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Małgorzata SZYMICZEK

ULTRASONIC AND THERMAL TESTING AS A DIAGNOSTIC TOOL FOR THE EVALUATION OF CUMULATIVE DISCONTINUITIES OF THE POLYESTER-GLASS PIPES STRUCTURE

BADANIA ULTRADŹWIĘKOWE I TERMOWIZYJNE JAKO NARZĘDZIE DIAGNOSTYCZNEJ OCENY KUMULACJI NIECIĄGŁOŚCI STRUKTURY RUR POLIESTROWO-SZKLANYCH*

The aim of the work was to develop a methodology for evaluating the accumulation of discontinuities with the application of non-destructive methods. Commercial polyester-glass pipes produced by a method of helical filament winding were tested. The observed discontinuities were the result of post-production flaws, but first of all, the aging-fatigue degradation process. Evaluation of the degradation degree directly related to the process of discontinuities propagation was performed with the use of active thermography and ultrasonic inspection. Diagnostic characteristics were the heating and cooling rate estimated from the temperature distribution on the heat-activated surface. In the case of ultrasonic inspection, as the value of the diagnostics was assumed, transition time of ultrasonic wave was determined by the application of the echo method. Structural changes were indirectly determined on the basis of water absorption. It has been found that there is a correlation between the properties set out in the non-destructive testing and water absorption. The higher absorption, which indicates a greater number of defects, the lower the heating and cooling rate and transition time of ultrasonic wave.

Keywords: *ultrasound, thermovision, ageing-fatigue tests, polyester –glass composites, pipes.*

Celem pracy jest opracowanie metodyki oceny kumulacji nieciągłości metodami nieniszczącymi. Badaniom poddano handlowe rury poliestrowo-szklane wytwarzana metodą nawijania śrubowego. Obserwowane nieciągłości były efektem zarówno wad poprodukcyjnych, ale przede wszystkim procesu degradacji starzeniowo-zmęczeniowej. Oceny stopnia degradacji bezpośrednio związanej z procesem propagacji nieciągłości dokonano przy użyciu termowizji aktywnej oraz defektoskopii ultradźwiękowej. Charakterystyką diagnostyczną w przypadku termografii była prędkość nagrzewania i chłodzenia określona na podstawie rozkładu temperatury na powierzchni aktywowanej cieplnie. W przypadku defektoskopii ultradźwiękowej jako wielkość diagnostyczną przyjęto czas przejścia fali ultradźwiękowej wyznaczony metodą echa. Zmiany struktury pośrednio określono na podstawie chłonności wody. Stwierdzono, że istnieje korelacja pomiędzy własnościami określonymi w badaniach nieniszczących a chłonnością wody. Im wyższa chłonność, co świadczy o większej liczbie wad, tym niższe prędkości nagrzewania i chłodzenia oraz czas przejścia fali ultradźwiękowej.

Słowa kluczowe: *ultradźwięki, termowizja, badania starzeniowo – zmęczeniowe, kompozyty poliestrowo – szklane, rury.*

1. Introduction

The wide spectrum of applications with reference to polymer composites causes that they are exposed to many degradation factors which have significant impact on their functional properties. These are both environmental factors (e.g temperature, microorganisms or UV radiation) [1, 2, 5, 6, 8, 10, 12] and fatigue ones (e.g. the value of tensions, frequency) [4, 18, 19]. Synergism of the phenomena triggered by these factors leads to gradual material degradation, which is particularly important in the case of polymer material [18, 22].

Properties of the polymer constructional composites, including glass-polyester ones depend, among others, on characteristics of input materials (reinforcements, matrixes), their mutual, adhesive connection and the technology of production. If wrongly matched, even one of the factors, causes the decrease in composite properties. Relatively large impact on the quality of obtained composites has the technology of their production [9, 11, 13]. It has been proved that in the winding technology [11] wrongly matched tension force causes bad fiber supersaturation, which in turn leads to appearance of flaws like voids or discontinuities.

(*) Tekst artykułu w polskiej wersji językowej dostępny w elektronicznym wydaniu kwartalnika na stronie ***www.ein.org.pl***

Identification of changes appearing in composites has been widely researched. Studies of epoxy-carbon composite structure conducted by Tarlej [18] allowed to define subsequent stages, from cracks individual fibres, through micro-cracks matrix, breaking the adhesive bonds up to delamination. These are, first of all, the effects of composite fatigue degradation. Particularly importance for the observed degradation processes is the heat, which, according to the Arrhenius law [10], accelerates to large extent the rate of reaction. Combination of thermal effect with e.g. UV radiation or water, results in, among others, the release of free radicals or hydrolysis reaction. Water absorption, which indirectly allows the evaluation of degradation level, is determined by material, exploitation temperature and the working environment [5, 6, 15].

