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Non-destructive diagnostic methods of polymer matrix composites degradation

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Analysis and modelling

ABSTRACT

Purpose: The aim of this paper is to present results of application of ultrasonic and termovision techniques to non-destructive evaluation of the degree of thermal degradation of fibre reinforced polymer composites. a model describing heat transfer taking place during Thermographic tests were undertaken to identify thermal properties of searched material and to correlate them with operational characteristics. In the same manner ultrasounds propagation characteristics were correlated with strength properties.

Design/methodology/approach: Epoxy-glass composites were exposed to thermal ageing and subsequently tested using thermography and ultrasounds. Finally destructive bending test was performed. Material characteristics evaluated in these test were compared and related to elaborate diagnostic relations.

Findings: The most essential result of the project is the methodology of applying thermovision and ultrasonic testing to diagnose the state of thermal degradation of polymeric composites.

Research limitations/implications: Results showed the possibility of non-destructive diagnosis of the degree of thermal degradation manifested by strength capacity deterioration of wide class of materials, namely polymeric composites.

Practical implications: Results of presented project together with results of planned experimental programme devoted to elaboration of diagnostic relations enable to apply thermography and ultrasonic testing directly to the state of polymeric structural materials assessment. Especially the degree of material degradation may be estimated.

Originality/value: Originality of the project is based on possibility of practical application of the thermovison and ultrasonic testing to non-destructive diagnosis of kinematics of degradation processes.

Keywords: Thermovision; Ultrasonic testing; Polymer composite; Diagnostics; Degradation

1. Introduction

In the case of polymers and polymeric composites subjected to degrading ageing processes, changes of surface appearance can not considered as a measure of the degree of material degradation. Usually surface brightness and colour changes occur within very thin outer layer and considerably precede any inner degradation processes. When degradation occurs in a dispersive way within the whole element volume, without any visible substantial external changes of physical and geometrical properties or when surface changes do not correspond to the degree of internal degradation processes, a classical inspection of a structure condition may not reveal any dangerous conditions. Therefore, in these cases there is a strong need of searching non-destructive methods of investigation of the degradation degree, which will be able to reveal these dispersive changes. Authors made such an attempt using non-destructive thermography technique and ultrasonic method with reference to thermally aged polymer composites.

The effectiveness of these two methods, based on two different physical phenomena and applying different characteristics evaluated in the experiment, was checked.

The first one is based on hypothesis that unambiguous relationship between changes of strength and thermal properties exists. Thermal properties influence temperature distribution evaluated in non-destructive thermographic method and being the result of activated thermal processes. Evaluated time and space temperature characteristics form the basis of the degree of degradation diagnosis.

The second one applies ultrasonic diagnosis methodology. A hypothesis was made that acoustic properties and strength properties are unambiguously related. Changes of strength properties due to thermal degradation evoke changes of acoustic properties evaluated in ultrasonic testing.

The common advantage of both methods is their non-destructive nature what makes them useful for diagnostic systems applied to composite structures in working conditions. Their dissimilarity makes it possibly to chose the one more accurate and easier applicable for given object and in given working environment.

Thermography is more and more widely used as non-destructive testing method of materials and among them polymers and polymeric composites [1-9]. Thermovision diagnosis makes use of time and space characteristics of surface temperature distribution of searched object. The surface temperature values and distribution may be a result of thermal processes taking place in the object in working conditions (passive thermography) or may be a result of thermal activation caused by researcher (active thermography). In the presented project the second case was described where thermal process was activated in composite samples by heating them using infra-red radiator. The purpose of all diagnosis techniques is to evaluate hazardous changes of different object properties, especially strength and rheological characteristics, due to various degrading processes [10-12].

