film stacks under consideration and collected in Stoletov's stack (Fig. 3), the contrast was calculated (Table 4).

It can be noted (Fig. 6) that for the majority of the studied polymer films, maximum contrast occurs in the stack between layer 1 and layer 2. This is very important for use in the production of two-layer materials with high-contrast protective elements. It can be seen that the contrast of adjacent areas in multilayer films decreases as the number of layers increases (Fig. 6). Among the films studied, the level of contrast is maximum in Biaxplen HGPL polypropylene film (Biaxplen, Russia) intended for packaging food and dietary products for children.

CONCLUSIONS

Based on the example of large-scale production thermoplastics, the study showed that a combination

Table 4. Maximum contrast values of adjacent areas

Material of polymer film	$Contrast_{max}$	Layers
Low-density polyethylene	44	Between layer 1 and layer 2
Polystyrene	46	Between layer 10 and layer 3
Polyvinyl chloride	57	Between layer 1 and layer 2
Polypropylene	63	Between layer 1 and layer 2

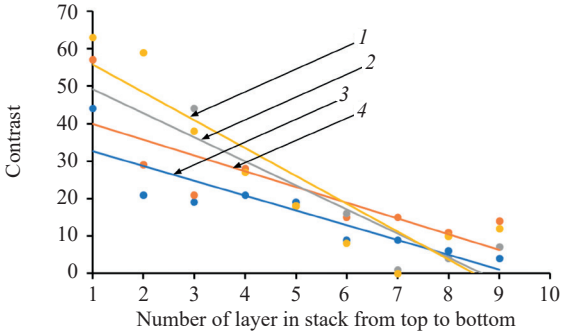


Fig. 6. Color contrast of adjacent layers in films of thermoplastic polymers: (*1*) polypropylene, (*2*) polystyrene, (*3*) polyvinyl chloride, and (4) polyethylene

of technological methods of stacking, perforating, and plasticizing films of transparent thermoplastic polymers can produce colored materials for use in various human activities: architecture and construction for creating stained glass and multicolor theatrical scenery; in the production of multilayer light filters; and in retail trade for the coding and protection of product packaging from counterfeiting. Multilayer polymer materials which change color in polarized light depending on the location of the polarizers, the direction of the transmitted light, and the position of the observer (pleochroism) are structured from films of glassy amorphous and amorphous–crystalline polymers with a high level of internal stress. Changing the level of internal stresses in films without changing their overall dimensions can be achieved by treating the outer layers of polymer films with volatile liquid solvents or plasticizers. The decrease in the level of internal stresses in the outer layers of duplicated polymer materials comprising N films after exposure to plasticizers, thus causing a change in the color of the film stack in polarized light to the color of materials comprising $N - 1$ layers of films, is referred to as pseudo-disappearance of the outermost layer. It forms the basis of a new technological method in production of decorative polymer materials and artistic works.

The pseudo-disappearance of the outermost layer and the perforation of the inner layers cause a significant color difference and contrast of adjacent areas of the film stack, reaching 150 and 60 units respectively. These contrast values are sufficient for the hidden barcoding of transparent product packaging, identifiable in polarized light.

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Authors' contributions

A.A. Nikolaev — planning the experiment, carrying out the study, collection and provision of the material, writing the article.

A.P. Kondratov — writing the article, scientific editing.

The authors declare no conflicts of interest.

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