The evaluation of the degradation level with reference to composite materials makes the basis to assess the usefulness of given construction for exploitation. The methods described in scientific works [2, 12, 18, 19] cause the failure of the construction which is costly and may prove unjustified. Constant monitoring of the impact of degradation processes on every stage of their use, up to its failures, makes it possible to predict the time of construction safe usefulness. Such observation is possible thanks to non-destructive research methods e.g. ultrasonic, thermovision and radiographic, which allow non-invasive diagnosing of the composite construction condition. [3, 8, 17, 20, 23, 24].

Within the presented work, the evaluation of progress in composite degradation was conducted in accordance with the established methodology, which makes use of measurement of transition time of the ultrasonic wave as well as the rate of the heating and cooling (thermovision). The changes in structure were indirectly established on the basis of water absorption.

2. Experimental research

2.1. Research program

The experimental part of the work include the following research:

- ageing-fatigue,
- thermovision,
- ultrasonic,
- water absorption.

Ageing-fatigue research, the aim of which was to obtain the proper of composite pipes degradation level, were conducted in the thermostatic water bath at the temperature of 30°C, with the cycle of stress amplitude of 3 MPa with rectangular extortion and the time of 7 s (1,5s time of pressure increase, 2s time of maintaining the maximum pressure). The way of pipes assembly and sealing, described in [22] assured the elimination of longitudinal stress which is in compliance with the theory of exerting pipes without bottoms, on the basis of the Huber's hypothesis of reduced stress [7]. The designed system of mounting caused that a pipe was only subjected to hoop and longitudinal deformation, which is shown in Fig.1, with radial stress negligible. On the inside surface they amount to 7 MPa, while on the outside surface they equal zero. Hoop stresses at the maximum load of 7 MPa are 96.95 MPa and 89.95 MPa respectively. Under given load, the increase of a pipe diameter occurs as well a decrease of length of a tested pipe.

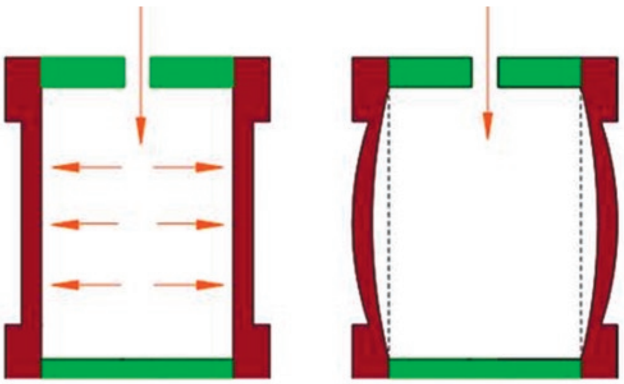

Fig.1. Scheme of the pipes deformation

Thermovision tests were conducted at the working station described in the work [17]. The changes of temperature during the heating and cooling were determined on the basis of thermovision tests by activating thermally the outside surface of a pipe for 60 s. The change in temperature in time, recorded by means of thermovision camera Flir A615, which was used to establish the rate of heating and cooling.

The transition time of the ultrasonic wave was determined in the aquatic environment with the use of heads Parametrics with the frequency of 2.25 MHz co-operating with the defectoscope UMT 17.

The research on water absorption, which indirectly allowed the evaluation of the structure condition, was conducted in accordance with the procedure described in the norm PN-ISO 8361-1:1994 [14]. The thermovision, ultrasonic and water absorption research were conducted successively, initially (up to 10×103 cycles) at 2.5×103 fatigue cycles, and the every 10×10^3 cycles.

2.2. Research material

The research was carried out glass-polyester pipes produced by winding method. The winding angle was 54°, with glass weight participation of 52%. For the matrix the composition of polyester resins Polimal 104 TS and Estromal 14.CNP -03/P was used. These are the resins containing flame retardant substances which make it possibile to obtain the flammability class V0 according to the norm PN-EN 60695. As the reinforcement glass roving ER 3003 was used with vinyl-silane aperture; as far as interlayers are concerned, the glass mat EMC 300 with silane preparation was applied. The endings of pipe samples were strengthened by hoop wound collars. The aim of the collars was to ensure the proper ring stiffness in the sealing places. Figure 2 presents the view of the sample with 16 measurement points marked 4 on each of forming, designated by its axial rotation of 90°.

