Publishing House
AKAPIT

COMPUTER METHODS IN MATERIALS SCIENCE

Informatyka w Technologii Materiałów

Vol. 13, 2013, No. 1

STATISTICAL AND PROBABILISTIC TECHNIQUES IN MODELING OF EPOXY CRACKING PHENOMENA

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Abstract

Epoxy resins filled with mineral particles, such as silica and alumina, are well known and extensively used as insulating materials in power product applications. They are relatively cheap and easy to process, and their dielectric, thermal and mechanical properties are appropriate to maintain their functionality in different electrical devices. However, the mechanical performance of particle filled epoxy systems can be often influenced by limited resistance to cracking.

It is frequently experienced during the lifetime of the epoxy cast products that some members of the production lot will crack during verification tests, while the others will remain healthy, even if the test loads are seriously increased. It is especially seen during low-temperature tests, when the thermal conditions are changed from +120°C to -60°C, when a few products can crack at +10°C, while others may easily survive till end of the tests. This phenomenon prohibits of using the classical material strength as a failure criterion. For this reason, some statistical and probabilistic approaches to describe the cracking resistance of the epoxy-based material have to be considered.

This work is focused on a study of process-induced residual stresses in epoxy components and their effect on material cracking likelihood. The proposed failure probability model incorporates Weibull distribution. The paper provides the experimental procedure for estimation of the Weibull model parameters (α, β), as well as implementation remarks. In addition, the Design of Experiment (DoE) method was used to support FEM calculations, and to find out the correlation between product design parameters and the failure likelihood. Finally, the article outlines some recommendations for applying statistical and probabilistic methods into numerical procedures of FEM analysis.

Key words: epoxy resin, failure criteria, failure probability, FEM analysis

1. INTRODUCTION

The mechanical properties of polymer materials, including epoxy based composites, are significantly time-dependent. In particular, an influence of loading history and/or temperature profile on failure stress has been reported in number of research works (Toeh et at., 1992, Gaudes, 2006). However, since the classical stress-strain analysis is based on continuum mechanics, it is a challenging task to predict a material failure in general, especially the failure of composites. This can be overcome by fracture mechanics, which allows considering the inclusion of defects into continuum models. Over the last decades many approaches for the prediction of the time dependent failures provided explicit equations to describe the lifetime. This paper incorporates probabilistic modeling, in order to cover large scattering of the material data, often experienced in real-world experiments of failure phenomena.

The classical theories, such as energy-based, or maximum strain failure criteria, are founded on the stored energy basis. These theories define the limit value for this energy, which is considered a material property. It is assumed, that when this threshold is exceeded, the failure takes place. Such methods

ISSN 1641-8581

provide direct relationships between mechanical modules and material strength, described by the Reiner-Weissenberg criterion as the first approach, (Guedes, 2004).

$$\frac{t_f}{\tau_0} = \left(\frac{1}{2-2^n}\frac{D_0}{D_1}\right)^{\frac{1}{n}}\left(\frac{1}{\gamma}-1\right)^{\frac{1}{n}}$$

$$\gamma = \sigma_0^2 / \sigma_R^2 \tag{1}$$

where: t_f is the lifetime under constant load σ_0, τ_0 is time unity (equal to 1 sec. or 1 hour, etc.), D_0, D_1, and n are material constants, while σ_R is material strength.

Similarly, the maximum strain criterion can be characterized as follows:

$$\frac{t_f}{\tau_0} = \left(\frac{D_0}{D_1}\right)^{\frac{1}{n}}\left(\frac{1}{\sqrt{\gamma}}-1\right)^{\frac{1}{n}} \tag{2}$$

Another theory of fracture, the kinetic rate theory, is based on a molecular approach, assuming kinetics of molecular flow and bond rapture of the polymer chains. Zhurkov (1984) first presented the model to predict materials lifetime, t_f in terms of constant stress, σ:

$$t_f = t_0 \exp\left[\left(U_0 - \psi\sigma\right)/kT\right] \tag{3}$$

where: k is the Boltzmann constant, T is the absolute temperature, while t_0, U_0, and ψ are material constants.