Application of ultrasonic methods for testing of polymers and polymeric composites has a long-lasting tradition [13]. Taking into account effects of wave reflection, refraction, absorption, dispersion or defraction, diagnostic methods allow measurements of thickness, hydrolocation or local heterogeneities and discontinuities – flaw detection [14-16]. Less information is available on application of ultrasonic testing of degradation processes resulting in dispersed micro-defects such as micro-cracks, polymer chain scission, oxidation and many other. In the paper an attempt of this kind of ultrasounds application is described

2. Thermovision investigation of polymer composites thermal degradation

In this part of research the following methodology was applied:

a) Samples of epoxy-glass composite were subjected to thermal ageing thus causing their degradation;
b) Physical model of heat transfer in sample heated and cooled in thermovision testing stand was developed;
c) On the basis of physical model, heat transfer numerical model and computational programme was elaborated;
d) Degraded composites were tested using active thermography;
e) Experimental results of temperature distribution and its time alternation were compared with flexural strength deterioration due to ageing.

2.1. Physical model of heat transfer process

The model of heat transfer in composite sample mounted in thermographic testing stand and assumed boundary conditions are schematically shown in Fig. 1. Because of sample symmetry only one half was modelled and is presented. The heat transfer process taking place through test piece is non-stationary. It is the result of continually changing conditions of heat exchange during testing and unstable thermal state of test piece in both stages of testing process. Two characteristic phases of thermal process taking place in the test piece and composing the experiment were:

a) thermal activation of test piece by IR heating;
b) cooling down of test piece.

In the described project experimental schedule assumes the following possibilities:

a) thermal activation of test piece in the constant and strictly defined conditions;
b) thermovision registration of surface temperature distribution being the result of activation process;
c) finding the temperature distribution characteristics highly correlated with functional characteristics of tested material;
d) elaboration of empirical diagnostic functions relating temperature distribution characteristics and functional properties of searched materials.

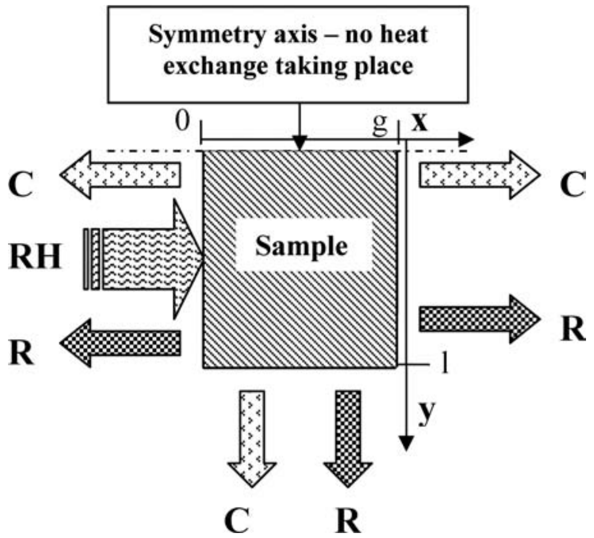

Fig. 1. The modelled half of sample cross section together with boundary conditions; RH – heater radiation flux, C – convection flux, R – sample radiation flux

Assumption stated in (b) is related with technical conditions of temperature registering with thermovision camera and also with accessibility of those surfaces of the object that are the most effective, taking into account the purpose of the research programme. Assumption (c) states the possibility of selection, among infinite number of time-space temperature distribution characteristics possible to determine in given experimental conditions, the one that is correlated with diagnosed material property in a manner enabling formulation of diagnostic function according to the assumption (d). The basic condition of effectiveness of elaborated method is fulfilment of the assumption (a). It means that experimental conditions must be such that it will be possible to separate changes of material properties analyzed in diagnostic procedure from other factors influencing chosen thermographic characteristic.

The beginning of the activation process is determined by initial thermal state of test piece. It is the thermal equilibrium state of the test piece placed in environment. The temperature is the same within all volume of test piece and equal to environment temperature. Together with the test piece exposition to the heater radiation begins the process of heat exchange between the test piece and environment. The dominant form of heat exchange, taking into account great temperature difference between heater and test piece surface, is radiation. Instantaneous heat flux absorbed by the test piece is the result of thermal balance of the heat flux reaching the surface of the test piece and going from the radiator and heat flux resulting from heat exchange between the test piece and environment [17-19].

2.2. Experiment of thermovision degradation process diagnosis

Epoxy-glass laminate (TSE-5, IZO-Erg, Poland) was subjected to thermal ageing. Samples with dimensions 250x20x10mm were aged at three temperatures: 200, 220 and 240°C. Samples were degraded up to 2600 hours. Aged samples were subsequently tested with thermovision camera. Schematic draw presenting thermovision test stand is shown in Fig.2.