Before the beginning of the ageing-fatigue research, all the samples were subjected to hardening process at the temperature of 60°C for 24 h. The result was to stabilize properties pipes.

a)

b)

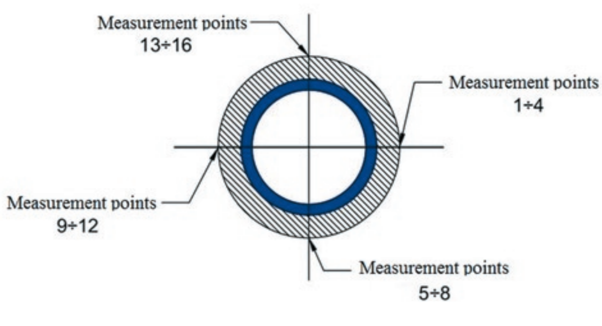

Fig. 2. View of the tested sample (a) schema of indicated measurement points (b)

The tests of water absorption were conducted on the samples in the shape of beams measuring 95×20×6 mm, cut out of the central part of pipes marked as C in the Fig. 2a.

2.3. The results and the analysis.

2.3.1. Ultrasonic tests

On the basis of the conducted ultrasonic tests, the transition time of the ultrasonic wave was determined in the marked measurement points (Fig. 2b) depending on the number of fatigue cycles. The changes in the transition time of the ultrasonic wave allow the evaluation of structural changes which occur during the process of ageing-fatigue degradation. It is much shorter in the areas in which discontinuities occur. Figure 3 presents the distribution of the transition time on the surface of the pipe (in accordance with Fig. 2b) On the circuit, the given measurement points were marked, with the transition time of the ultrasonic wave on the axis **y**. This is the standard diagram for the pipe subjected to fatigue load according to given parameters and $50x10^3$ fatigue cycles. The observed discrepancies for the sample not subjected to the process of ageing-fatigue degradation result from variable wall thickness of the pipe, which is connected with the production technology.

Figure 4 shows the change in transition time of the ultrasonic wave for two points 14 and 10, chosen on the basis of observed changes for the investigation characteristics. In point 14 a rapid decrease in the transition time occurs which confirm macrocracks. Point 10 shows slow, continuous shortening of transition time. The changes in transition time of the ultrasonic wave illustrate the progressive process of

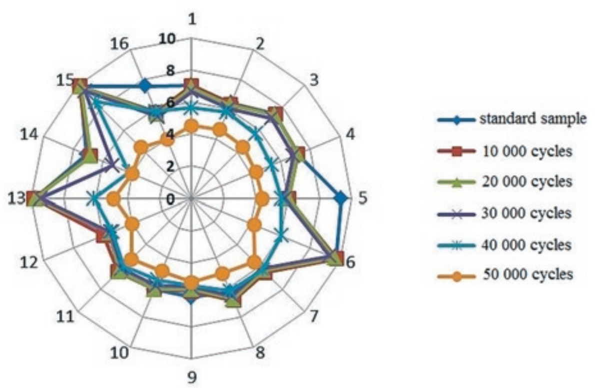

Fig. 3. Change of the transition time of the ultrasonic wave in the function of fatigue cycles

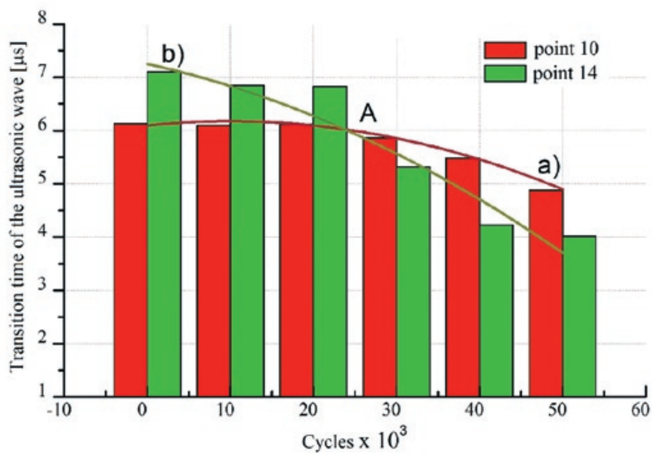

Fig. 4. Change of the transition time of the ultrasonic wave in the function of fatigue cycles in point a) 10 and b) 14 for a chosen pipe

ageing -fatigue degradation which manifests itself in microdefects in the first stage (minimal shortening of the transition time) being the potential area of cracks nucleation leading to delamination.

The changes in transition time of the ultrasonic wave are described by a second degree polynomial:

- curve a):

$$\mathbf{y} = 7.25 - 0.03\mathbf{N} - 7.34\mathbf{e}^{-4}\mathbf{N}^2 \tag{1}$$

with a correlation coefficient **R = 0,92**,

- curve b):

$$\mathbf{y} = 6.09 + 0.02\mathbf{N} - 7.99\mathbf{e}^{-4}\mathbf{N}^2 \tag{2}$$

with a correlation coefficient **R = 0,989**.