Many authors carried out the extensive work in the area of fracture mechanics. Schapery (1970) introduced a theory of crack growth, which was applied to predict a failure time for an elastomer under uniaxial and biaxial stress states. Based on that, Christensen (2002) proposed a kinetic crack formulation model to evaluate creep rupture lifetime for polymers:

$$\frac{t_f}{\tau_0} = \frac{\alpha}{\sqrt{\gamma}}\left(\frac{1}{\sqrt{\gamma}^{\,1/m}}-1\right) \tag{4}$$

where m is the exponent of the power law relaxation function, and α is a parameter governed by the geometry and viscoelastic properties.

There is a number of other theories, offering more complex models, however almost all criteria require model parameters to be curve-fitted to the experimental lifetime data. In most cases, the practical realization of the lifetime measurements involves the Time-Temperature-Superposition principle to accelerate the tests (Nowak et al., 2009). But, since the background theories lead to simple expressions recurring to empirical laws, in consequence it becomes impossible to determine these parameters based on more universal properties, like geometry, elastic and viscoelastic data, or instantaneous strength. Because the model parameters are not described universally, thus most of the failure criteria cannot be generalized, and their application may be limited. Moreover, some of the researchers (Witemberg-Perzyk, 2008) suggest, that for time-dependent loads, none of the main energy components is constant, thus energy-based criteria, or maximum strain criteria cannot be directly used as failure indicator.

This paper is organized in the following way. Chapter 2 provides a short description on an engineering approach for the estimation of the epoxy resin cracking phenomena, while next paragraph introduces probabilistic model and its application into FEM analysis. Finally, the concluding remarks are provided in last chapter.

2. DESCRIPTION OF EPOXY CRACKING PHENOMENON – AN ENGINEERING APPROACH

As it is shortly presented in the previous chapter, there is a substantial problem with modeling of the epoxy cracking phenomenon in practice, and developed models relay on empirical parameters, which must be fitted by experimental data. Producers of composites in general, and manufacturers of epoxy resins in particular, have realized that there is no single material property, which could, in clear and quantitative way, describe the behavior of the polymer exposed to time-dependent load. It was found, that crack resistance should be rather understood as a combination of different mechanical and thermal properties, like high tensile strength, high elongation at break, high intensity stress factor, but low Young modulus, low glass transition temperature, low thermal expansion coefficient, as well as high thermal conductivity. For the sake of simplicity, the resin producers developed the material property test, which became *de facto* an industrial standard. During this test the number of specially prepared samples (figure 1) is exposed to the low-temperature cycling.

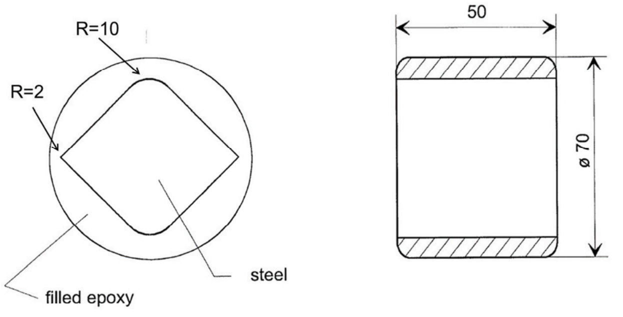

Fig. 1. A specimen for Crack Test [CIBA, 1997]. (Sharp edges of steel insert have R=2 radii, which serve as crack initiators).

The temperature profile used during the test was comprised of a series of increasing thermal loads, figure 2. Since the sub-ambient temperatures were progressing, the test escalated in severity (higher loading). The temperature level, at which the specimen fails, is recorded. A measure described as the Average Crack Temperature is calculated from the mean value achieved for 18 samples.

