Evaluation of Nonuniformity of Elastic Properties of Sheets Made from Closed-Cell Polyolefin Foams by Acoustic Method

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Received 19.12.2020
Accepted for publication 17.02.2021

Abstract

The widespread use of polyolefin foams in strategically important industries is due to their high thermal, sound and vibration insulation properties. The aim of the work was to evaluate the non-uniformity of elastic properties over the area of sheets of polyolefin foams of various types using the acoustic non-contact shadow amplitude method of testing and confirmation by the structural analysis method.

The article presents the developed installation and a new method of non-contact acoustic testing of sheets made of closed-cell polyolefin foams based on recording the amplitude of the pulse that passed through the sheet and allowing to assess to the unevenness of its elastic properties during scanning. Studies of uneven elastic properties were carried out on sheets of closed-cell polyolefin foams of the *ISOLON* 500 and *ISOLON* 300 brands which differ in material and manufacturing technology (technique of cross-linking, method and multiplicity of foaming).

It is shown that the absolute amplitude of the signal and its spread relative to the average value is affected by the structure of the foam polyolefin material and its heterogeneity over the area of the studied sheet determined by the production technology which is confirmed visually using microscopy.

Studies have shown the effect on the indications unevenness of the method of obtaining and the apparent density of the material. It is shown that the most uneven elastic properties and structure belong to sheets of polyolefin foam obtained by chemical cross-linking technology (the unevenness of Δ was 6.5 %). Among the physically cross-linked sheets of polyolefin foam the most uniform in structure and elastic properties are samples made of ethylene vinyl acetate with Δ = 3.8 %, as well as sheets with a high foaming rate (Δ = 3.9 %). The unevenness of structure of the studied sheets of polyolefin foams was confirmed by optical microscopy of sections in two mutually perpendicular directions.

Keywords: polyolefin foam, elastic properties, acoustic shadow method, unevenness, structure.

DOI: 10.21122/2220-9506-2021-12-1-58-66

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Для цитирования:
O.P. Bogdan, D.V. Zlobin, O.V. Murav'eva, S.M. Molin, A.V. Platunov.
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Приборы и методы измерений.
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Оценка неравномерности упругих свойств листов из закрытоячеистых пенополиолефинов акустическим методом

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Поступила 19.12.2020
Принята к печати 17.02.2021

Широкое использование пенополиолефинов в стратегически важных отраслях промышленности обусловлено их высокими тепло-, звуко- и виброизоляционными свойствами. Целью работы являлась оценка неравномерности упругих свойств по площади листов пенополиолефинов различных типов с использованием акустического бесконтактного теневого амплитудного метода контроля и подтверждением методом структурного анализа.

Разработаны установка и новая методика бесконтактного акустического контроля листов из закрытоячеистых пенополиолефинов, основанная на регистрации амплитуды импульса, прошедшего сквозь лист, и позволяющая оценить неравномерность его упругих свойств в процессе сканирования. Исследования неравномерности упругих свойств проведены на листах из закрытоячеистых пенополиолефинов марки *ISOLON* 500 и *ISOLON* 300, различающиеся материалом и технологией изготовления (способ сшивки, метод и кратность вспенивания).

Показано, что на абсолютную амплитуду сигнала и её разброс относительно среднего значения влияет структура материала пенополиолефина и её неоднородность по площади исследуемого листа, определяемая технологией производства, что подтверждено визуально с использованием микроскопии.

Исследования показали влияние на неравномерность показаний способа получения и кажущейся плотности материала. Показано, что наиболее неравномерные упругие свойства и структуру имеют листы из пенополиолефинов, полученных по технологии химической сшивки (неравномерность Δ составила 6,5 %). Из физически сшитых листов пенополиолефинов наиболее равномерными по структуре и упругим свойствам являются образцы, изготовленные из этиленвинилацетата с Δ = 3,8 %, а также листы с высокой кратностью вспенивания (Δ = 3,9 %). Неравномерность структуры исследованных листов пенополиолефинов подтверждена оптической микроскопией срезов в двух взаимно перпендикулярных направлениях.

Ключевые слова: пенополиолефин, упругие свойства, акустический теневой метод, неравномерность, структура.