Relations 1 and 2 allow to obtain the best fit of approximation function for the research results. These are however, the significant relations for the tested glass-polyester wound pipes subjected to the ageing -fatigue degradation process within the established range.

As it can be observed in Fig. 3 and 4, in the first phase of the research, the transition time of ultrasonic wave does not change or it changes slightly. Developed curves a and b (Fig. 4) cross in point A i.e. about 30×10^3 fatigue cycles. The clear shortening of wave transition time correspond to structural changes observed, first of all as a result of fatigue degradation.

Due to the differences in the measured transition time, resulting from the thickness of samples (7±1 mm) and progressive destruction process, the analysis of the frequency in which the given value of transition time in the subsequent phases of degradation process appeared. Assuming the frequency function of occurrence of examined value to be in accordance with Gauss' distribution, the most common value was established. Such established value was taken as the basis to determine the relation between the transition time of the ultrasonic wave and the number of cycles. Figure 5 shows the function of given value occurrence frequency for the exemplary samples, not subjected to the ageing-fatigue process. It is described by the following equation:

$$y = y_0 + \frac{A}{w\sqrt{\pi/2}} e^{-2\frac{(x-X_c)^2}{w^2}} \tag{3}$$

where: $\mathbf{y_0 = 17.18; X_c = 3.51}$ **[µs]; w = 1.61 [µs]; A = 1378.75[µs]**.

correlation coefficient of the function described by relation 3 equals **R = 0.99**.

Together with the progression in the ageing-fatigue degradation process, the decrease in the correlation coefficient with reference to occurrence frequency function for given transition time of the ultrasonic wave value was observed (it complied with the Gauss' distribution as much as up R = 0.68 at the number of cycles 150×10^3) as well as its flattening, which confirms the increase in the results dispersion. The obtained values are still within the 30% dispersion range, which is acceptable in case of composites.

Figure 6 shows the relation of the transition time of the ultrasonic wave in the function of the number of fatigue cycles, determined in accordance with the above described procedure.

The results were approximated by exponential function, which, like in relations 1 and 2 is the best way to describe the transition time by the function of the number of cycles (correlation coefficient **R=0.95)**, in the following form:

$$\mathbf{t_u} = -0.41\mathbf{e}^{(-\mathbf{N}/-82,81)} + 3.92 \tag{4}$$

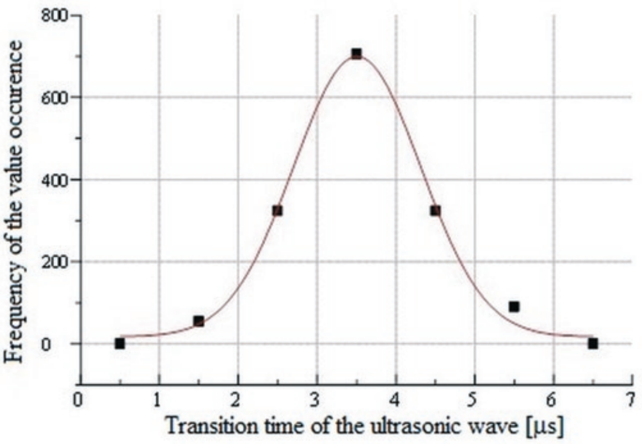

Fig. 5. Functions of occurence frequency with reference to the value of the transition time of the ultrasonic wave for the standard sample

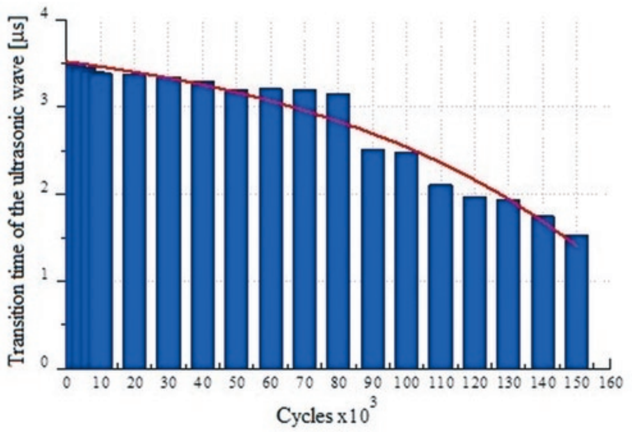

Fig. 6. Relation between the transition time of the ultrasonic wave and the number of fatigue cycles

where: t_u – transition time of the ultrasonic wave [μs], N – number of fatigue cycles.