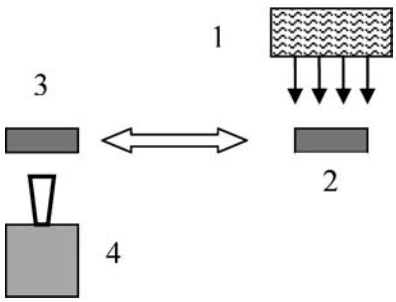

Fig. 2. Termovision test stand: 1 – IR heater, 2 – the sample in heating position, 3 – the sample in temperature scanning position, 4- thermovision camera

The constructed test stand consists of:

- Clamping device with test piece,
- Heat activator (IR radiator),
- Thermovision camera,
- Auxiliary equipment enabling constant experimental conditions (shields, slides etc.).

Thermovision camera was used to observe heat flow process activated in sample by IR heater with constant surface temperature. The heater and the sample were mounted vertically at constant distance. The purpose of the project was to define experimental conditions for which searched diagnostic relation was the most explicit. It was assumed that diagnostically effective thermographic characteristic had to be defined for changing in time temperature distribution measured at surface opposite to heated one. This distribution was among others affected by material thermal properties, defined in cross section of sample, which on the other hand are affected by degrading processes being the result of ageing. The starting moment of temperature distribution evaluation was determined by time of heating and time needed to rearrange testing stand from heating to temperature scanning position. Examples of thermovision images registered 30 and 165 seconds after the end of heating for samples aged 1992 hours at 200 °C are shown in Fig. 3 and Fig. 4.

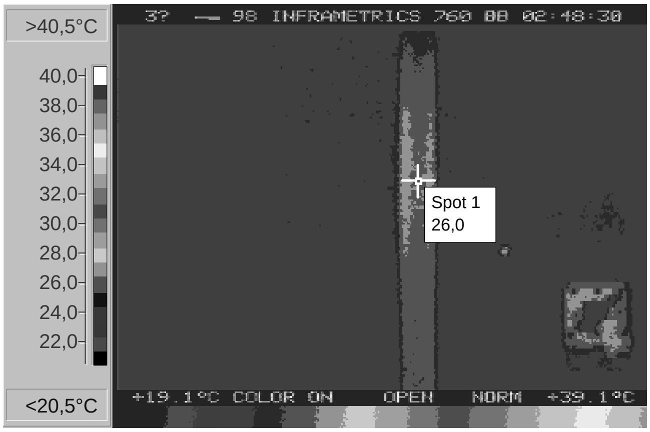

Fig. 3. Thermographic image of sample aged 1992 hours in 200 °C captured 30 seconds after the end of heating

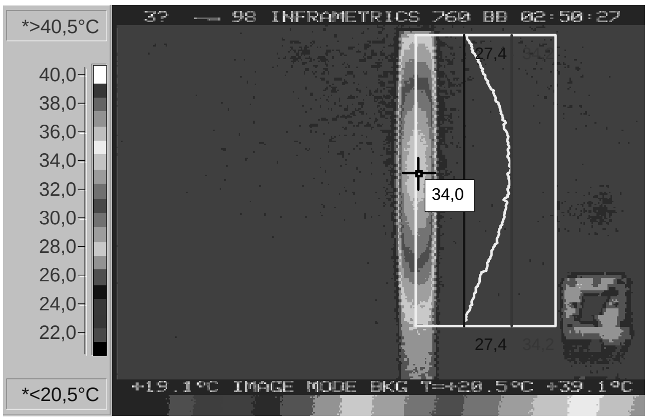

Fig. 4. Thermographic image of sample aged 1992 hours in 200 °C captured 165 seconds after the end of heating

The images were taken every three seconds and registered by computer being a part of thermovision camera equipment. A special software in this computer enabled temperature distribution evaluation. For the purpose of described research the temperatures at the central point of the sample was the most important. These temperatures allowed to plot curves describing temperature increase at this point with time. Examples of such curves are presented in Fig. 5 and 6 for two temperatures of ageing: 200 °C and 240 °C. Lower temperatures registered for samples aged at 240 °C are clearly visibly.