DOI: 10.21122/2220-9506-2021-12-1-58-66

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Introduction

Polyolefin foam is a cross-linked gas-filled polymer material with a closed or open cellular structure. Polyolefin foams are featured by low values of density, thermal conductivity, water absorption, vapor permeability, high chemical resistance, flexibility and elasticity, sound-absorbing properties, resistance to temperature and mechanical influences, as well as environmental safety. The non-uniformity of the elastic properties of polyolefin foams can lead to the fact that the latter will not meet the requirements of heat and sound insulation and vibration damping which is unacceptable for particularly important strategic objects.

Technical requirements for the production of polyolefin foams are defined by GOST R 56729-2015 (EN 14313: 2009)) which involves selective testing of characteristics. The main methods of studying the sound absorption of polyolefin foams are tests in a reverberation chamber [1] (GOST 31704-2011 (EN ISO 354: 2003)) and tests in an impedance tube with two or four microphones [2, 3]. None of the methods regulated by the state standards allows us to assess the uneven properties of the produced sheets of polyethin foams.

Due to the fact that most of the characteristics of polyethin foams are related to their mechanical properties, the acoustic method of research is the most physically justified since it uses elastic vibrations of the same physical nature. Therefore the study of the acoustic properties of polyolefin foams is of particular interest to scientific schools. Most studies of the acoustic properties of polyolefin foams are limited to the study of sound absorption under various conditions and its dependence on porosity, apparent density, tortuosity of pores, and radiation frequency [1–9]. A number of studies [1–9] show the possibility of improving the acoustic characteristics (sound absorption, sound attenuation, and vibration damping property) by optimizing the structure and synthetic formula with various inorganic and organic fillers [3, 10–13]. The influence of porosity and tortuosity of pores on the speed of sound, Poisson's ratio, elastic modulus, and radiation frequencies is studied [14–17].

The questions of the uniformity of the distribution of acoustic properties over the thickness of polyolefin foams are evaluated using the simulation of the macroscopic foaming process [18, 19]. There is information about studies of the uneven acoustic properties in gas pipeline metals, which characterize the tendency to failure [20, 21].

The aim of the work was to evaluate the non-uniformity of elastic properties over the area of sheets of polyolefin foams of various types using the acoustic non-contact shadow amplitude method of testing and confirmation by the structural analysis method.

Materials and methods

The object of the study was sheets of polyolefin foam, which is a hydrophobic polymer material obtained by chemical or physical cross-linking. Chemical cross-linking of polyolefin foam is realized by appending chemicals into the material, while the sheet has a rough surface and a closed fine-pored structure (pore size < 1 mm). Physical (radiation) cross-linking of polyolefin foam is realized by irradiating the material with an electron beam, while the sheet after processing has a smooth surface and a closed microporous structure. The pore size of polyolefin sheets depends on the multiplicity of foaming K of the material, which occurs in a special vertical or horizontal furnace and determines the apparent density of the material ρ (TI 2244-037-00203476-2012 Cross-linked polyethylenes of the *ISOLON* 500 and *ISOLON* 300 brands):

$$\rho = \frac{N}{K},$$

where $N = 1000$ kg/m^3.

The greater the multiplicity of foaming, the larger the size of the pores in the sheets of polyolefin foam and the lower their apparent density.

Studies to assess the uneven properties of polyolefin foam by the shadow acoustic method were carried out on closed-cell samples of the *ISOLON* brand, manufactured according to TI 2244-037-00203476-2012 with dimensions of 180×150×4 mm. The samples are made of polyethylene foam and ethylene vinyl acetate obtained by different methods (physically and chemically cross-linked, foamed in a horizontal and vertical furnace). The properties of the studied sheets of polyolefin foams are presented in Table 1.

The estimation of the unevenness of the samples of polyolefin foams is realized using the contactless acoustic amplitude shadow transmission method [22, 23], based on the registration of changes in the amplitude of the acoustic signal that passed through the sample.

