

DEVELOPMENT OF RESIDUAL STRESSES IN CAST PRODUCTS MADE OF EPOXY RESIN

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Abstract

Epoxy resins filled with dielectric mineral particles are frequently used as insulating materials in power industry applications. Due to their excellent dielectric properties and relatively good thermal performance (resistance, ageing and conductivity) their usability is common and extensive. However, processing of epoxy resin based products is quite difficult, and often results in quality problems or even cracks. Normally, for such as complex products, manufacturing-induced residual stresses cannot be totally avoided, but numerical simulations may provide good estimation of their level.

This paper deals with mechanical problems of power industry products and explains the methodology for numerical modeling of failure in silica filled epoxy systems subjected to severe temperature gradients. The exemplary product cast of Huntsman's CY228 epoxy system was simulated using commercial ABAQUS software package. Special subroutines describing progress of polymerization, exothermic effects of the reaction, as well as viscoelastic-driven stress relaxation process were implemented into general purpose FEM code. In addition, the model describing probability of epoxy cracking was proposed, calibrated and introduced into the software package. This approach allowed modeling the authentic phenomena taking place during real production processes, and studying the influence of design and manufacturing parameters on product quality. The outcome of numerical analysis was compared with the results of strain-gauge measurements, showing good agreement. As a result the optimal settings of curing and post-curing operations could be evaluated, driving to minimal cast product distortion and low probability of failure.

Key words: epoxy resin, viscoelastic material, residual stress, failure probability

1. INTRODUCTION

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. Generally, processing of epoxy resin based products is comprised of three main stages: mold filling, curing and post-curing. The sequential nature of the reactive molding process causes the products to be exposed to various environments and temperature changes during all manufacturing stages. These severe thermo-

mechanical conditions (thermal gradients in the material, chemical shrinkage of the resin, and variation of material properties driven by exothermal polymerization process) influence mechanical performance of particle filled epoxy systems, which finally may suffer due to limited resistance to material cracking (Weitsman, 1979; Ashcroft et al., 2006). Such a behavior can be observed, for example, in a product having metallic terminals (inserts) embedded in epoxy encapsulation. Described system is dimensionally defined at the early stage of manufacturing process when, at elevated temperature (usually near glass transition temperature, T_g), epoxy resin solidifies around the metallic inserts immersed within. At the certain point of resin's polymerization, its di-

mensions are fixed till the temperature starts to change. If temperature is lower than reference (solidification state), difference in coefficients of thermal expansion (CTE) of metal and resin generates thermal strains within the product body. Since free deformation is often not possible (metal and resin are joined together preventing natural, stress-free shrinkage) potential energy is accumulated within the product. There are only two ways of managing this energy: (1) either it must be released, what manifests in cracking (new surfaces are generated), or (2) it must be distributed within the body - assuming that generated stresses will not exceed the level of material strength.

In the structures containing small radii or sharp corners these thermally-driven residual stresses can reach really high values resulting in failure during the manufacturing process. Even if the components survive manufacturing process, the value of residual stresses appearing in the structure is significant and the failure can only be postponed. Subsequent contribution to the temperature gradient (e.g. winter conditions) may develop the damage and complete drop in functionality of the device.

The estimation of residual stresses caused by the thermal gradients and mismatch between two materials having significantly different CTEs is analytically possible only for simple geometries like disks or cylinders, where Lamé's Theory can be applied. This approach however works well only for elastic materials having the properties independent on the temperature and time. As the thermo-mechanical behavior of epoxy resins is distinctly time-dependent, it is necessary to characterize their properties with the use of viscoelastic material model (Ferry, 1980).

This paper describes the methodology of accurate evaluation of the residual stresses in epoxy-silica solid insulating composites. The methodology uses FEM analysis and viscoelastic modeling of epoxy materials for prediction of failure caused by mismatch of CTEs in the structure.

2. MATERIAL MODEL

2.1. Kinetics of curing

Polymerization kinetics of the material under study can be described by Kamal's model (Kamal & Sourour, 1973):

$$\frac{d\alpha}{dt} = \left(A_1 e^{-E_1/T} + A_2 e^{-E_2/T} \alpha^m \right) (1-\alpha)^n \quad (1)$$

where: α – degree of curing, t – time, T – temperature, A_1, E_1, A_2, E_2, m, n – material constants. The degree of curing, α , a common manufacturing parameter describing the progress of polymerization, is often defined by equation:

$$\alpha = \frac{H(t)}{H_U} \quad (2)$$

where: $H(t)$ – heat generated by the exothermal cross-linking reaction up to time t , H_U – ultimate heat of reaction.

