SYNTHESIS AND PROCESSING OF POLYMERS AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

Method for hidden marking of transparent polypropylene film

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Abstract

Objectives. *To quantitatively describe the thermochromic properties of films of isotactic polypropylene, a large-tonnage polymer widely used in the production of flexible packaging for goods and foodstuffs, as well as substantiate the possibility of covert labeling of transparent packaging.*
Methods. *Differential scanning calorimetry, polarization photometry, infrared Fourier spectrometry, gravimetry, temperature control, physical and mechanical strength testing.*
Results. *The identified thermochromic effect of dichroism in polarized light on industrial samples of transparent biaxially oriented film of isotactic polypropylene was studied. A change in the phase composition of the film-forming composition during short-term heating during marking was established. The absence of heat shrinkage and change in transparency in non-polarized light was shown, which provides the possibility of hidden recording of information and its contrast manifestation in a passing light stream at a certain arrangement of light filters.*
Conclusions. *The causes and optimal conditions of the thermochromic effect are established. It is proposed to use local contact heat treatment of a polypropylene film for covert recording of information and marking of product packaging in order to protect against counterfeiting.*

Keywords: *oriented film, polarized light, heat treatment, isotactic polypropylene, differential calorimetry, dichroism, color difference, hidden marking*

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НАУЧНАЯ СТАТЬЯ

Скрытая маркировка прозрачной пленки полипропилена

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Аннотация

Цели. *Количественно описать термохромные свойства пленок изотактического полипропилена – крупнотоннажного полимера, широко используемого в производстве гибкой упаковки товаров и продуктов питания, и обосновать возможности скрытой маркировки прозрачной упаковки.*
Методы. *Дифференциальная сканирующая калориметрия, поляризационная фотометрия, ИК Фурье-спектрометрия, гравиметрия, термостатирование, физико-механические испытания, в том числе прочности.*
Результаты. *Обнаружен и исследован термохромный эффект дихроизма в поляризованном свете на промышленных образцах прозрачной двуосноориентированной пленки изотактического полипропилена. Установлено изменение фазового состава пленкообразующей композиции в процессе кратковременного нагревания при маркировке. Показано отсутствие термоусадки и изменения прозрачности в неполяризованном свете, обеспечивающее возможность скрытой записи информации и ее контрастного проявления в проходящем потоке света при определенном расположении светофильтров.*
Выводы. *Установлены причины и оптимальные условия термохромного эффекта. Предложено использовать локальную контактную термообработку пленки полипропилена для скрытой записи информации и маркировки упаковки товаров и продуктов с целью защиты от подделок.*

Ключевые слова: *ориентированная пленка, поляризованный свет, термообработка, изотактический полипропилен, дифференциальная калориметрия, дихроизм, цветовое различие, скрытая маркировка*

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INTRODUCTION

The central problem of innovative materials science for mass—demand products is the optimization of the ratio of the price of the material and the product result of its use in industrial production. The pleochroism effect in anisotropic polymer films can become the basis of new technologically sophisticated methods of protecting marketable products and packaging of unique products from counterfeiting that do not require the use of expensive materials [1]. The pleochroism effect of polyolefins has previously been studied in multilayer polyethylene films used for food packaging, as well as in direct contact with rapidly perishable products. Several layers of a film of the same polymer, layered and glued together, and the resulting color effects in polarized light make it possible to apply such a technology to create coding according to the Microsoft Tag system [2–4].

In the works presented earlier, it was proposed to use multilayer polymer films to obtain identification and protective elements on goods of value. These elements are used both in the visible light range of 400–700 nm, but at different viewing angles, and in the infrared and ultraviolet ranges using special devices and auxiliary elements (polarizers, light sources in the wavelength range of 360–400 nm, etc.) [5–8]. Patent sources describe high-cost and material-intensive, but similar in technology, methods for marking multilayer birefringent films [9].