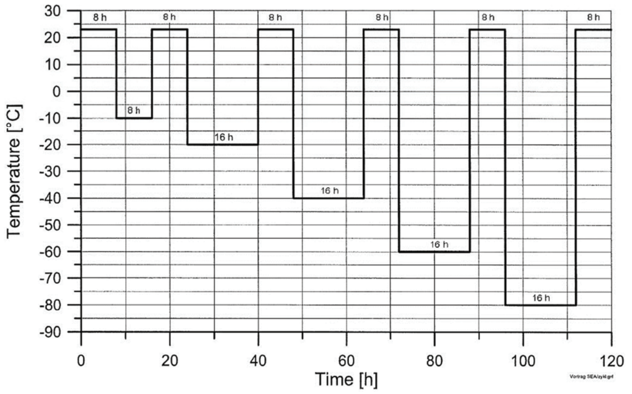

Fig. 2. Crack test temperature profile [CIBA, 1997].

Both, sample production process (material preparation, moulding, polymerization, de-moulding, post-curing) and testing procedure are well described and regulated, thus achieved results should be repeatable and stable. However, the measurements may demonstrate a very big scatter, as shown in figure 3, for example.

One can note that about 30% of samples cannot survive the temperature load of -10ºC, while 10% is able to withstand as much as -80ºC. With such a wide range in achieved results for theoretically identical samples, no-one is able to provide a reliable fracture criterion. Material suppliers use the mean value of Crack Resistance (as -45ºC, in this particular case), however also this measure is not specific enough. Therefore, there is a need for replacing the fracture criterion represented by a discrete number (used in energy-based criteria, maximum strain criteria, etc.) with a probabilistic approach. One the other hand, the failure likelihood (measured in “per cents”) is a very convenient indicator for practical applications, since it allows manufactures to estimate the level of potential production scrap.

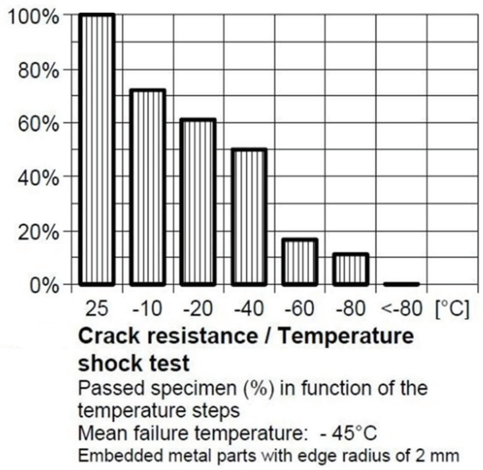

Fig. 3. Results of Crack Resistance test for CY225/HY225 epoxy system [Vantico, 2002].

The very interesting research problem aiming to explain such a big spread in the results of virtually indistinguishable test samples (but also real products) is not studied in this paper, and needs to be addressed elsewhere.

3. EPOXY CRACKING – PROBABILISTIC MODEL FOR FEM ANALYSIS

The overall idea of the probabilistic approach to the mechanical calculations relays on the concept that both, load-induced stress $f(S)$ and material strength $f(M)$ may be described by the normal distribution $N()$ characterized by its mean value, μ, and standard deviation, σ. Thus, probabilistic density functions: $N(\mu_S, \sigma_S)$ and $N(\mu_M, \sigma_M)$ are used to described the load-induced stress and the material strength, respectively. The probability of failure $\Phi(z)$, which is shown schematically in figure 4 as an overlap, can be defined by equation (5). Naturally, the reliability is expressed as $1-\Phi(z)$.

$$z = -\frac{\mu_M - \mu_S}{\sqrt{\sigma_M^2 + \sigma_S^2}} \tag{5}$$

where μ is a mean value, and σ is a standard deviation. Lower indexes M and S refer to material strength and mechanical stress, respectively.