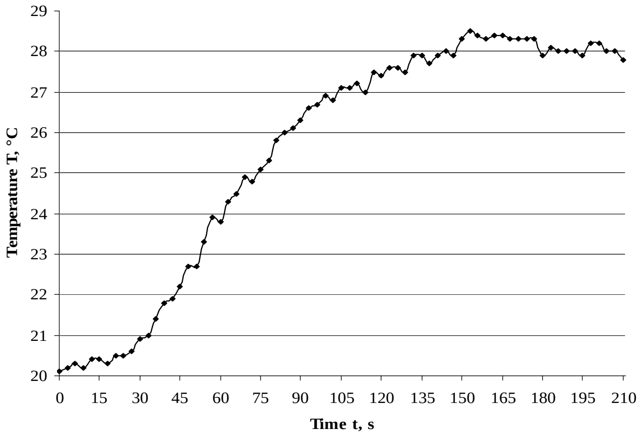

Fig. 5. Temperature – time dependence for central point of the sample aged 1992 hours in 200 °C

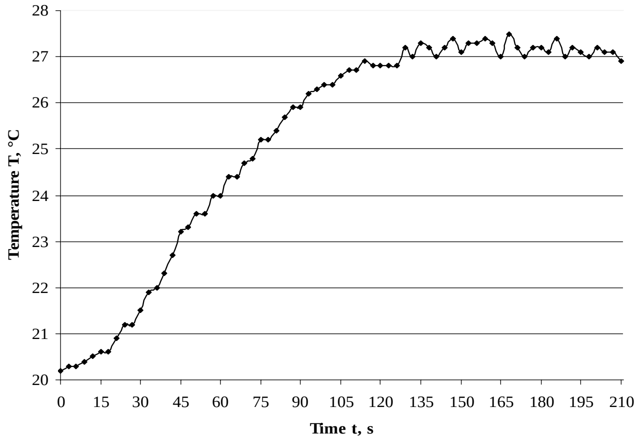

Fig. 6. Temperature – time dependence for central point of the sample aged 1992 hours in 240 °C

In presented methodology very important was experimental conditions constancy and repeatability. This repeatability of conditions refers mainly to:

- Heater surface temperature;
- Heater position;
- Time of radiation;
- Moment of the beginning of temperature registration;
- Geometry and physical properties of samples and their surface (roughness, thermal emissivity);
- Position and measuring range of thermovision camera;
- Test stand environment.

Fulfilment of above mentioned repeatability conditions allows to expect that differences in thermal processes registered by thermovision camera were the result mainly of the changes of material thermal properties of tested sample. If these changes were the result of structural degradation of tested polymer or composite then the hypothesis that thermographic characteristics are correlated with material characteristics seems to be justifiable.

Conditions of samples testing were in accordance with section 2. Constant heating time (20 seconds) was applied for all samples. Samples were heated with infrared radiator (Elstein HTS/2) with 500W thermal power. Thermovisionc images were registered using Inframetrics 760 camera. On the basis of thermographic images the temperature of central point of the surface opposite to heated one was determined.

Presented model was applied to evaluate the influence of structural changes caused by degradation on chosen diagnostic thermographic characteristics. Decrease of main strength characteristics, static, dynamic and fatigue, is the most pronounced manifestation of degrading processes advancement. Results of mentioned degrading processes are of microscopic scale and are dispersed in whole volume of the object. Structural changes take place in polymeric matrix and in interface between polymer matrix [20]. They bring to micro-cracks formation and propagation. Micro-cracks facilitates aggressive media penetration and further accelerate degradation. Finally reinforcing particles (fibres) can be damaged. All these imperfections and discontinuities are supposed to influence thermal properties of composite. Especially thermal conductivity and specific heat of the composite are expected to be influenced by matrix degradation ,micro-cracks, adhesion loss and other discontinuities.

Taking into account the above mentioned problems it is reasonable to compare changes of thermovision characteristics influenced by thermal conductivity and specific heat and strength properties deterioration.