As it can be observed in Fig. 6, in the first phase of degradation process, i.e. about 5×10^3 fatigue cycles, the transition time of the ultrasonic wave changes to a very limited extent. Micro-cracks that accompany the degradation process manifest moderate and even decrease in the transition time. More rapid change, in an individual case may be caused by the accumulation of discontinuities or delamination. In the diagram (Fig. 6) this effect is observed in the rapid form after about 80×10^3 number of cycles. The decrease is explained by the advancement level of the fatigue defects accumulation. Quantitative identification of established correlation may be assumed as diagnostic basis for the degree of exhaustion for the loading capacity of the examined materials.

2.3.2. Thermovision test

On the basis of thermovision temperature distribution on the external surface of the pipe obtained during the thermovision research (Fig. 7) the analysis was conducted which referred to the changes in the material condition, depending on the number of fatigue cycles. The analysis was conducted on the basis of designated changes in rate of heating and cooling. In figure 8, the pie charts of changes in the heating (Fig. 8a) and cooling rate (Fig. 8b) are shown for a chosen pipe subjected to the process of ageing-fatigue degradation after 30×10^3 of cycles. Area A, in which structural changes occur, was marked.

Figures 8 and 9 show the diagrams of changes in rate of heating and cooling observed in points 10 and 14 (clear cracks) of the same pipe. The results of its ultrasonic tests were presented in Fig. 3 and 4.

Obtained results were approximated by second degree polynomial. The changes in heating rate may be described:

- curve (a) in the area without delamination:

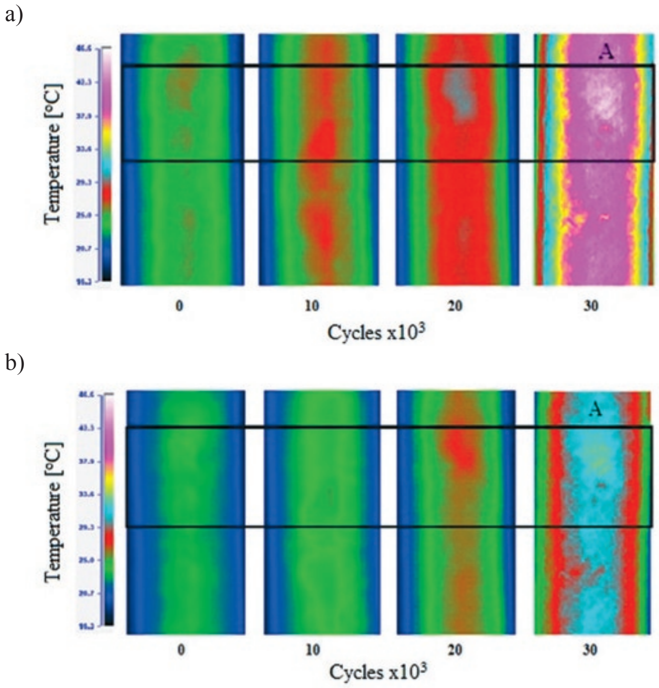

Fig. 7. Thermogram with the area of damage marked (A) in the course of (a) heating and (b) cooling process

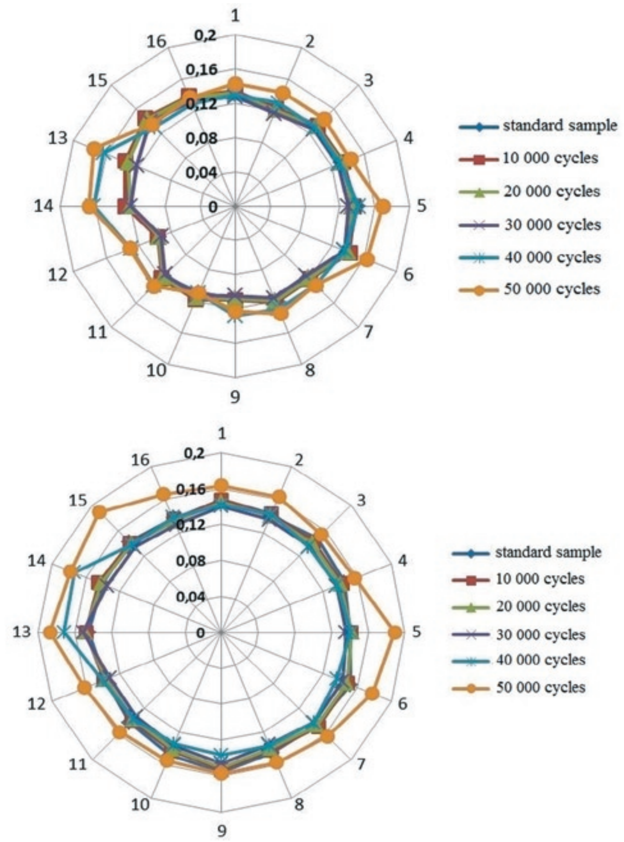

Fig. 8. Change of the heating a) and cooling b) rate, in the function of the number of fatigue cycles

a)

b)