In this paper, an experimental substantiation of the possibility of labeling polypropylene film packaging by short-term local heat treatment under pressure using industrial equipment for thermal welding of thermoplastic polymers and thermogravimetry is proposed [10]. With a local thermal effect on the film with a certain periodicity and frequency of repetition of heat treatment along the length or width, its properties change, and the homogeneous film material becomes interval. To clarify and further use the term "interval material," it should be indicated that this term refers to a special case or one of the variants of the implementation of the so-called "gradient" polymers. This term was first introduced by A.A. Askadsky in reports at conferences on the results of theoretical and experimental work on the technology of inhomogeneously crosslinked elastomers, after which he received international recognition [11]. Periodicity or frequency, size and amplitude, differences in the properties of intervals can act as a key in recording and reading information and be used in labeling packaging. If the difference in the properties of the intervals is not visually determined, but requires a special light source, a method of illumination, or the use of polarizers [12], then such interval materials are promising in lighting engineering [8], technology of hidden optical and relief marking [13], and the fight against counterfeiting of packaged products or goods [9].

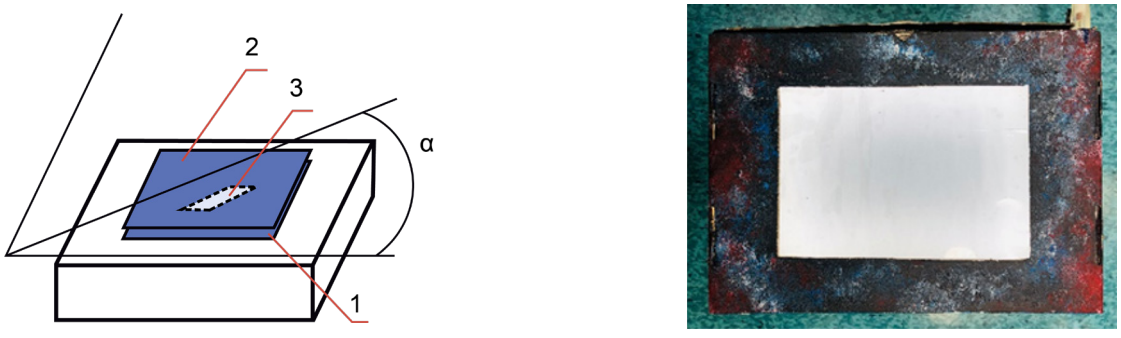
MATERIALS AND METHODS

The objects of research were commercial samples of a biaxially oriented "sleeve" film of isotactic polypropylene (*Bureaucrat*, Russia and *Business Center*, Russia) with a thickness of 22 ± 1.5 μm (hereinafter BOPP-22) and a thickness of 105 ± 2 μm (hereinafter BOPP-105), respectively.

To assess the mechanical properties and calculate the possible anisotropy of the films, the tensile strength was measured at a constant speed of 50 mm/min in accordance with GOST 11262-2017 (ISO 527-2:2012[1]) .

In order to assess the internal stresses and control the shrinkage of films at a temperature corresponding to the temperature of thermomechanical processing, in the process of marking and obtaining samples of interval materials, a laboratory technique and specialized equipment have been developed to prevent warping and sticking of the film when heated to the melting point of polypropylene. The technique involves the production of an envelope with an anti-adhesive Teflon coating, inside which film samples of 50 × 50 mm are placed. An envelope with a sample of polypropylene film is heated on the surface of the molten Wood alloy to a temperature of 150°C for 5–50 s.

To evaluate the optical properties of the film in transmitted polarized light, a viewing table [14] modified to produce polarized light was used (Fig. 1). The light source was a light-emitting diode strip with a luminous intensity of 300 lm and a color temperature of 6500 K. In order to



a b

Fig. 1. Viewing table: (a) schematic representation; (b) top view (photo).
1 – polarizer; 2 – analyzer; 3 – polypropylene film; α is the angel of rotation of the polypropylene film.

[1] ISO 527-2:2012. Plastics – Determination of tensile properties. Part 2: Test conditions for mouldings and extrusions plastics. Publication date: 2012.02.

polarize the light, a general-purpose polarizer with a single permeability of 44.5%, a polarization effect of 95.8% (*Nitto*, Japan) was used. Shade *a* is *N*-bromosuccinimide (NBS) 25 – (–1.6), shade *b* is NBS 25 – (–0.9).

The description of optical effects following heat treatment and measurement of film color parameters in transmitted light were carried out from photographs obtained in room (laboratory) conditions under different lighting conditions using a 12 MP camera and a Display P3 color profile.

The color effect was evaluated by the color coordinates of the equal-contrast color evaluation system *L*a*b**, as well as by the magnitude of 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 coordinates of the red-green shade, *b** are the coordinates of the yellow-blue hue, according to the CIELab color space (more precisely, CIE 1976 *L*a*b** or CIE-76).