Table 1

Properties of the studied sheets of polyolefin foams

Grade	Stock	Type of cross-linking	A method of producing	Thickness, mm	Foaming multiplicity K	Apparent density ρ, kg/m^3
ISOLON 500 3004 *AV W*	polyethylene foam	physically cross-linked	foamed on a vertical furnace	4	30	33
ISOLON 500 1004 *AV W*	polyethylene foam	physically cross-linked	foamed on a vertical furnace	4	10	100
ISOLON 500 1504 *SV W*	ethylene vinylacetate	physically cross-linked	foamed on a vertical furnace	4	15	66
ISOLON 500 2004 *AH B*	polyethylene foam	physically cross-linked	foamed on a horizontal furnace	4	20	50
ISOLON 300 2004 *AH D*	polyethylene foam	chemically cross-linked	foamed on a horizontal furnace	4	20	50

The block diagram of the experimental setup for the study of uneven sheets of foam is shown in Figure 1. An electric pulse from the probing pulse generator 1 is fed to the radiating acoustic converter 2. The emitted acoustic wave passes through a sheet of polyolefin foam (object of study 3) and is received by an acoustic transducer 4, which converts it into an electrical signal. The registered electrical pulse enters the processing unit 5, in which it is amplified and its amplitude is recorded using a peak detector, the value of which is transmitted to the ADC of the microcontroller 6. Data from the microcontroller is transmitted to a personal computer 7, on the screen of which the measurement results are displayed using specialized software. Using a microcontroller 6, powered by a personal computer 7, the probe pulse generator 1 is also controlled.

A mandatory requirement is the use of identical emitting 2 and receiving 4 acoustic transducers located coaxially on opposite sides of the object of study 3. To minimize the influence of acoustic noise and interference on the measurement results, to increase the accuracy and reliability of the measurement, the object of study 3, emitting 2 and receiving 4 acoustic transducers are acoustically isolated using a sound-proof chamber 8.

Main characteristics of the developed experimental setup:

– informative parameter-the amplitude of the transmitted signal;

– type of acoustic transducer-W-06A;

– operating frequency range: 7–15 kHz;

– applied voltage to the emitter – 20 V;

– amplification constant- not less than 40 dB;

– method of excitation-delta pulse;

– protection against air acoustic interference;

– installation power supply-from the USB port of the main computer (+5 V);

– the distance between the emitter and the receiver – 35 mm;

– the diameter of the acoustic transducer is 40 mm.

To assess the unevenness of the elastic properties, a transverse-longitudinal scan of the sheet surface is performed through transducers with a step of 30 mm, with the exception of 15 mm at the edges in order to avoid the appearance of an edge effect.

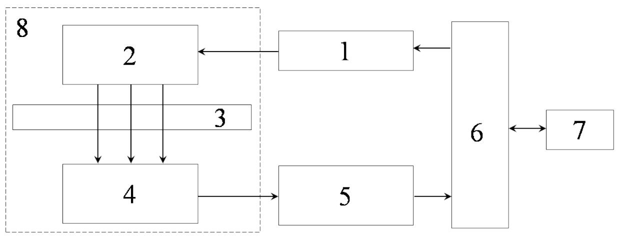

Figure 1 – Block diagram of the experimental setup: 1 – probe pulse generator; 2 – emitting acoustic transducer; 3 – object of study; 4 – receiving acoustic transducer; 5 – processing unit with amplifier and peak detector; 6 – microcontroller; 7 – personal computer; 8 – sound insulation chamber

The amplitude of the signal transmitted through the sheet of polyolefin foam depends on the structure and thickness of the object under study. In particular, for porous materials, acoustic energy losses will be affected by acoustic (elastic) properties (acoustic impedance, density, sound absorption, sound velocity, elastic modulus determined by the sheet structure – foaming coefficient, porosity, pore tortuosity coefficient, and other structural characteristics of the material [22, 23]. In this case, a local change in the elastic properties over the sample area leads to fluctuations in the amplitude of the transmitted signal during the scanning of the sheet. Thus, a local increase in the density of a sheet of polyolefin foam or a local decrease in the multiplicity of foaming (uneven foaming) leads to a decrease in the amplitude of the acoustic signal transmitted through the object relative to the average value.

In the case of an inhomogeneous porous layer, which includes a sheet of polyolefin foam, the coefficient of passage of an acoustic wave through the layer is more complex and can be described taking into account the Biot-Frenkel equation, the equation of the classical theory of elasticity for the case of two-phase media [16].

In addition to evaluating the uniformity of the properties of polyolefin foams, the developed method and experimental setup can be used to detect various defects in the production and operation of sheets, such as dents, cuts, foreign inclusions, and others [22].