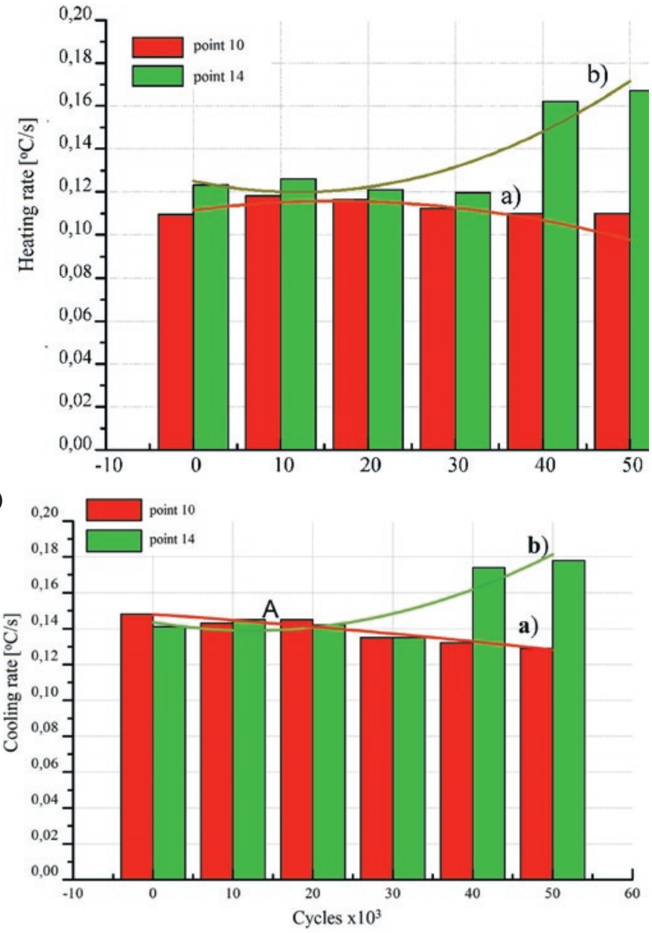

Fig. 9. Change of the heating a) and cooling b) rate, in the function of the number of fatigue cycles in points 10 (curve a) and 14 (curve b)

$$\mathbf{y} = 0.125 - 8.52\mathbf{e}^{-4}\mathbf{N} + 3\mathbf{e} - 5\mathbf{N}^2 \quad (5)$$

with the correlation coefficient R = 0.91.

- curve (b) in the area with clear macrocracks:

$$\mathbf{y} = 0.11 + 5.03\mathbf{e}^{-4}\mathbf{N} - 1.55\mathbf{e} - 5\mathbf{N}^2 \quad (6)$$

with the correlation coefficient R = 0.93,

The changes in the cooling rate are described as follows:

- curve (b) in the area without delamination:

$$\mathbf{y} = 0.15 - 2.77\mathbf{e}^{-4}\mathbf{N} - 3.17\mathbf{e} - 6\mathbf{N}^2 \quad (7)$$

with the correlation coefficient R = 0.99,

- curve (a) in the area with clear macrocracks:

$$\mathbf{y} = 0.14 - 7.43\mathbf{e}^{-4}\mathbf{N} + 3\mathbf{e} - 5\mathbf{N}^2 \quad (8)$$

with the correlation coefficient R = 0.88.

It was observed that the flaws in the shape of microcracks do not essentially influence the changes of examined thermal characteristics. As far as delamination and cracks are concerned, they result in rapid changes in both heating and cooling temperatures. It is mainly caused by the changes in thermal conductivity and specific heat, which in turn is indispensably connected with the appearance of structural flaws. In the quoted example, the areas (points 13-16, and 5-6) with clear cracks in composite layer could have been easily observed.

Due to discrepancies in the designated heating and cooling rate in the whole population of examined samples, in order to determine the value of given rate in the next phase of degradation, the analysis was conducted, concerning the frequency of occurrence with the described function, in compliance with Gauss' distribution. Figure 10 shows designated functions of occurrence frequency with reference to heating (Fig. 10a) and cooling (Fig.10b) rate for a pipe which was not subjected to degradation with coefficients respectively for a process:

- thermal activation: $\mathbf{y_0}$ **= 6.80986,** $\mathbf{x_c}$ **= 0.1179 [ºC/s], w = 0.01534 [ºC/s], A = 4.58613 [ºC/s].** Correlation coefficient equals R = 0.99,
- flow of heat after turning off the source of activation: $\mathbf{y_0}$ **= 9.04342,** $\mathbf{x_c}$ **= 0.17883 [ºC/s], w = 0.01706 [ºC/s], A = 4.3785 [ºC/s]**. Correlation coefficient equals R = 0.99.