The color coordinates *L*, *a*, *b* were measured from photographs in Adobe Photoshop software (*Adobe Systems*, USA) at 10 points of the field with the calculation of the average value. The photo was opened in the program using the built-in DisplayP3 color profile. Without changing the color profile in the photo, the Eyedropper tool selected 10 arbitrary points, which correspond to the color coordinates of the *L*a*b** system.

To measure thermochromic effects, several series of 5 samples of two types were prepared. Rectangular samples of 30 × 50 mm in size were cut from polypropylene films with a thickness of 22 ± 1.5 μm and 105 ± 2 μm in mutually perpendicular directions both along the direction of the predominant orientation of macromolecules (maximum strength) (Arrow 1) and perpendicular to the direction of the predominant orientation of macromolecules (Arrow 2) (Fig. 2).

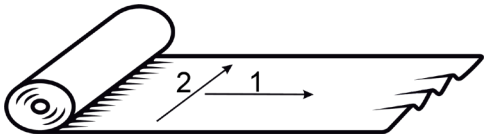


Fig. 2. Preparation of a biaxially oriented polypropylene film (=),
Arrow 1 is the direction of preferential orientation macromolecules,
Arrow 2 is the perpendicular direction (⊥).

To determine the optimal mode of observation and optical measurements, the dependence of the color difference of adjacent film intervals on the location of the sample between the polarizers in a crossed (closed) position with a step of 15° was investigated (Fig. 1a). The condition for photofixation of the maximum thermochromic effect was established based on the results of optical measurements and calculation of the color difference of adjacent film intervals (Fig. 3).

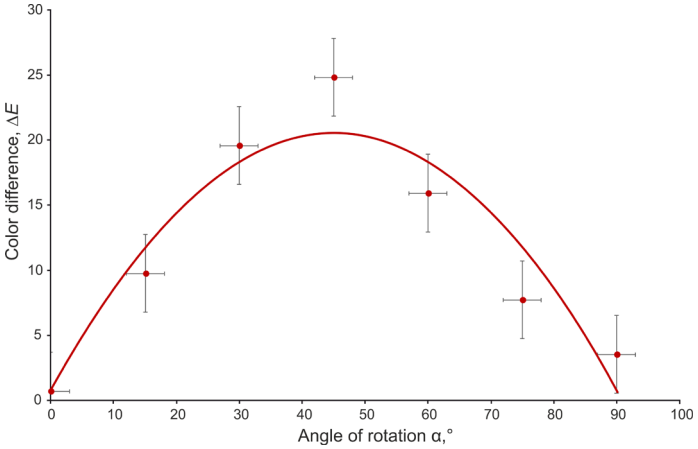


Fig. 3. Dependence of the color difference intervals in a polypropylene film 105 ± 5 μm thick on the angle of their arrangement between polarizers.

The greatest color difference is observed when the film sample is rotated by 45° relative to the polarizers, and the smallest when rotated by 0° and 90°. Based on this effect, then the position of 45° of the film in relation to the polarizers will be called ***active***, and when located at 0° or 90°— ***inert***.

Short-term (≤1 s) thermomechanical treatment was performed on a laboratory heat-welding stand of the HSE-3 brand (*RDM Test Equipment*, Great Britain) under a pressure of 0.2 MPa (30 psi). Cooling took place spontaneously on the surface of the table at the temperature of the laboratory room under the same conditions for all samples. The stand for welding films in automatic mode uniformly and uniformly compresses the flat surfaces of the heater.

Thus, objects for the study of optical properties and thermochromic effects were obtained: two types of samples of interval films with a thermomodified structure of intervals periodically repeating along their length and located in mutually perpendicular directions along or across the direction of the predominant orientation of polypropylene macromolecules.

A differential scanning calorimeter (DSC) was used to evaluate the crystal structure of interval polypropylene films with a thermomodified structure DSC 204 F1 Phoenix (*NETZSCH*, Germany). Diagrams in the DSC sensor signal–temperature coordinates were obtained at a heating rate of 10°C/min.

The chemical composition of the film was studied by the method of multiple disturbed total internal reflection on the FSM 2201/2202 IR Fourier spectrometer (*Infraspec*, Russia) using spectrum libraries: HR Specta Polymers and Plasticizers by ATR, HR Hummel Polymer and Additives, Hummel Polymer Sample Library, Synthetic Fibers by Microscope and HR Nicolt Sampler Library.