2.2. Viscoelastic model of thermo-rheological simple material

The viscoelastic properties of the material can be measured in long-lasting creep or relaxation tests, but for practical reasons, it is more convenient to measure the modulus of thermosetting material as a function of temperature and frequency (instead of time). The storage $G'(\omega)$ and loss moduli $G''(\omega)$, as well as the stress relaxation modulus $G(t)$, can be approximated by a sum of Maxwell elements (ABAQUS, 2008):

$$G'(\omega) = G_0 \left[1 - \sum_{i=1}^N g_i \right] + G_0 \sum_{i=1}^N \frac{g_i \omega^2 \tau_i^2}{1 + \omega^2 \tau_i^2}$$

$$G''(\omega) = G_0 \sum_{i=1}^N \frac{g_i \omega \tau_i}{1 + \omega^2 \tau_i^2} \quad (3)$$

$$G(t) = G_0 \left[1 - \sum_{i=1}^N g_i (1 - e^{-t/\tau_i}) \right] \quad (4)$$

where G_0 is the value of the instantaneous modulus for the material (what corresponds to the glassy state for polymers), t is time, and ω is the radian frequency. Factors τ_i and g_i describe i^{th} relaxation time and relaxation factor for i^{th} time, respectively. Long-term modulus G_∞ as $t \rightarrow \infty$ (or rubbery state) can be expressed as follows:

$$G_\infty = G_0 \left[1 - \sum_{i=1}^N g_i \right] \quad (5)$$

The constitutive behavior of viscoelastic material can be illustrated by considering a relaxation test in which a strain γ is suddenly applied to a specimen and then held constant for a long time. The beginning of the experiment is taken as zero time, so that responding shear stress $\sigma(t)$ is:

$$\sigma(t) = \int_0^t G(t-s) \dot{\gamma}(s) ds = G(t) \gamma,$$



$$\text{since } \dot{\gamma} = 0 \text{ for } t > 0 \quad (6)$$

In case of materials which are assumed to be thermo-rheological simple (TRS) the technique of time-temperature superposition can be applied and the relaxation modulus at given temperature T , can be obtained if the relaxation modulus at reference temperature T_{ref} , and horizontal shift factor a_T , are known:

$$G(T) = a_T G(T_{ref}) \quad (7)$$

The temperature shift factor may be expressed in form of commonly used Williams-Landell-Ferry (WLF) equation (1955):

$$\log a_T(T) = \frac{C_1(T - T_{ref})}{C_2 + T - T_{ref}} \quad (8)$$

where C_1 and C_2 are material constants.

2.3. Failure probability

The probability of product failure (cracks appearing during production process) was evaluated using one of the most widely used lifetime distributions – the Weibull model.

$$D(x) = 1 - \exp\left[-(x/\beta)^\alpha\right] \quad (9)$$

where: $D(x)$ – failure probability (cumulative form); x – equivalent VonMises stress [MPa] developed during manufacturing process; α, β – parameters of Weibull model, calibrated for specific resin during initial experiments.

3. NUMERICAL ANALYSIS

3.1. Geometry

This work is focused on a study of residual stresses developing during manufacturing processes in epoxy product made of Huntsman’s CY228 epoxy system (resin filled with silica particles). The sample product, having steel insert (cubic form with 40 mm edge) embedded in the resin (cylinder with 100 mm diameter), figure 1a, was chosen to represent a typical design of Medium Voltage apparatus (magnetic core isolated by the resin). The specimens of this design are also used by commercial resin suppliers during measurements of crack resistance properties. The model of analyzed product is presented in figure 1b (due to symmetry only half of the component is shown).