The correlation coefficients presented in Fig. 10 were decreasing together with the progress of degradation process. It is a process similar to the one observed in case of the transition time of ultrasonic wave.

An important advantage of non-destructive thermovisual diagnostics is the posssibility of observing lad localize the flaws on the depth as early as at the stage conducting registration of temperature distribution. It is possible, among others, thanks to the temperature contrast principle [23]. The higher under the surface the flaw is located, the higher is the heating temperature. On the basis of the non-destructive thermovisual research conducted, the characteristics concerning the

a)

b)

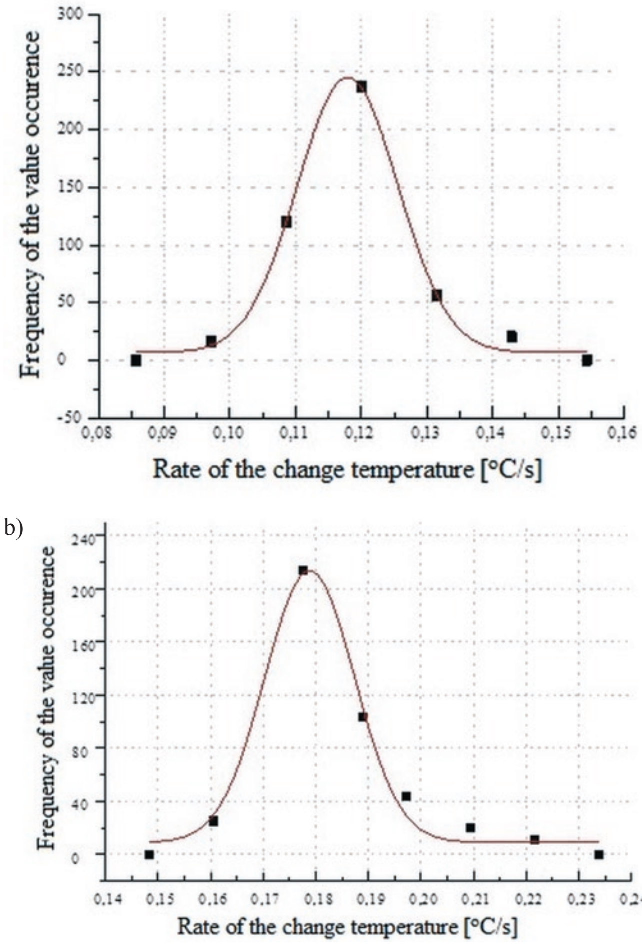

Fig. 10. Functions of occurence frequency with reference to the value of temperature changes in the process of (a) thermal activation and (b) cooling for the standard sample

changes in heating and cooling temperatures in the function of the number of cycles were created (Fig. 11). The dependence were approximated by the second degree polynomial, which has the following shape:

- for the heating phase:

$$\mathbf{v_n} = -1.36\mathbf{e}^{-6}\mathbf{N}^2 - 1.83\mathbf{e}^{-4}\mathbf{N} + 0,17 \tag{9}$$

with correlation coefficient **R = 0,97,**

- for the cooling phase:

$$\mathbf{v_{ch}} = -1.11\mathbf{e}^{-6}\mathbf{N}^2 - 1.43\mathbf{e}^{-7}\mathbf{N} + 0,12 \tag{10}$$

with correlation coefficient **R = 0,93.**

Higher rates were observed for the cooling process.

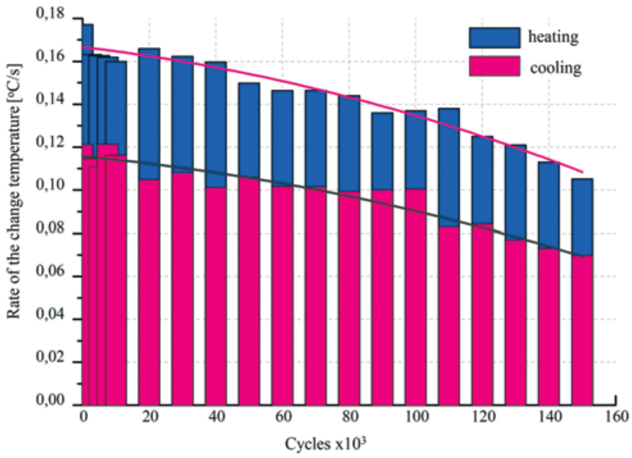

Fig. 11. Changes in heating and cooling rate in the function of the number of cycles

2.3.3. Water absorption research

The results of water absorption in the function of the cycles number is shown in Fig. 12. Water absorption in a given point is the average out of 15 values after rejecting the extreme ones. As it can be observed water absorption increases with the progress in the propagation of damage, which results from fatigue degradation.