Results and discussion

To assess the uniformity of the properties of the sheets of polyolefin foam during production, the amplitude of the transmitted signal through the object of study was measured. The measurements were carried out at 30 points over the area of the polyolefin sheet in two directions along the sheet (direction 1–6) and across the sheet (direction I–V). Average list penopoliuretana amplitudes of the transmitted signal are presented in Table 2. It is seen that the amplitude of the transmitted through the sheet signal is influenced by the type of stitching and the apparent sheet density at constant thickness of the sheet. For physically cross-linked sheets of polyolefin foam, a decrease in the amplitude of the transmitted signal is observed with an increase in the apparent density. Comparing sheets of polyolefin foam with the same apparent density (50 kg/m^3) obtained using a different type of cross-linking that affects the synthetic formula of the sample (physically cross-linked *ISOLON* 500 2004 *AH B* and chemically cross-linked *ISOLON* 300 2004 *AH D*), it can be concluded that chemically cross-linked polyolefin foam is more transparent to the acoustic wave, since it has a large amplitude of the transmitted signal in contrast to the physically cross-linked one, and therefore has worse sound-absorbing properties.

Table 2

The value of the average amplitude of the transmitted signal over a sheet of polyolefin foam

Grade	The average amplitude of the transmitted signal on the sheet $\bar{A}$, relative unit
ISOLON 500 3004 *AV W*	655
ISOLON 500 1004 *AV W*	236
ISOLON 500 1504 *SV W*	280
ISOLON 500 2004 *AH B*	262
ISOLON 300 2004 *AH D*	406

In order to make a comparative assessment of the degree of unevenness of the acoustic properties of the samples of polyolefin foam in a wide range of amplitudes, the measured values of the A_i amplitude of the transmitted signal were normalized by the average value of $\bar{A}$ for the studied sheet of polyolefin foam:

$$A_n = \frac{A_i}{\bar{A}}.$$

The results of scanning the studied sheets of polyolefin foam are presented in Figure 2 in the form of surfaces with isolines corresponding to a certain percentage of deviation from the average values. It can be seen that the most uneven in its pro-perties is the sheet of *ISOLON* 300 2004 *AH D* foam (Figure 2*e*) (chemically cross-linked foam, foamed on a horizontal furnace), for which the deviation of the normalized amplitude from the average value of the signal transmitted through the sheet reaches $\Delta = \pm 6.5$ %. In this case, the greatest deviation is observed along line I along the entire sheet (direction 1–6), as well as along line 5 in positions III–V.

The smallest deviation from the uniformity of pro-perties is observed in the samples *ISOLON* 500 3004 *AV W* (Figure 2*a*) $\Delta = \pm 3.9$ % (physically cross-linked foam, foamed on a vertical furnace), for which the value of $\Delta = \pm 3.9$ % and *ISOLON* 500 1504 *SV W* (Figure 2*c*) (ethylene vinyl acetate physically cross-linked, foamed on a vertical furnace, for which $\Delta = \pm 3.8$ %.