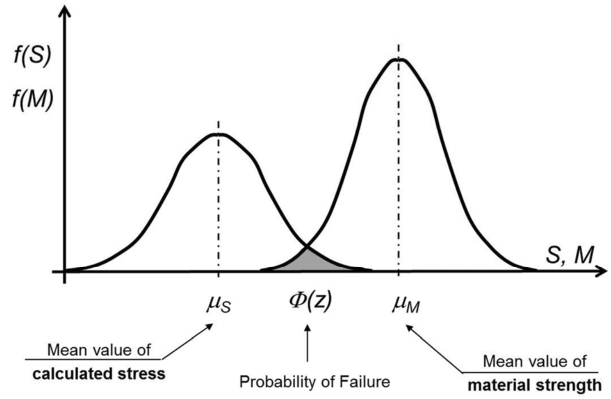

***Fig. 4.** Probabilistic model for material failure.*

In most engineering applications, the Weibull distribution model is used, especially for the lifetime estimations of the real objects. Thanks to its versatility, this model can take on the characteristics of other types of distributions, based on the value of the shape and scale parameters, α and β. The Weibull probabilistic density function, $f(x)$ is given by:

$$f(x)=\alpha\beta^{-\alpha}x^{\alpha-1}e^{-(x/\beta)^{\alpha}} \tag{6}$$

where x – the mean value (of calculated stresses, or material strength, for example)

The probability of failure as described by the Weibull distribution (the cumulative form of density function), $F(x)$ is given by:

$$F(x)=1-e^{-(x/\beta)^{\alpha}} \tag{7}$$

Since most of today's FEM software packages allow extending the standard, numerical code by user defined subroutines or additional libraries, it is also possible to implement the probabilistic failure models into mechanical calculations. In this case, FEM package calculates mechanical stresses (x) normally, but in addition, it can also use equation (7) to provide respective failure probabilities, $F(x)$. The shape and scale parameters (α, β), appearing in this equation, must be provided by the user. It is proposed in this paper, that model calibration procedure aiming to define the Weibull model parameters, utilizes low-temperature tests, as described in the previous chapter (figure 4). The graphical explanation of the recommended model calibration approach is presented in figure 5. Firstly, the real experiment of material's thermal shock must be performed and its results must be recorded (*step 1*). The probability of failure is plotted against the cooling temperature