On the basis of the temperature increase curves analysis (like this presented in Fig 5 and 6) the rate of temperature increase and the time of temperature stabilization were evaluated. The time of thermal ageing was related with these thermovision characteristics. The best correlation was achieved between ageing time and the time of temperature stabilization, presented in Fig. 7.

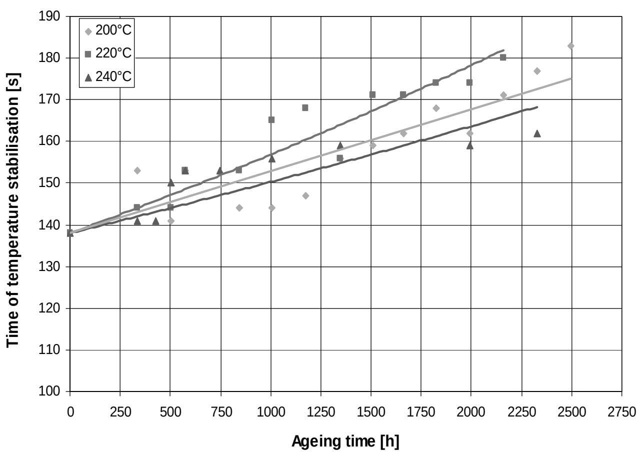

Fig. 7. Relationship between ageing time and the time of temperature stabilization

Aged samples were also subjected to flexural strength tests. The relationship between flexural strength and ageing time is presented in Fig.8.

Relation between flexural strength and time of temperature increase was evaluated (Fig. 9)

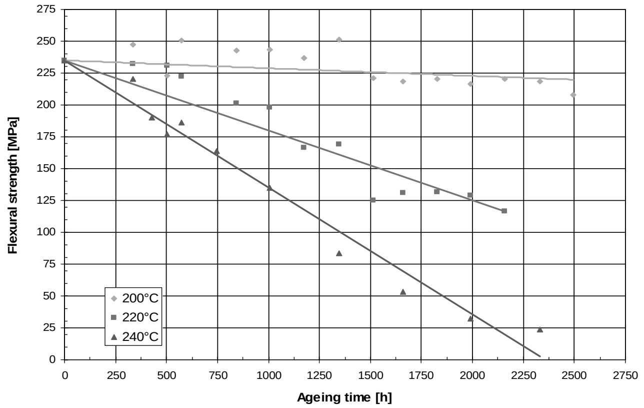

Fig. 8. The influence of ageing time on flexural strength

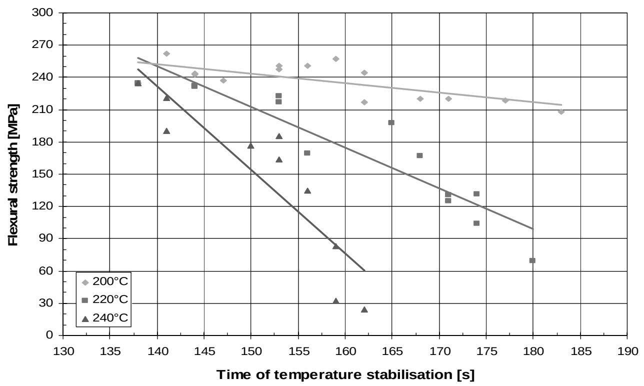

Fig. 9. Relation between flexural strength and time of temperature increase

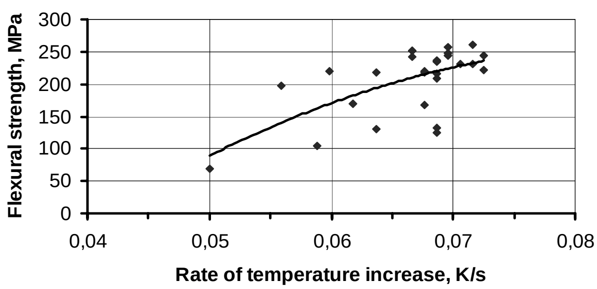

Fig. 10. Relation between flexural strength and rate of temperature increase

Finally relation between flexural strength and the rate of temperature increase for all samples was compared and shown in Fig.10. As can be seen it may be used as a basis of non-destructive thermovision evaluation of the degree of strength degradation of the composite.