RESULTS AND DISCUSSION

Earlier [15, 16], it was shown that when natural sunlight and light obtained using modern indoor lighting systems pass through a "polarizer–multilayer polymer film (Stoletov's foot)–polarizer" system, the pleochroism effect is observed, providing a significant color difference between films having different number of layers. The color and quantitative parameters of the polarized light passing through the multilayer package depend on the chemical composition of the film-forming polymer, the structure and the presence of internal stresses in the films. The brightest colors were found on shrink films made of glassy polymers—polystyrene and polyvinyl chloride obtained by uniaxial orientation extraction, which is characterized by a high level of internal stresses and thermostimulated shrinkage of up to 60%. The macromolecular disorientation and reduction of internal stresses by heat treatment affects the intensity of the pleochroism effect, which is proposed for use to record information and hidden labeling of film packaging made of these polymers [3, 4].

Since most industrially produced films of isotactic polypropylene widely used in the production of flexible packaging of goods and food do not demonstrate the pleochroism effect when assembled multilayer materials, they cannot be used in hidden labeling. The absence of optical activity of polypropylene films may have been associated with a low level of internal stresses, insufficient anisotropy or orthotropy of the films. However, it is reasonable to assume that when polypropylene films are heated to a temperature close to the melting temperature range, recrystallization and/or disorientation of macromolecules will occur. This would allow optical marking of films in the form of "watermarks" that are visually distinguishable in polarized light.

Before searching for technological ways of optical marking of transparent polypropylene packaging, the presence of internal stresses in the studied samples was evaluated by the magnitude of shrinkage and anisotropy of mechanical properties, which are known factors determining the effects of dichroism or pleochroism [17].

The results of the strength tests of the films and the assessment of shrinkage at a temperature of 150°C are presented in Table 1.

While the samples of isotactic polypropylene film differ in thickness by 5 times, both samples are anisotropic. The fact that the tensile strength of the thin film in the direction of the predominant orientation of macromolecules exceeds the strength of the film having a thickness of 105 μm by almost 2 times is a consequence of the extrusion effect and a greater multiplicity of

Table 1. Mechanical properties of polypropylene films in mutually perpendicular directions

Film (thickness, μm)	BOPP-22		BOPP-105	
Die cutting direction	1 (=)	2 (⊥)	1 (=)	2 (⊥)
Tensile strength, MPa	67 ± 2	41 ± 2	45 ± 2	25 ± 2
Strength anisotropy	1.63		1.8	
Thermal shrinkage of the film, %	2 ± 0.5	2 ± 0.5	2 ± 0.5	2 ± 0.5
Degree of crystallinity, %	44.4		37.6	

extraction during its manufacture. The anisotropy of the mechanical properties of the films as determined by the ultimate strength is 60–80%; however, this does not significantly manifest itself during heat shrinkage.

Since industrial grade polypropylene macromolecules have deviations from isotacticity and different average lengths, as well as forming defective crystals that correspond to different melting temperatures [17], it is reasonable to expect a change in the structure and optical characteristics of films and the intensity of the dichroism effect when they are heated in the temperature range below the average melting temperature of isotactic polypropylene. Local heat treatment in the temperature range below the average melting point of isotactic polypropylene is considered in this study as a method for hidden optical marking of the film since it is unlikely to affect the shape and overall dimensions of a package or label of a thermally fixed film or cause their noticeable warping.

For testing, the temperature interval of the metal heat transmitter was selected by contact heat exchange from the maximum temperature of the product packaged in the film to the melting temperature of the polymer; here, the time intervals of their contact were comparable to the time of the packaging in high-performance packaging equipment that seals the packaging with thermal welding and/or labeling.

Thermomechanical processing of samples of polypropylene film with a thickness of 105 ± 2 μm at a temperature of 120°C and under a pressure of 0.207 MPa (30 psi) for 0.5 s led to a noticeable change in the lightness of the sample in polarized light. Since the heat-treated area of the film sample (hereinafter referred to as the modified interval) scatters the passing stream of polarized light, it has a lighter shade in the photo (Fig. 4). When varying the angle at which the interval film sample is positioned between two polarizers in a crossed (closed) position, a visually noticeable lightening of the modified film interval is observed while preserving the color of the unmodified part (Fig. 4).