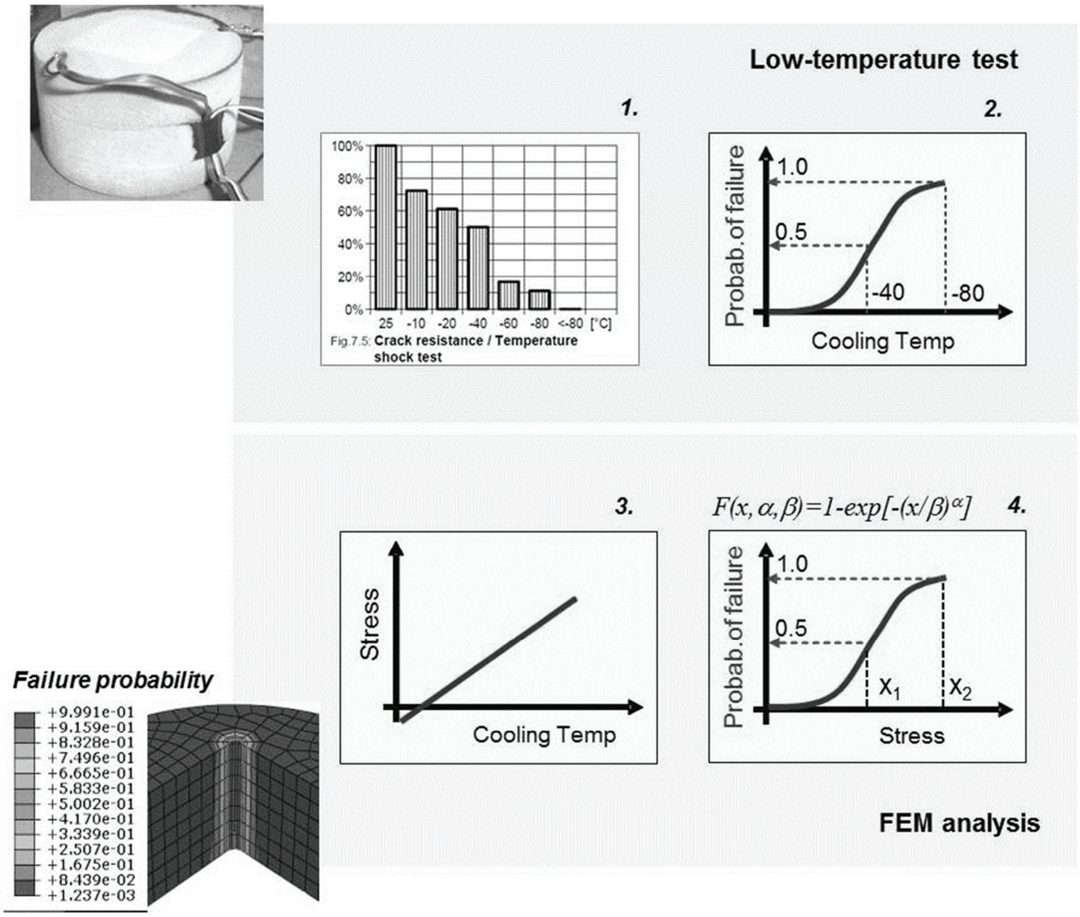

***Fig. 5.** Schematic representation of an approach for modeling of resin failure probability.*

(*step 2*). Next, the same experiment is virtually repeated by means of numerical simulations. The level of stresses, for energy-based criteria, is calculated for different cooling temperatures (*step 3*), as they were used during real thermal shock test. Since the results achieved in steps *2* and *3* have a common denominator – the cooling temperature, the relationship between the measured failure probability and the calculated stress level may be found. Finally, required parameters of Weibull model can be provided (step 4). As the failure probability model is introduced into FEM code, the mechanical response for various product designs (having different geometries) may be evaluated.

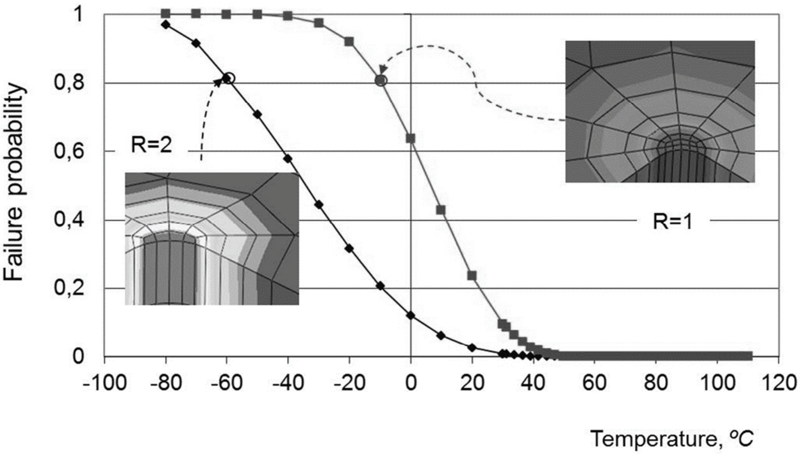

Fig. 6. *Failure probability during low-temperature test for two design variants: R = 1 and R = 2.*

The procedure shown schematically in figure 5 was managed in practice for the CY225/HY225 epoxy system by Vantico, what allowed estimating the parameters of failure probability model to: $\alpha = 3.25$, and $\beta = 81.77$. Next, the developed model was introduced into ABAQUS software package (2009), using UVARM subroutine. Before calculating the failure probability result, the full and properly defined analysis was performed. All the material properties were defined with respect to temperature dependency. Also contact behavior between resin and metal insert was established, with friction coefficient set to 0.6. The performed simulations were repeated for different mesh sizes in order to evaluate the influence of the model discretization on results quality, and to find out the distribution of calculated stresses.