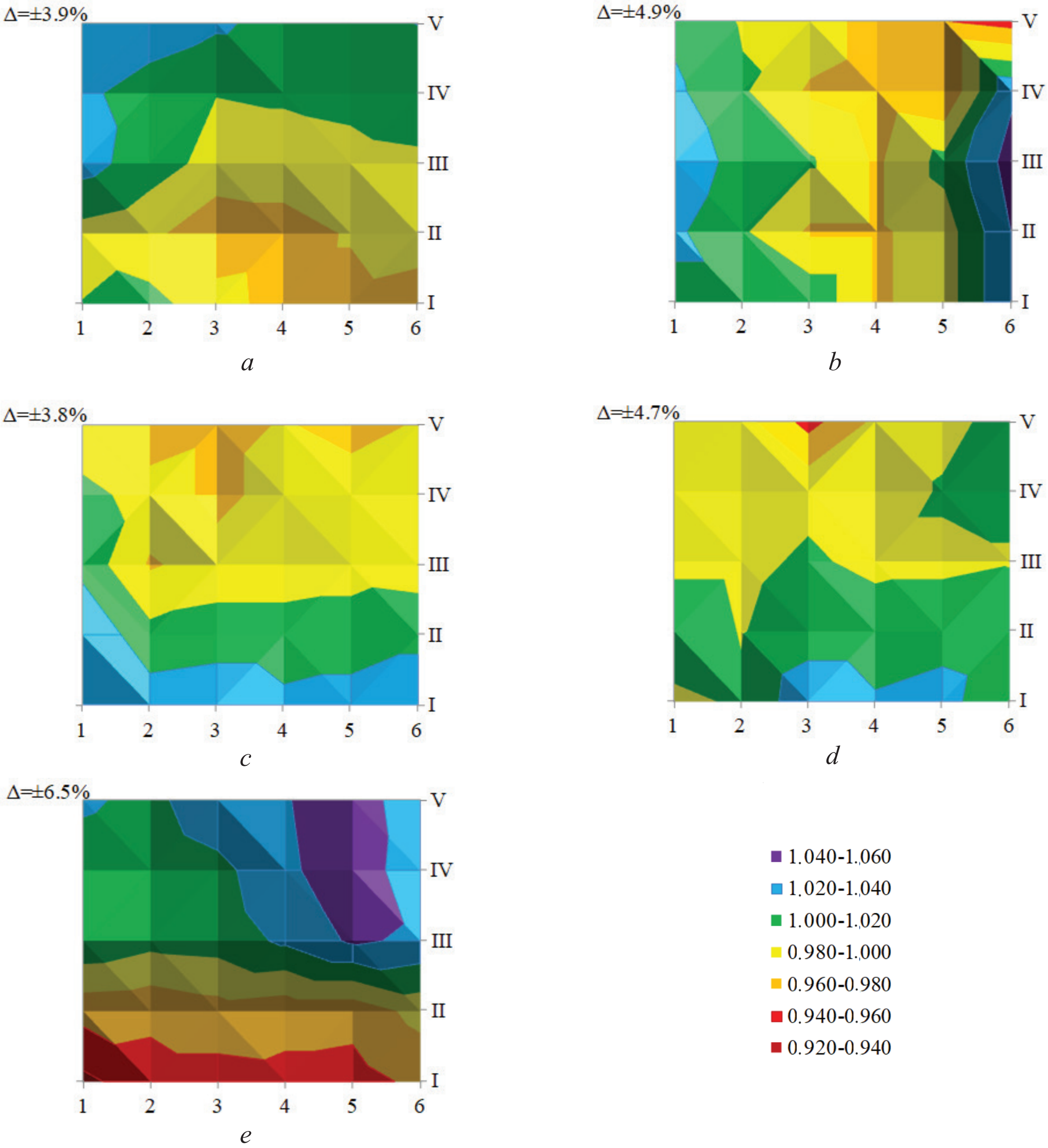

Figure 2 – Surfaces, describing the distribution of the normalized signal amplitude over the area of a sheet of polyolefin foam: *a* – *ISOLON* 500 3004 *AV W*; *b* – *ISOLON* 500 1004 *AV H*; *c* – *ISOLON* 500 1504 *SVJ*; *d* – *ISOLON* 500 2004 *A,B*; *e* – *ISOLON* 300 2004 *AH D*

Comparing the results of samples *ISOLON* 500 *AV* 3004 *W* (Figure 2*a*) and *ISOLON* 500 1004 *AV W* (Figure 2*b*), made of foam on one technology, but a different rate of expansion, and, consequently, the apparent density (Table 1), we can conclude that the increase in the multiplicity of foaming leads to a reduction in inequality in the worksheet, which made for a sample of *ISOLON* 500 *AV* 3004 *W* $\Delta = \pm 3.9$ %, and for *ISOLON* 500 1004 *AV* $\Delta = \pm 4.9$ %. This is due to the fact that at small values of the multiplicity of foaming, the spread of pore sizes in the sample is greater than at a large multiplicity of foaming.

The studied samples *ISOLON* 500 2004 *AH B* (Figure 2*d*) and *ISOLON* 300 2004 *AH D* (Figure 2*e*) are made of polyethylene foam foamed in a horizontal furnace, have the same foaming multiplicity of 20 and the same apparent density of 50 kg/m^3 (Table 1), but were produced using different cross-linking technologies (*ISOLON* 500 2004 *AH B* – physically cross-linked, *ISOLON* 300 2004 *AH D* – chemically cross-linked). All other things being equal, the chemical cross-linking of the polyethylene foam leads to an unevenness across the sheet of $\Delta = \pm 6.5$ %, while the physical cross-linking leads to $\Delta = \pm 4.7$ %.