The dichroism in the sample of the polypropylene interval film may be explained in terms of a change in the supramolecular and/or crystal structure of the orthotropically oriented film extract at different temperatures or the chemical composition of the film-forming polymer due to heating in contact with the metal electrode of the welding unit under pressure.

To separate the influence and quantification of thermal, mechanical and chemical (prescription) factors on the optical properties of the film, interval film samples were studied by IR spectroscopy, gravimetry and differential scanning calorimetry (DSC).

It was found on the IR spectrometer that in both samples of the polymer film there may be an impurity of the low molecular weight olefin fraction (Fig. 5a), which after heating from the film to 130–150°C is removed and not identified on the surface of the film (Fig. 5b).

The sublimation of the component from the polypropylene film can be confirmed by comparing the masses of the films before and after heating on high-precision scales. Pre-prepared samples (20 pieces with a total weight of about 5 g) are weighed on analytical scales of various designs with an accuracy of up to 4 digits and placed in a preheated thermostat. After thermostating, the total mass of the film samples decreased by an average of 0.003 g, which is an order of magnitude higher than the mass measurement error.

Based on the gravimetry of the film samples before and after thermostating, it can be assumed that this component of the thermoplastic composition of isotactic polypropylene is sublimated during heating (with hidden labeling), which affects its optical (color) characteristics. This assumption is confirmed by the DSC result (Fig. 6). On the endothermic DSC curves of samples following thermomodification in the

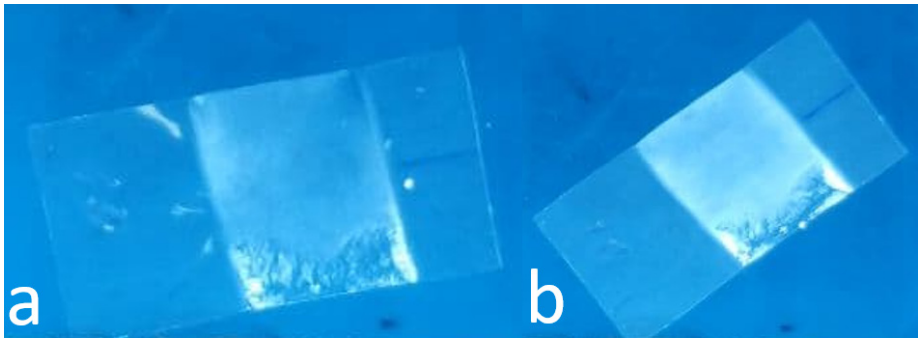
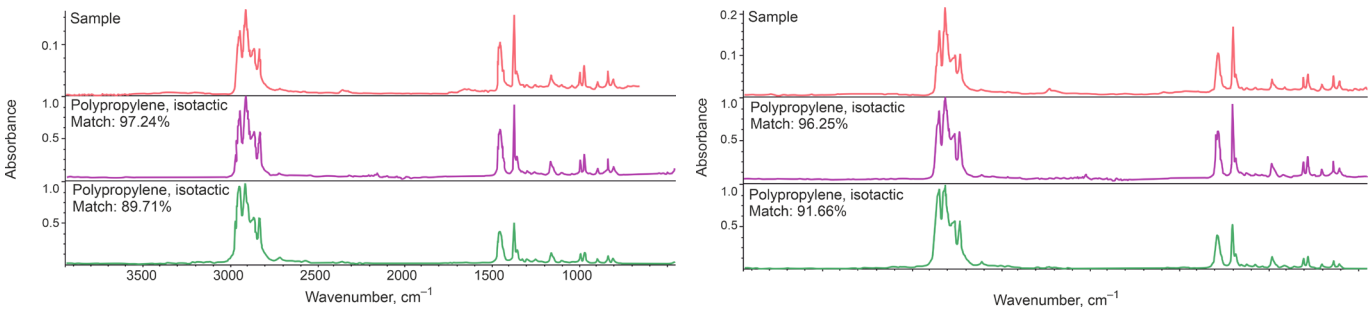


Fig. 4. Dichroism effect in a sample interval polypropylene film in transmitted polarized light when film sample is located relative to polarizers at angles 15° (a) and 45° (b).