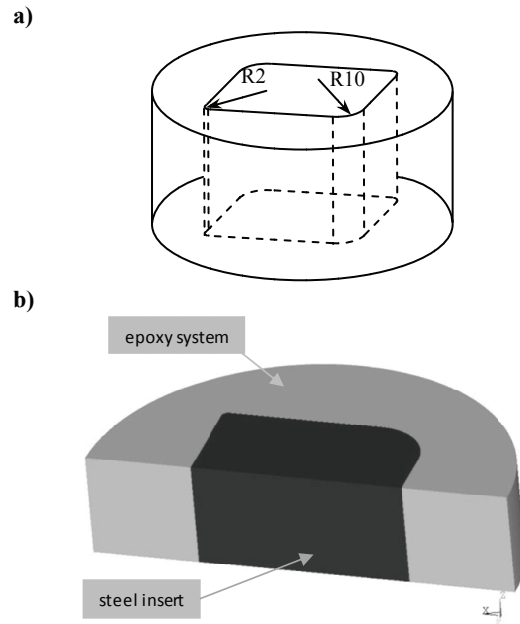


Fig. 1. a) Sample used for FEA simulation and verification tests, b) CAD model

3.2. Material properties

The general mechanical properties of the resin system are given in table 1.

Table 1. General properties of CY228 epoxy system

E [GPa]	T _g [°C]	CTE(<T _g) [ppm/K]	CTE(>T _g) [ppm/K]
12.9	75	41	186

The reduced curves for the storage and loss moduli were measured using DMA method (Nowak et al., 2006), and calibrated for 80°C as the reference temperature. The master curve, as described by equation 3, is given in figure 2. It was fit by weighting factors and relaxation times, as shown in table 2.

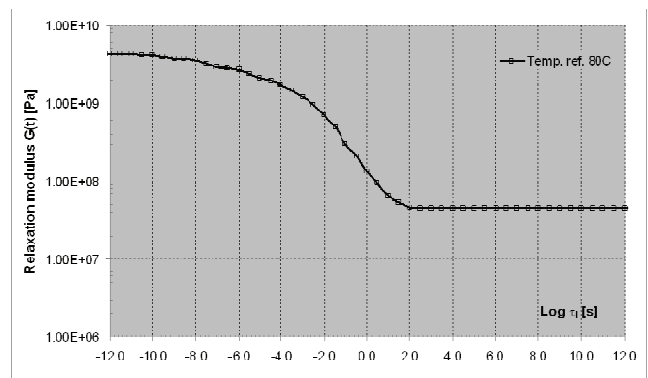


Fig. 2. The master relaxation curve for CY228 epoxy resin, $T_{ref} = 80^\circ\text{C}$.

Table 2. Prony series representation for CY-228 system.

τ_i	g_i
3.16E-14	0.07169
3.16E-13	0.04585
3.16E-11	0.00697
3.16E-10	0.08540
3.16E-08	0.19158
3.16E-06	0.14466
3.16E-05	0.06654
3.16E-04	0.11455
3.16E-03	0.09886
3.16E-02	0.10532
3.16E-01	0.03790
3.16E+00	0.01628

The other parameters describing polymerization kinetics (equations 1-2), time-temperature superposition (equation 8), as well as failure probability (equation 9) for CY-228 system are provided in table 3 (Nowak et al., 2009).

Table 3. Parameters of curing kinetics, WLF, and cracking models for CY-228 system.

A_1 [1/s]	2.293E+9
A_2 [1/s]	1.163E+5
E_1 [K]	11654
E_2 [K]	7182
m	0.438
n	1.118
H_U [J/kg]	103340
T_{ref} [°C]	80
C_1 [1/°C]	14.1
C_2 [°C]	69.6
α	3.4
β	82

3.3. Analysis procedure

The performed simulations reflected normal manufacturing process conditions during polymerization and post-curing process managed in the tunnel oven. The low-temperature cycling test was also simulated in the following step of analysis. The FEM results were expressed in the form of probability of failure distribution and compared to the strain gauge measurements. The commercial FEM code

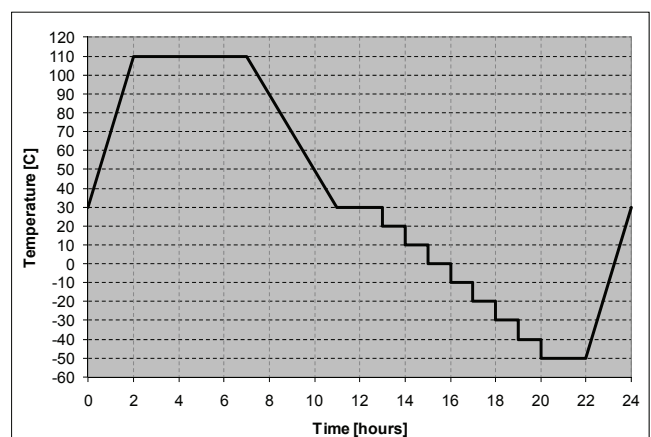
was extended about user-defined subroutines to cover all the phenomena described in Chapter 2.