3. Ultrasonic investigation of polymer composites degradation

In this part of experiment the following methodology was applied:

a) Samples of epoxy-glass composite were subjected to thermal ageing thus causing their degradation;
b) Degraded composites were tested using ultrasonic methodology;
c) Experimental results of ultrasounds propagation characteristics were compared with flexural strength deterioration due to ageing.

3.1. Experimental investigation system

In order to compare both non-destructive methods the same set of samples was used as in thermography experiment.

At defined intervals, the group of heated samples was subjected to non-destructive tests on the ultrasonic test station, thus identifying the values of diagnostic characteristics of ultrasounds wave propagation and to destructive tests in static bending in order to evaluate the strength properties deterioration. Samples with the same dimensions 250x20x10mm were used. Ultrasonic tests were carried out with an ultrasonic defectoscope UMT-16 (ULTRAMET S.c., Poland) help. Single 2,25 MHz measuring head was used. The scheme of testing stand is shown in Fig.11.

A time of a sound wave transition through tested samples expressed in µs was measured. Sound wave propagation velocity through a sample was next calculated taking into account measured time and specimens thickness.

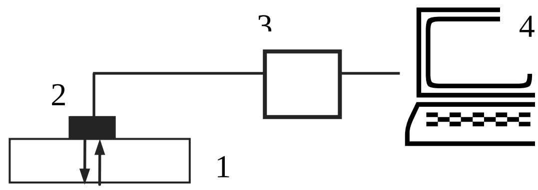

Fig. 11. Ultrasonic testing stand: 1) specimen, 2) ultrasonic head, 3) defectoscope, 4) computer system with software to results registering and analysis

3.2. Results and discussion

Results of ultrasonic tests for all aged samples are presented in Fig.12. For all ageing temperatures increase of ultrasound velocity with ageing time was observed. During aging composites became more stiff and it resulted in higher ultrasound wave propagation velocity. For highest ageing temperature and for longest ageing times brittleness of composite was observed. Composites aged in these conditions exhibited the highest velocities of ultrasounds propagation. It is in accordance with generally observed relation between material stiffness and ultrasounds velocity in all materials.

Fig. 13 presents results of flexural strength tests performed on aged samples. As can be seen the higher was ageing temperature the lower was flexural strength. It is generally observed tendency in thermal ageing investigations. In thermal ageing process two main factors, heat and oxygen, influence composites properties. Degrading processes results in many defects not visible in macro-scale such as micro-cracks, polymer chain scission, oxidation, free radicals formation and many other that substantially decrease mechanical strength.

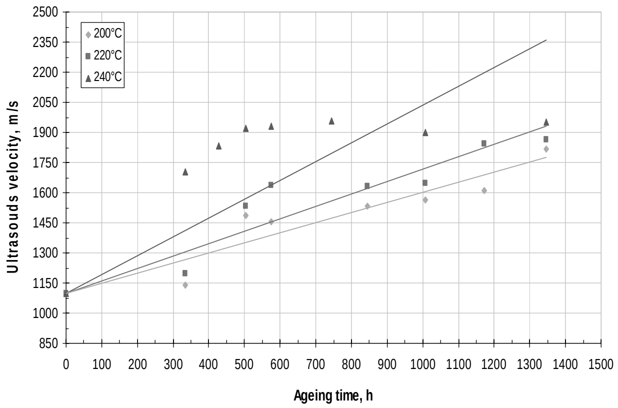

Fig. 12. Dependence of ultrasound wave propagation velocity on ageing time and temperature