a b

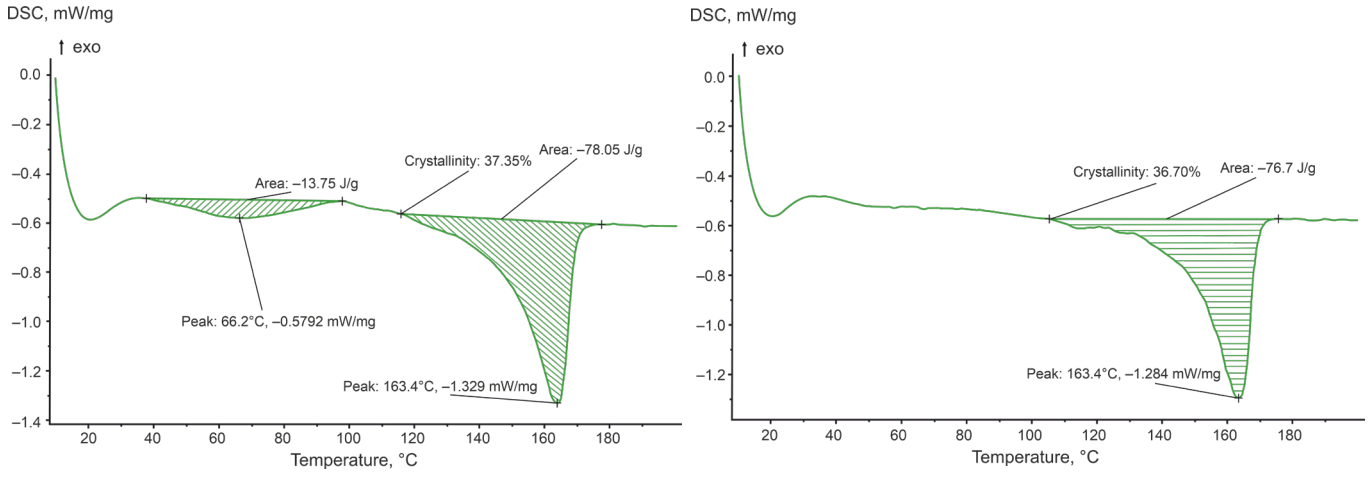
Index	Match, %	Compound	Index	Match, %	Compound
67	97.24	Polypropylene, isotactic	67	96.25	Polypropylene, isotactic
942	89.71	Polypropylene, isotactic	942	91.66	Polypropylene, isotactic
41	88.46	Polypropylene, atactic	566	90.29	Polypropylene, atactic
67	88.12	Polypropylene, isotactic	324	89.86	Polypropylene+poly(ethylene:propylene)
943	87.31	Polypropylene, atactic	67	89.85	Polypropylene, isotactic
129	86.24	Olefin	1061	86.94	Poly(propylene:butanone), 2:1
566	86.22	Polypropylene, atactic	41	86.52	Polypropylene, atactic

Fig. 5. Identification of the IR-spectra BOPP-105 film: (a) before heat treatment; (b) after heat treatment.

low temperature range (60–80°C), the endopic "disappears." The "disappearance" of the endopic on the DSC diagrams at 60–80°C reflects the sublimation, pre-polymerization and crystallization of olefin. The degree of crystallinity increases one and a half times from 37% to 57%. The added crystallinity appears to be defective and represents small supramolecular structures scattering the polarized light flux passing through the modified sections of the film.

According to the DSC diagrams (Fig. 6), the temperature interval at which the heat treatment of the polypropylene film can cause maximum color change and provide contrast of adjacent intervals in polarized light is selected. For isotactic polypropylene, this film temperature range is 60–100°C. However, taking into account the high thermal resistance of the contacting surfaces and the need to minimize the contact time of the marked film and the heating tool to increase the productivity of the process, the temperature range of 60–170°C was investigated.

The marking result is presented in the form of photographs of fragments of transition zones and modified intervals obtained in transmitted polarized light on film samples with a thickness of 22 ± 1.5 μm, cut in direction 1 (Tables 2 and 3). Similar results were obtained on a film with a thickness of 105 ± 2 μm.



a b

Fig. 6. Differential scanning calorimetry diagrams of polypropylene films: (a) before heat treatment; (b) after heat treatment.