3.3.1. Kinetics of curing

Simulation of curing process was performed to find out the solidification point of the resin where, at certain temperature, the mutual dimensions of both insert and resin are fixed and balanced. This analysis was carried out using CFD software package and temperature at the point when 65% of curing occurred was taken as a solidification point. The temperature distribution in the cured resin body was also taken as an initial conditions for further FEM analysis aiming the post-curing behavior.

3.3.2. Post-curing behavior and Temperature cycling test

Post-curing behavior was examined using transient analysis, and visco-elastic behavior of the resin system was studied to evaluate the level of residual stresses in the sample subjected to heating. During post-curing process the analyzed model was exposed to thermal load, according to recommendations provided by resin supplier. The product was kept in elevated temperature of 110°C for a few hours, allowing for stress relaxation process, and next was cooled to room conditions. Finally, the thermal testing in sub-ambient temperatures was modeled. The complete profile for combined post-curing and temperature cycling tests is shown in figure 3.


Fig. 3. Profile of thermal loading used in post-curing and temperature cycling tests

As the result of post-curing step analysis, the values of residual stresses developing during thermal loading were obtained.



3.3.3. Probability of failure

In this study, Weibull probability function (equation 9), was coded into the FEM system, allowing to visualize contours of product failure likelihood. This is the most effective and useful method to express the outcome of structural analysis, since the manufacturer can have the direct estimation of production yield and scrap ratio. Based on this information, further optimization of manufacturing process settings or design changes is easily possible.

5. RESULTS AND DISCUSSION

5.1. Curing phase

Based on performed calculations, which included heat delivered to the system as well as the exothermal nature of curing process itself, the temperature at the resin-metal interface for 65% degree of curing was noticed to be around 95°C, and stated as the reference value for further analysis, figure 4.

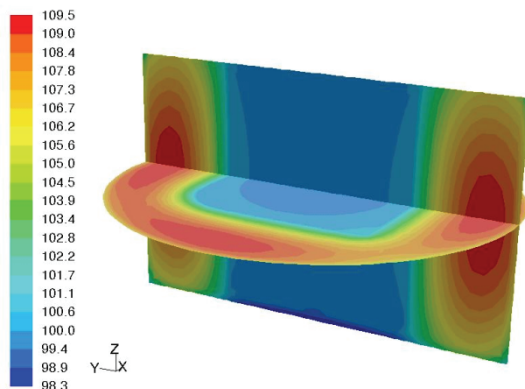


Fig. 4. Temperature distribution [°C] of the analyzed system for 65% degree of curing (two perpendicular cross-sections of the model are shown only)

5.2. Post-curing behavior

Since structural analysis of post-curing process assumed visco-elastic modeling of resin system, as described in chapters 2.2 and 3.2, the stress relaxation effects could be covered. The analysis of results indicates possibility of residual stress development in the pairs of resin-metal where both materials under thermal loadings significantly differ in values of thermal expansion coefficients. Thermally-induced stresses can reach significant values, and for the system with metallic insert having 1mm radius, they were reported to be as high 65 MPa, figure 5.

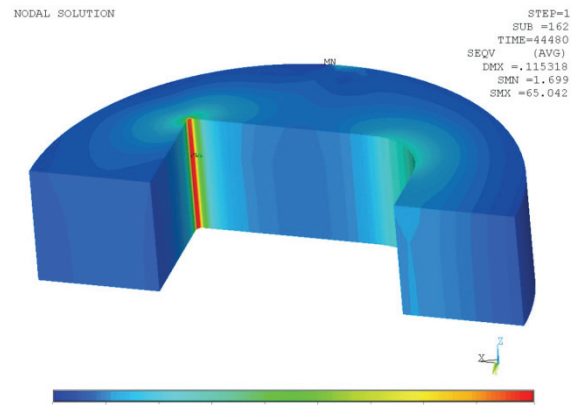


Fig. 5. Distribution of von Mises stress in epoxy resin based system at the end of post-curing process (temp. 20°C) (metal insert not shown for clarity)