To confirm the uneven foaming of the sheets of polyolefin foams, the structure was photographed using an Altami *MET* 1*M* metallographic microscope with a *WF*10*X*/22 mm eyepiece and a planachromatic

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lens at infinity *PL L* 5*X*/0.12 with an increase of 50x and an operating distance of 26.10 mm with the following shooting parameters: the method of contrast in reflected light-polarization (polarized light), illumination – bar with light filter (blue light filter for samples *ISOLON* 500 3004 *AV W*, *ISOLON* 500 1004 *AV W*, *ISOLON* 500 1504 *SV W*, yellow light filter for samples *ISOLON* 500 2004 *AH B*, *ISOLON* 300 2004 *AH D*), image resolution – 1024×822. Photos of the structure of the sheets of polyolefin foams are shown in Figure 3.

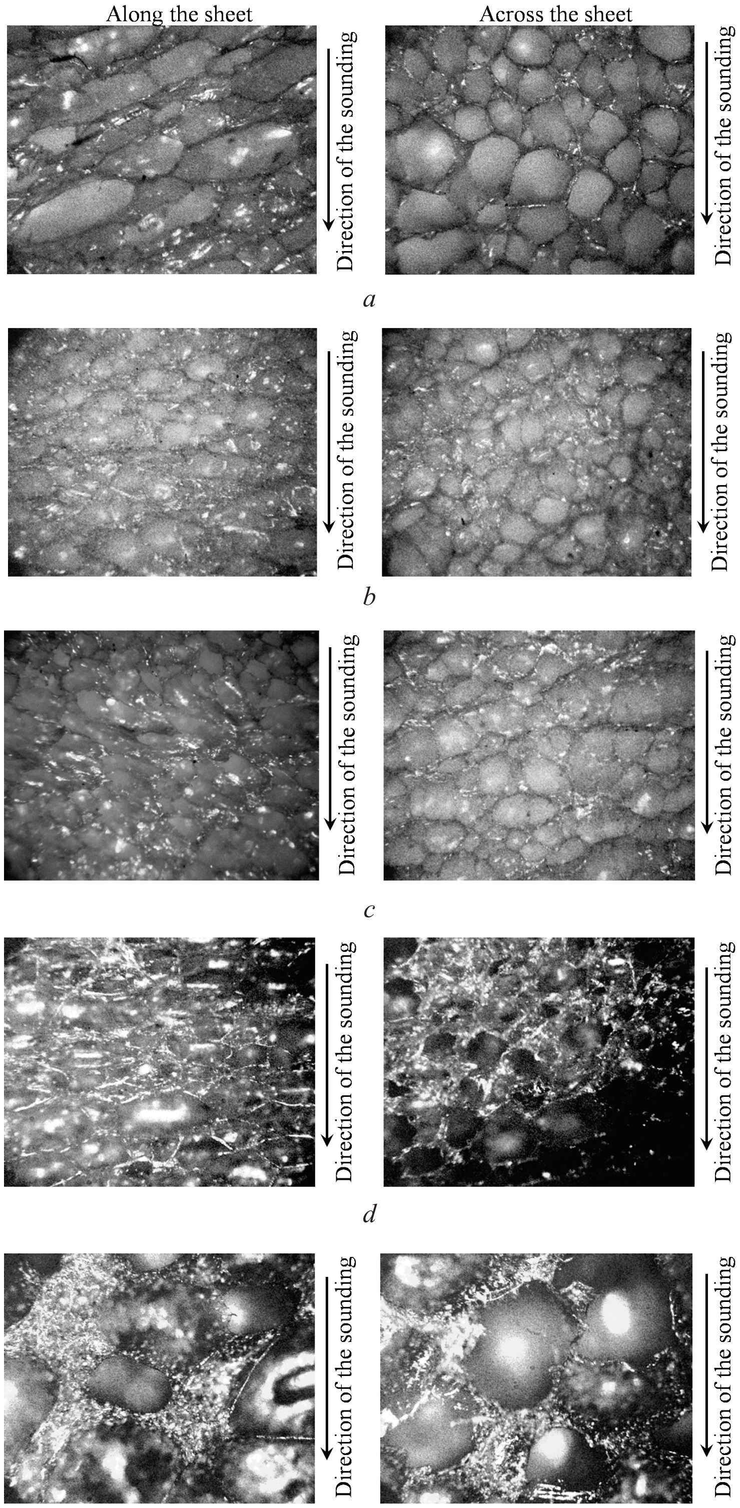

Figure 3 – Image of the structure of polyolefin foam sheets: *a* – *ISOLON* 500 3004 *AV W*; *b* – *ISOLON* 500 1004 *AV W*; *c* – *ISOLON* 500 1504 *SV W*; *d* – *ISOLON* 500 2004 *AH B*; *e* – *ISOLON* 300 2004 *AH D*

The study of the structure of the samples was carried out on sections which was made along the thickness of the sheet in two directions: along the sheet (direction 1–6) and across the sheet (direction I–V), while the direction of sounding of the sheet along the thickness is shown by an arrow. In all the samples studied, there is a clear orientation of the porous cells in the direction 1–6 along the sheet, which coincides with the direction of the rolled product during the foaming process. In this direction, the pores have an elongated shape, and the intercellular space conditionally has the form of channels, which contributes to better sound transmission along the sheet. In the direction I–V across the sheet, on the contrary, the porous cells have a more rounded appearance and sound transmission in this direction is difficult due to the absence of obvious channels in the intercellular space, which is typical for all the samples under study.

Analysis of the structure of the studied samples of polyolefin sheets in the direction of sounding (vertical image) shows that the size of the porous cells in the thickness of the sheet may differ several times for all samples. For sheets with minimal values of nonuniformity of amplitudes across the sheet $\Delta = \pm 4$ % (*ISOLON* 500 3004 *AV* (Figure 2*a*) and *ISOLON* 500 1504 *SV W* (Figure 2*c*)), it can be seen that the inter-cell space has a minimal spread in thickness. At the same time, the difference in the thickness of the space between the porous cells increases with an increase in the unevenness