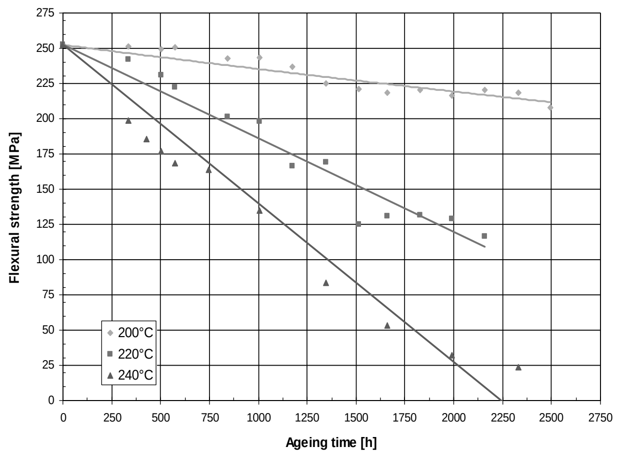

Fig. 13. Dependence of flexural strength on ageing time

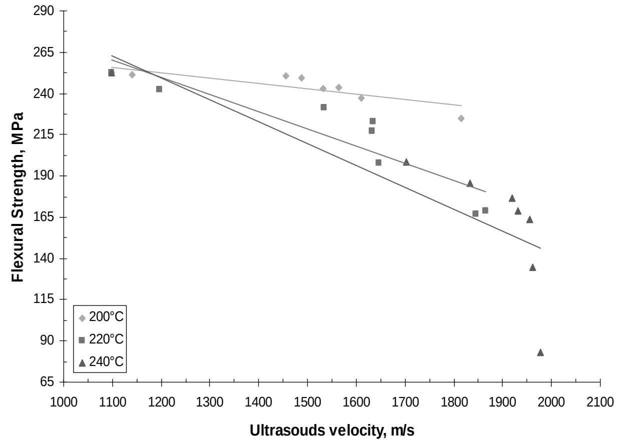

Fig. 14. Relationships between flexural strength and ultrasounds propagation velocity

Relationships between flexural strength and ultrasounds propagation velocity are shown in Fig.14.

Finally the most interesting relation between flexural strength and ultrasounds velocity for all samples together is given in Fig.15.

Presented results proved the possibility of applying non-destructive ultrasonic method to diagnosis of the state of thermal degradation of polymeric composites. Strong correlation between mechanical strength and ultrasonic wave propagation velocity was observed. Together with strength lowering ultrasounds propagation velocity decreased.

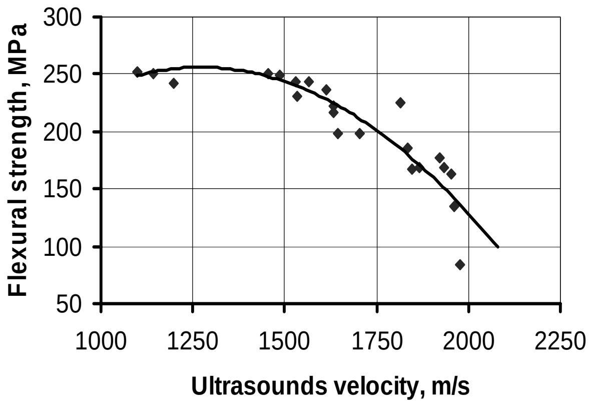

Fig. 15. Experimental relation between flexural strength and ultrasounds velocity

Elaborated method may be directly employed in the testing of degree of strength degradation of construction elements made of fibre reinforced polymeric composites. However, for quantitative interpretation of ultrasonic testing results, it is necessary to know diagnostic dependences individually identified for each tested material. To trace degradation processes ultrasonic tests have to be performed in defined time intervals from the beginning of working period of a given construction.

4. Conclusions

- Degrading processes taking place during thermal ageing of composites cause increase of its thermal resistance what influences the heat transfer process during thermographic tests. Properties of this heat transfer process may be correlated with changes of mechanical properties due to thermal degradation.
- The best correlation was achieved between time of temperature stabilization and the flexural strength. It proves that non-destructive thermographic method can be applied for diagnostic evaluation of the state of thermally degraded composite.
- The possibility of non-destructive evaluation of thermal properties of structural materials can be applied as a method of determination of strength and other properties deterioration.
- Ultrasonic tests results together with experimentally determined unequivocal relation between wave propagation velocity and flexural strength of the tested material form the basis of the non-destructive diagnostics method of plastic materials load capacity.
- The method may be directly employed in the testing of construction elements made of polymeric composites. However, for quantitative interpretation of results, it is necessary to know diagnostic dependences individually identified for each tested material.
- Comparing both methods applied in described laboratory experiment it is hardly to indicate which will be better one in real industrial working conditions. The choice depends on this working conditions, accessibility of object surfaces to thermovision or ultrasonic scanning and many others. Very important are also statistical parameters of diagnostic relations evaluated for given composites. These statistical parameters determine reliability of chosen method in working conditions. For example, the higher is correlation coefficient the more unambiguous is the relation.
- Results presented in the paper and presented scope of the research are of exemplary character. Real, individual problem defined for specific material characteristics needs basic research in order to determine diagnostic relation appropriate for searched material, searched material and for given working and experimental conditions.
- Results achieved in decribed research allow to expect that both non-destructive methods may be effective relative to wide class of polymeric performance materials.