The results were approximated by the exponential function in the following form:

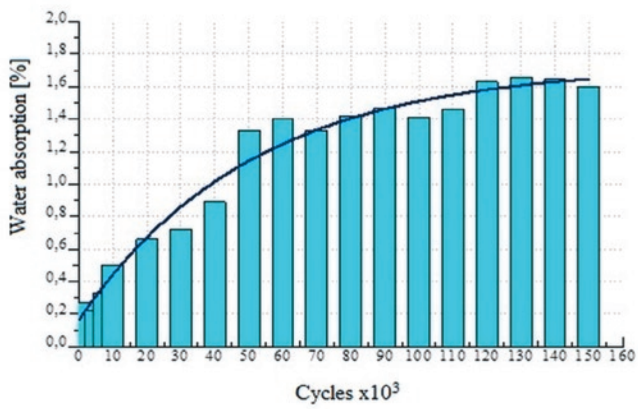

Fig. 12. Dependence of water absorption on the number of cycles

$$\mathbf{W} = 1.67 - 1.51\mathbf{e}^{-\mathbf{N}/46,66}, \tag{11}$$

with correlation coefficient R = 0.98.

The observed changes in water absorption may indirectly characterize the condition of the composite layer structure. The more defects within the volume of material, the higher is the water absorption, which was published, among others in work [21]. Microcracks appearing as a result of gradual joining, lead to creating bigger discontinuities which fulfill deformation criterion FPF (First Ply Failure) [1]. The accumulation of dissipated microcracks may lead to the cracks propagation which violate composite continuity. It is important from the point of view of strength properties of the tested material.

The differences observed in the course of researched characteristics result from the adopted methodology of research. In case of water absorption, clear increase in absorption value was notified for 50×10^3 of fatigue cycles (Fig. 12). It is caused by the deformation condition of pipes during ageing-fatigue research in accordance with Fig. 1. The samples were taken from the area with the largest deformation (Fig. 2a), and in connection with that, the biggest structural changes were notified, which are the effect of microcracks creating capillaries absorbing water.

In case of ultrasonic research, conducted with the use of the echo method, the ultrasonic wave deflects from the first discontinuity encountered. Taking into account the fact that the largest hoop stresses appear on the internal surface of the pipe, in compliance with the Huber's hypothesis, the destruction of layers occurs at that side. The clear shortening of transition time of the ultrasonic wave at 80×10^3 fatigue cycles is the effect of the flaws appearing from the side of internal surface (Fig. 6). This method however, requires the access to the examined object. The results of the thermovision research confirm the structural changes in the material, however, the point of clear decrease in the heating or cooling rate cannot be decidedly determined (Fig. 11). Macro-defects cause significant increase in the rate of heating or cooling (Fig. 9).

4. Conclusions

On the basis of the conducted research we can conclude that:

1. The transition time of the ultrasonic wave was in decrease together with the number of fatigue cycles. The applied echo method, taking advantage of longitudinal wave, allows identification of discontinuities appearing in plane which is perpendicular in relation to the direction of ultrasonic wave propagation.
2. Changes in the heating and cooling rate, thermovisually registered (by the reflection method) in the adopted measurement conditions were in decrease together with the increase in the number of cycles. The main cause of such a phenomenon is the process of discontinuities accumulation which leads to delamination. The structural changes cause lowering of composite thermal properties (e.g. thermal conductivity coefficient).
3. In the area of macro defects the rapid shortening of ultrasonic wave trasition time occurs, which is not observed in case of micro discontinuities appearing gradually. Both the heating and cooling rate in the area of microdefects are in decrease. The process of microcracks accumulation leading to delamination in the final stage, results in the rapid increase of measured thermovision values.
4. Distribution of measured thermovisual and ultrasonic values on the pipe circuit (Fig. 3 and 8) are comparable and allow identification of the areas exposed to destruction.
5. For the appropriate evaluation of the polymer composites degradation level, non-destructive tests should be conducted by means of at least two methods, and the results should refer

to standard sample, not subjected to the process of ageing-fatigue degradation.
6. The effect of progressing ageing-fatigue process of composite pipe shells were discontinuities in the structure, which resulted, first of all, fatigue load. Those defects influenced the increased water absorption in the examined composite. Water absorption in the final stage amounted to about 1.6%.

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Małgorzata SZYMICZEK
Department of Theoretical and Applied Mechanics
Silesian University of Technology
ul. Konarskiego 18A, 44-100 Gliwice, Poland
E-mail: malgorzata.szymiczek@polsl.pl