of the amplitudes along the sheet Δ. For example, for the sample *ISOLON* 500 1004 *AV W* (Figure 2*b*), it can be seen that the intercellular space around a small pore is thicker than near a large pore, this is due to the low value of the foaming multiplicity, as a result of which the pore growth did not have time to develop evenly over the entire thickness. For the samples *ISOLON* 500 2004 *AH B* (Figure 2*d*) and *ISOLON* 300 2004 *AH D* (Figure 2*e*), an increase in the difference in the thickness of the inter-cell space is also observed, especially for chemically cross-linked foamed polyethylene foam in a horizontal furnace.

Conclusion

A method and a device for assessing the uneven distribution of elastic properties of polyolefin sheets by the acoustic amplitude-shadow non-contact method have been developed.

It is shown that the absolute signal amplitude and its spread relative to the average value are affected by the structure of the polyolefin foam material and its heterogeneity over the area of the studied sheet, which is confirmed visually using microscopy. Studies have shown the effect on the unevenness of the indications of the method of production and the apparent density of the material. Based on the results obtained, it can be concluded that it is inappropriate to use chemically cross-linked sheets of polyolefin foam for strategically important areas of industry due to the large heterogeneity of properties, which can cause negative consequences. The lowest indicators of unevenness of the structure and elastic properties are the sheets of polyolefin foam obtained by physical cross-linking from a material of ethylene vinyl acetate with a low apparent density, and, consequently, a high foaming coefficient.

The developed method is quite simple to implement, has high reliability and reproducibility, and can be used not only to assess the unevenness of elastic properties, but also to detect various types of defects, including in production conditions, in order to prevent areas of the sheet with defects and a high degree of heterogeneity for further use.

Acknowledgments

The study was carried out with the financial support of the grant of the Russian science foundation No. 18-79-10122 (methodology and experimental setup, research results) and the Kalashnikov Izhevsk State Technical University within the framework of the scientific project No. PAV / 20-90-17 (structuroscopy of the studied samples) using the UNU "information and measurement complex for studies of acoustic properties of materials and products" (reg. number: 586308).

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