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References

[1] P.V. Xavier Maldague, Theory and Practice of Infrared Technology for Nondestructive Testing, John Wiley, Interscience, New York, 2001.

[2] S. Poloszyk, Active thermovision in non-destructive testing, Proceedings of the Conference Manufacturing'01, Poznań, 2001, 221-228 (in Polish).

[3] D. Bates, G. Smith, D. Lu, J. Hewitt, Rapid thermal non destructive testing of aircraft components, Composites B31 (2001) 75-185.

[4] N. Rajic, Principal component thermography for flaw contrast enhancement and flaw depth characterization in composites structures, Composite Structures 58 (2002) 521-528.

[5] C. Meola, G. M. Carlomagno, A. Squillace, A. Vitiello, Non-destructive evaluation of aerospace materials with lock-in thermography, Engineering Failure Analysis 13 (2006) 380-388.

[6] E.G. Hanneke, K.L. Reifsnider, W.W. Stinchcomb, Thermography - an NDI method for damage detection, Journal of Metals 31 (1979) 11-15.

[7] G. Muzia, Z. Rdzawski, M. Rojek, J. Stabik, G. Wróbel, Diagnostics basis of thermographic investigation of epoxy-glass composites' degradation process", Proceedings of the International Conference "Machine Building and Technosphere of XXI Century", Donieck, 2007, 167-170.

[8] G. Muzia, Z. Rdzawski, M. Rojek, J. Stabik, G. Wróbel, Diagnostic basis of thermographic investigation of epoxy-glass composites' degradation process, Journal of Achievements in Materials and Manufacturing Engineering 24/2 (2007) 123-126.

[9] G. Wróbel, G. Muzia, Z. Rdzawski, M. Rojek, J. Stabik, Thermographic diagnosis of fatigue degradation of epoxy-glass composites, Journal of Achievements in Materials and Manufacturing Engineering 24/1 (2007) 131-136.

[10] I.M. Daniel, T. Liber, Non-destructive evaluation techniques for composite materials, Proceedings of the 12th Symposium on NDE, ASNT and NTIAC. San Antonio, 1979, 226-244.

[11] J. Deputat, Non destructive testing of materials properties, Gamma Publisher, Warsaw, 1997.

[12] P.K. Mallick, Nondestructive tests. Composites Engineering Handbook, New Jork, Basel, Hong Kong, 1997.

[13] A. Śliwiński, Ultrasounds and their application, WNT, Warsaw, 1993 (in Polish).

[14] R. Selzer, K. Friedrich, Mechanical properties and failure behaviour of carbon fibre-reinforced polymer composites under the influence of moisture, Composites 28A (1997) 595-604.

[15] A.P. Mouritz, C. Townsend, M.Z. Shah Khan, Non-destructive detection of fatigue damage in thick composites by pulse-echo ultrasonics, Composites Science and Technology 60 (2000) 23-32.

[16] A.S. Chen, D.P. Almond, B. Harris, Impact damage growth in composites under fatigue conditions monitored by acoustography, International Journal of Fatigue 24 (2002) 257-261.

[17] F. Kreith, Principles of heat transfer, IEP – A Dun-Donnelley Publisher, New York 1976.

[18] S. Ochęduszko, Applied thermodynamics, WNT, Warsaw 1970 (in Polish).

[19] E.H. Wichmann, Quantum physics, PWN, Warsaw 1973 (in Polish).

[20] N. Grassie, G. Scott, Polymer degradation and stabilisation, Cambridge University Press, 1985.