Such accumulation of high residual stresses results in overdosed decrease in fatigue strength and even a single exposure to sub-ambient temperature leads to the unexpected failure. FEM analysis indicated that the residual stresses appear in the system slightly below glass transition temperature, T_g (around 50-70°C). They cannot be fully relaxed in limited time, since this process is effective only well above T_g . In real industrial application condition, where post-curing process is stopped at certain time, the stresses remain in the structure and are very dangerous for durability of poor-designed products.

5.3. Temperature cycling test and probability of failure

The results of temperature cycling test simulations confirm that below certain temperature the effectiveness of post-curing and relaxation is really limited and subsequent drop of temperature of the product below zero can result in catastrophic failure. These results were verified in the cold climatic chamber by strain gauges measurements, where local strains and deformation fields were monitored. Severe deformations at small radii R1 and R2 were observed during the experiment. The first fracture of samples during thermal loading was reported just shortly after passing through -20°C.

The measurement results were collected in the form of cumulative damage diagram, figure 6a.

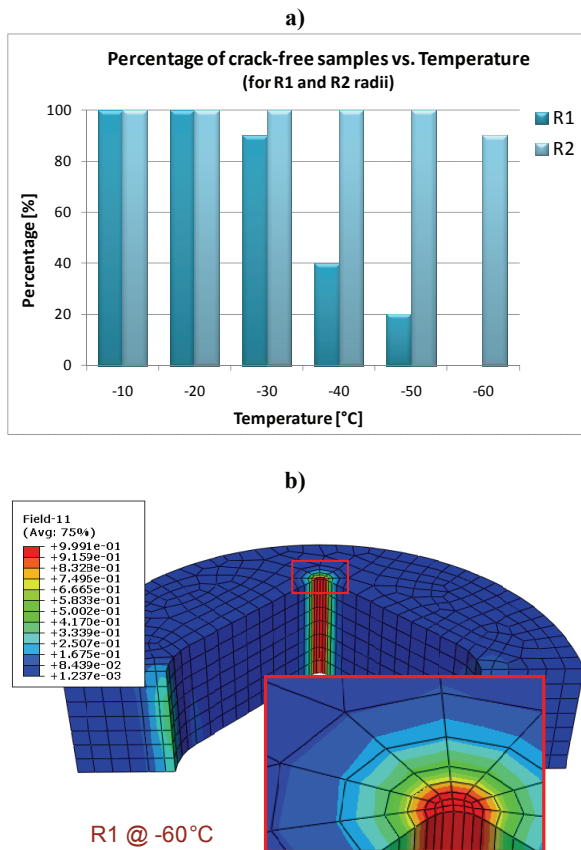


Fig. 6. a) Cumulative damage diagram for samples with R1 and R2 radii; b) Failure probability for R1 sample at -60°C thermal load – result of FEM analysis

Probability of failure was estimated based on the relationship between the level of residual stress and the number of samples which failed at certain temperature. The relationship was incorporated into the FEA package so that failure distribution could be plotted, figure 6b.

5.4. Strain gauges verification

Both: simulation and measurement results confirmed high likelihood of product to cracking. Eight samples out of ten having R1 radius broke during the temperature cycling tests. The typical strain readings are presented in figure 7a. Characteristic drop in the shape of curve is easy to notice and reflects the cracking of sample and following failure of strain sensor. Two curves in figure 7a represent two sensors located in hoop direction on opposite sides of the metal insert. The exemplary cracks are show in figure 7b.

6. CONCLUDING REMARKS

Various aspects of epoxy resin behavior modeling have been covered in this article, including progress of polymerization process, visco-elastic stress

relaxation as well as stochastic cracking. The relevant numerical models were proposed and introduced into FEM software package. The real manufacturing process of an exemplary epoxy-based product was simulated and results were compared with strain gauge measurements.