As an example of the performed case studies, the modified design of specimen shown in figure 1 is evaluated. It was checked how the failure probability will be affected if the radii of sharp corner is changed from R = 2 to R = 1, respectively. The material properties were set according to the data provided by resin supplier (Vantico, 2002), while the temperature profile followed the curve in figure 2. The viscoelastic model was used in the temperature range between glass transition and ambient temperature. Below room temperature – the standard, elastic material behavior was assumed. The summary of calculated results is given in figure 6.

As calculated, the influence of the sharp corner on the cracking likelihood is significant. One can note, for example, that analyzed resin will crack with 80% of probability at: -10ºC in case of R = 1, and at -60ºC for R = 2. Also some cracks (about 20%) for the R = 1 radius are expected even at room-temperature level, which is not the case for R = 2.

In following study, it was checked if an application of stress-relief holes may reduce the expected failure probability of the specimens under study. The structured series of FEM calculations, driven by Design of Experiments methodology, was conducted. The small holes were virtually introduced near the sharp corner (crack initiator), with different design settings, figure 7a. The stress-relief hole was made at one side of the metal insert only, while the other side remained unaffected, serving for reference.

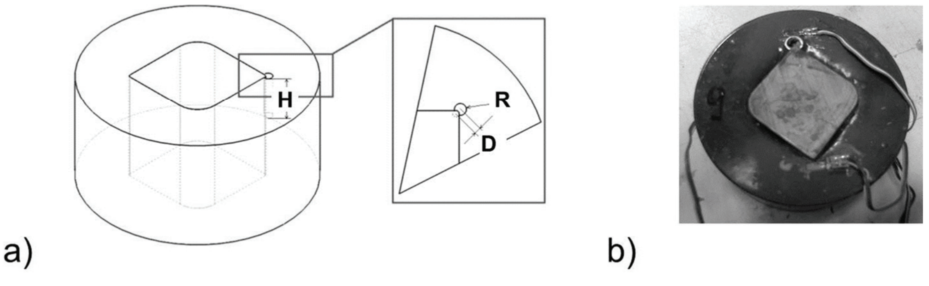

Fig. 7. *a) Design parameters (R, H, D) analyzed during DoE study on stress-relief holes b) Test sample used to validate FEM results by strain gauge measurements.*

The hole depth *H*, and its radius *R*, as well as distance to the corner *D*, were set on 3 testing levels each, what resulted in 27 (3^3) experiments. Based on achieved numerical results and performed Analysis of Variance (ANOVA), which are not shown in this paper, one could conclude, that reduction of the stress level $\Delta\sigma$ [%] may be described by very simple equation (with 90% of the confidence):

$$\Delta\sigma = -3.8 + 27.3H + 10.3R^2 + 22.1H^2 + 9.6D^2 - 8.4R^2H^2 - 8.4R^2D^2 + error \qquad (8)$$

where each independent variable can be set on 3 levels: {-1 / 0 / 1}. In case of hole depth, H - it corresponds to 10, 25 and 50 mm; for radius, R it is equal to 2, 3 and 4 mm; while for distance, D it matches to 0, 0.5 and 1 mm, respectively.

Using equation (8), one can show for example, that the sample design marked as (R_0, H_1, D_0), R = 3 mm, H = 50 mm, D = 0.5 mm, gives as much as 45% of stress reduction near the hole, however, the similar design (R_0, H_{-1}, D_0), with the hole depth set to 10mm, increases the stresses by 9%.

The optimal design, as provided by FEM study, was also tested in real experiment, figure 7b. It was observed, that introduction of the stress-relief hole generally reduces the measured stresses (in comparison to the reference strain gauge located near the corner, which was not protected by the hole). It translates into lower failure probability. It was noted that all cracks were initiated at the side without the hole. One should also mention an important manufacturing aspect – it was experienced, that the stress-relief holes should by cast during the production, not drilled after casting, since this second option introduces some residual stresses and facilitates cracking.