Table 2. Color difference of intervals in a BOPP-22 polypropylene film with active α = 45° arrangement of samples (Fig. 3)

Temperature, °C	60	70	80	90	100	110
Visual evaluation of the effect						
Color difference, ΔE	0	1.86	15.73	24.81	32.51	33.80
Temperature, °C	120	130	140	150	160	170
Visual evaluation of the effect						
Color difference, ΔE	47.51	48.80	53.80	39.14	32.39	30.90

From the fragments of photographs and the values of the color difference of adjacent intervals, a color change in the transmitted polarized light is already observed following heat treatment with the active arrangement of the film starting from 70°C; however, the value of the color difference is lower than necessary for the sensitivity of the human eye [3, 4]. The maximum color difference of the adjacent intervals of the polypropylene film with a thickness of 22 μm observed at a temperature of 140°C is 53.8. A similar change in the optical properties of the film after heat treatment is also observed at the inert position of the interval film samples between polarizers, but at higher temperatures. The maximum color difference as a result of short-term contact (0.25 s) reaches 59 at a temperature of 170°C, i.e., in the middle of the melting interval of polypropylene

Table 3. Color difference of intervals in a BOPP-22 polypropylene film with an inert α = 90° arrangement of samples (Fig. 3)

Temperature, °C	60	70	80	90	100	110
Visual evaluation of the effect						
Color difference, Δ*E*	0	0	0	2.11	1.94	3.15
Temperature, °C	120	130	140	150	160	170
Visual evaluation of the effect						
Color difference, Δ*E*	0.94	12.03	9.78	57.68	57.53	59.00

crystallites [17]. At the same time, the maximum color difference of adjacent intervals of the polypropylene film with a thickness of 105 μm significantly depends on the location of the samples between the polarizers (Fig. 7).

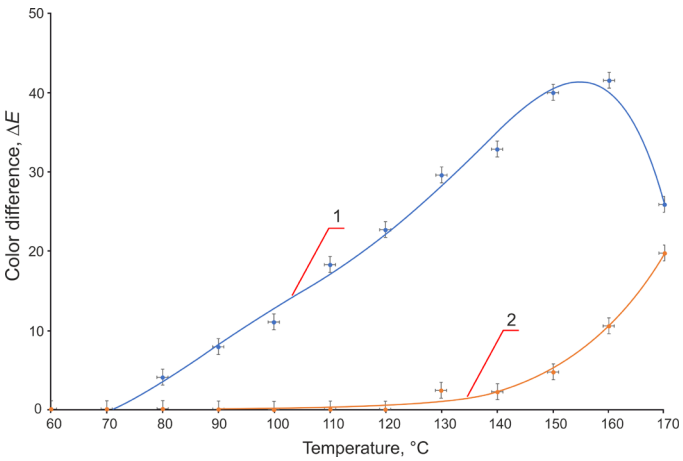


Fig. 7. Color difference between adjacent intervals of a polypropylene film (BOPP-105) after thermal marking with active α = 45° (1) and inert α = 90° (2) arrangement of samples polarizers.

Since a further increase in temperature or an increase in processing time does not increase the color change, the most effective treatment mode for polypropylene film with a thickness of 22 μm is 130°C for 0.1 s. However, at 130°C, the film softens and can stick to the processing equipment; therefore, it is recommended to either reduce the exposure time to 0.05 s or process the film at 125°C for 0.1 s.

CONCLUSIONS

The possibility of changing the color of a transparent biaxially oriented "sleeve" film of isotactic polypropylene in passing polarized light using the thermochromic effect for hidden labeling of transparent packaging by short-term local heat treatment is demonstrated. Heat treatment of a biaxially oriented isotactic polypropylene film stimulates partial removal and prolonged amorphization of the low molecular weight olefin fraction without heat shrinkage, changes in the degree of crystallinity and average melting point of the crystal structure.

The optimal temperature-time conditions for the contact treatment of the film with a tool heated to a temperature below the melting point of isotactic

polypropylene have been established to obtain the maximum color difference between adjacent sections of the treated and untreated film.

The demonstrated significant dependence of the color difference of adjacent polypropylene film intervals on the location of the heat-treated film section between crossed polarizers can be used in the instrumental design of a new method for identifying thermochromic labeling of transparent polymer packaging.

Authors' contributions

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

A.P. Kondratov – writing the text of the article, scientific editing.

The authors declare no conflicts of interest.

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