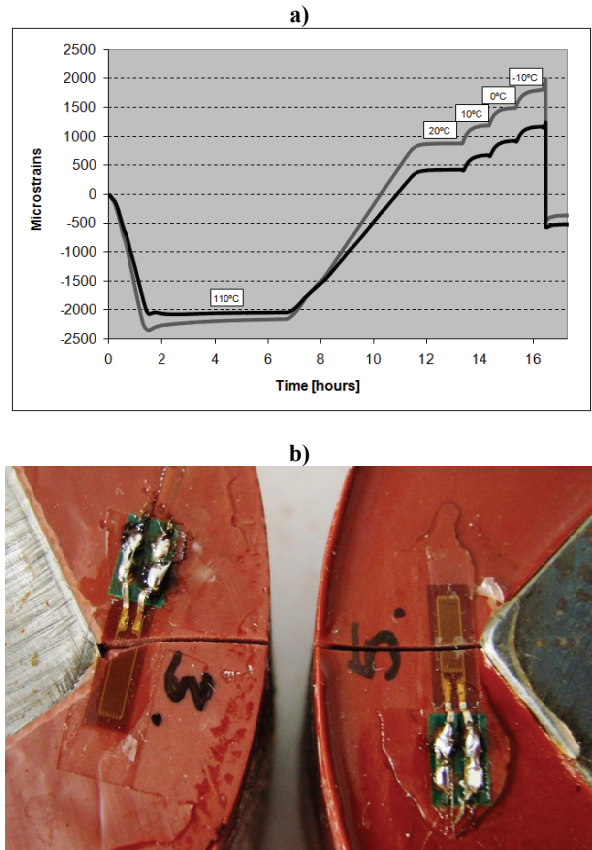


Fig. 7. a) Exemplary strain gauge readings during temperature cycling test - in hoop direction, close to R1 radius; b) Typical cracks appearing during the test.

Based on the work it can be concluded that failure of epoxy based products with metal inserts is caused mainly by tensile stresses in tangential direction. High differences in CTEs, as well as thermal gradients across the wall thickness generate significant stress level, up to about 50 MPa (at -50°C). If additionally, some stress concentrators (such as sharp corners) exist, the overloaded material may fracture. It was shown, that even small modification of insert's geometry (e.g. from R1 to R2) may drastically improve the percentage of crack-free products. Since the proposed methodology sufficiently reflects the behavior of resin system components during curing, post-curing processes and thermal cycling tests, author will work on analysis of further possible modifications of design and production process, allowing to reduce the failure rate. In this context, the application of stress relive holes is foreseen as a topic of a next study.



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MODELOWANIE NAPRĘŻEŃ RESZTKOWYCH W KOMPONENTACH ODLEWANYCH Z ŻYWICY EPOKSYDOWEJ

Streszczenie

Żywica wypełniona cząstkami krzemionki jest w urządzeniach elektrycznych powszechnie stosowanym materiałem izolacyjnym, spełniającym nierzadko również funkcje konstrukcyjne. Wynika to z bardzo dobrych właściwości dielektrycznych tego typu kompozytów, dobrych właściwości mechanicznych oraz ich powszechnej dostępności i stosunkowo łatwej technologii otrzymywania. Częstym problemem związanym z zastosowaniem tych materiałów są jednak przypadki zniszczenia aparatów zainicjowane na granicy faz metal-osnowa, a wywołane przez naprężenia resztkowe powstające już podczas schładzania po procesie odlewania żywicy. Prezentowana praca dotyczy oceny i modelowania naprężeń resztkowych przy użyciu metody elementów skończonych, z uwzględnieniem lepko-sprężystych właściwości materiału polimerowego. W artykule przedstawiono podstawowe modele zjawisk towarzyszących procesowi produkcyjnemu elementów z żywicy epoksydowej z napełniaczem mineralnym, a więc kinetykę sieciowania żywicy wraz z reakcją egzotermiczną, zjawisko relaksacji naprężeń w materiale lepko-sprężystym, a także opis prawdopodobieństwa rozwoju pęknięcia w wyrobie. Zaproponowane podejście pozwoliło wyjaśnić termo-mechaniczne zachowanie się wyrobu w procesie produkcyjnym i wskazać na naprężenia obwodowe występujące w pobliżu karbu jako główne źródło zniszczenia podczas testów niskotemperaturowych. Zaproponowano zmiany konstrukcyjne mające na celu ograniczenia prawdopodobieństwa pęknięcia wyrobów podczas testów.

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