4. CONCLUDING REMARKS

The mathematical modeling of material failure is a complex task, since cracking phenomenon itself is very complex. It involves several variables related to the material structure, design and manufacturing aspects, which must be finally incorporated into few parameters. Consequently, the lifetime prediction is not trivial, especially for the brittle epoxy resin systems. In many cases, the classical failure theories, based on discrete criterion, do not provide the required level of confidence, thus probabilistic models may enter the research arena. The practical application of Weibull distribution into modeling of epoxy resin composite was provided in this paper. The structured methodology for calibration of the material model was given and validated. It was shown, that probabilistic models can be successfully applied into FEM analysis procedures, what enables producers of epoxy-based components for detailed estimation of potential production yield. In addition, the DoE methodology was involved to study the influence of stress-relief hole's design parameters on mechanical behavior of the product. It should be concluded, that probabilistic and statistical methodologies play an important role in both: engineering and research analysis, while allow to describe the real-world phenomena with better confidence.

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STATYSTYCZNE I PROBABILISTYCZNE TECHNIKI W MODELOWANIU ZJAWISK PĘKANIA ŻYWIC EPOKSYDOWYCH

Streszczenie

Żywice epoksydowe zawierające napełniacze mineralne, takie jak tlenek glinu czy krzemionka, są chętnie stosowane jako materiały izolacyjne w produktach przemysłu energetycznego. Materiały te oferują dobre właściwości dielektryczne, termiczne oraz mechaniczne, są stosunkowo łatwe w obróbce, a jednocześnie ekonomiczne. Jednak żywice epoksydowe z napełniaczami mineralnymi wykazują także pewne wady, wśród który najbardziej kłopotliwą jest niska odporność na pękanie. Cecha ta, jest tym bardziej uciążliwa, iż ma charakter silnie stochastyczny. Często zdarza się bowiem, że realizacja cyklicznych testów zmienno-temperaturowych w zakresie roboczym od -60°C do +120°C skutkuje zniszczeniem części próbek już przy pierwszym cyklu przy temperaturze +10°C, podczas gdy inne produkty zachowują swoją integralność przez cały zakres testu. Taki charakter materiału w znaczniej mierze utrudnia zastosowanie klasycznie rozumianej wytrzymałości materiału, jako kryterium zniszczenia. Zachęca natomiast do zastosowania metod statystycznych, dzięki którym zjawisko pękania żywicy epoksydowej można opisać w sposób probabilistyczny - bliższy rzeczywistości.

W artykule przedstawiono analizę naprężeń mechanicznych wywołanych procesem produkcyjnym elementu z żywicy epoksydowej z napełniaczem mineralnym, zawierającego metalowe elementy. Z uwagi na stochastyczny charakter zjawiska pękania takich wyrobów, zastosowano model zniszczenia w oparciu o rozkład Weibulla. Zaproponowano doświadczalną procedurę dla wyznaczenia parametrów modelu Weibulla (α, β), jak również wskazano algorytm uzupełnienia deterministycznych metod obliczeniowych MES o model prawdopodobieństwa. Wykorzystano statystyczną metodę projektowania eksperymentu (DoE) by przeprowadzić analizy numeryczne MES w taki sposób, by uzyskać liczbową korelacją pomiędzy parametrami geometrycznymi projektowanego wyrobu a jego prawdopodobieństwem zniszczenia. Artykuł przedstawia także kilka zaleceń praktycznych zarówno dla wykorzystania metod statystycznych i probabilistycznych w obliczeniach numerycznych, oraz sposobów obniżenia naprężeń rezydualnych w wyrobach z żywicy epoksydowej.

Received: September 27, 2012
Received in a revised form: November 5, 2012
Accepted: November 21, 2012