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# Durability of Belzona 1111 and Belzona 1812 adhesive composites

**Summary** — The paper presents research of durability (static long-lasting life and fatigue life) of selected adhesive composites Belzona, which are epoxy adhesives physically modified with metallic and ceramic fillers. The tests were conducted by determining creep curves, short-term strength of lap joints, static long-lasting life of adhesive joints and adhesive composites. Numerical calculations, conducted with the Finite Element Method, were made in order to explain (on the basis of distribution of stress and strain) the phenomena occurring during a long-term load of an adhesive-bonded joint, which are unobservable during experimental research. With regard to comparative analysis' needs, the research results gained for Belzona composites were compared to the results gained for an adhesive based on physically unmodified Epidian 57 epoxy composition hardened with triethylenetetramine (Epidian 57/TETA).
**Keywords**: strength of materials, adhesive materials, mechanical properties of adhesive composites.

TRWAŁOŚĆ KOMPOZYTÓW ADHEZYJNYCH BELZONA 1111 I BELZONA 1812
**Streszczenie** — Określano trwałość: statyczną czasową oraz zmęczeniową tworzyw adhezyjnych firmy Belzona (1111, 1812), będących tworzywami epoksydowymi, modyfikowanymi fizycznie dodatkiem napełniaczy metalicznych lub ceramicznych. Ocenie poddano próbki tworzyw utwardzanych dwustopniowo, ponieważ jednostopniowe utwardzanie materiałów nie gwarantowało minimalnej (koniecznej do realizacji badań) statycznej trwałości czasowej (rys. 1, 2). Wyznaczano krzywe pełzania badanych kompozytów klejowych (rys. 3), określano wytrzymałość doraźną połączeń zakładkowych wykonanych przy ich użyciu (rys. 4) oraz wyznaczano statyczną trwałość czasową (rys. 5), a także trwałość zmęczeniową połączeń klejowych (rys. 6, 7) i trwałość zmęczeniową samych tworzyw (rys. 8). Badania numeryczne realizowano metodą elementów skończonych wykorzystując system Nastran for Windows. Obliczenia wykonano w celu wyjaśnienia, na podstawie analizy rozkładu odkształceń i naprężeń, zjawisk pojawiających się w trakcie długotrwałego obciążenia spoiny klejowej, niemożliwych do zaobserwowania podczas doświadczeń (rys. 11—13). Wyniki badań kompozytów Belzona porównano z wynikami badań kleju opartego na niemodyfikowanej fizycznie kompozycji epoksydowej Epidian 57 utwardzanej trietylenotetraaminą (Epidian 57/TETA). Stwierdzono, że połączenia, w których stosuje się kompozyty klejowe modyfikowane cząstkami cechuje większa statyczna trwałość czasowa i większa trwałość zmęczeniowa, a z charakteru krzywej pełzania można prognozować szacunkową zdolność spoin do długotrwałego przenoszenia obciążeń. Zjawisko pełzania kompozytów klejowych można również w istotnym stopniu ograniczyć dotwardzając je w temperaturze wyższej niż temperatura pokojowa, zwiększając jednocześnie trwałość tworzywa i połączeń wykonanych przy jego użyciu.
**Słowa kluczowe**: wytrzymałość materiałów, tworzywa adhezyjne, właściwości mechaniczne kompozytów klejowych.

The Belzona adhesive composites are materials applied in repairing damaged elements of machines, vehicles, and aircraft [1, 2]. Constructional application of this kind of adhesive materials requires, among others, determining their long-term static load transmission ability (static long-lasting life), as well as resistance to changing load ability (fatigue life) [3, 4].

The Belzona adhesive composites are materials based on an epoxy resin, and in connection with this, they reveal viscoelastic properties, which, among others, have influence on the durability of joints made with these composites [5, 6]. Viscoelastic properties of adhesive composites manifest themselves in various phenomena, among which the most significant in the engineering practice is the creep phenomenon [7, 8]. As opposed to mechanical joints, where the creep phenomenon occurs in the high

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temperature, a problem of adhesive materials' creep in joints is already visible in the temperature similar to the ambient temperature [2, 9, 10].

The aim of the paper was to determine the durability (both static long-lasting life and fatigue life) of the selected materials of Belzona company on the basis of experimental research, as well as numerical calculations carried out with the Finite Element Method. Numerical calculations were made in order to explain (on the basis of distribution of stress and strain) the phenomena occurring during a long-term load of an adhesive-bonded joint, which are unobservable during experimental research.

The Belzona adhesive composites are materials which are physically modified by adding metallic or ceramic fillers. Therefore, with regard to comparative analysis' needs, the research results gained for Belzona composites were compared to the results gained for an adhesive based on physically unmodified Epidian 57 epoxy composition hardened with triethylenetetramine (Epidian 57/TETA).

## EXPERIMENTAL

### Materials

The objects of research were adhesive composites Belzona 1111 Super Metal and Belzona 1812 Ceramic Carbide FP, products of Belzona Polymerics Limited Company, and the epoxy composition Epidian 57 hardened with triethylenetetramine (TETA), products of Zakłady Chemiczne "Organika-Sarzyna" S.A.

### Preparation of the samples

Hardening of the researched materials was performed with two following methods:

— Single-stage hardening in the ambient temperature (approx. 25 °C) for the time recommended by particular manufacturers of adhesive composites (Belzona 1111 — 20 h, Belzona 1812 — 3 days, Epidian 57 — 7 days);

— Two-stage hardening — the single-stage hardened specimens were subsequently re-hardened in the temperature of 80 °C for 6 h.

Two-stage hardening was applied because, on the basis of the earlier research [11], it seems that the ambient temperature is too low to cross-link materials based on epoxy resins. All the investigated materials are two-component materials, thus, while preparing compositions for further research, these were mixed in the weight proportions recommended by the manufacturers (Belzona 1111 — 5 mass parts of the basis to 1 mass part of the hardener, Belzona 1812 — 4.5 mass parts of the basis to 1 mass part of the hardener, Epidian 57 — 10.5 mass parts of hardener TETA to 100 mass parts of the adhesive). Specimens for materials' testing were cylinder-shaped, 12.5 mm in diameter and 25 mm in length.

What is more, the shear loaded single-lap joints 12.5 mm long, sized with the above-mentioned materials were researched [12]. Surfaces for gluing were prepared with the sand blasting method using aloxite of F46 grain granulation. Glued elements of the specimens were made of 2024 T4 aluminum alloy. They were approximately 2 mm thick and approximately 20 mm wide.

### Methods of testing

#### Creep curves

Creep curves [$\varepsilon = f(t)$] were gained by compression of cylindrical test specimens 12.5 mm in diameter and 25 mm long. The research was conducted with the usage of devices presented in paper [11], where the load element was a compressed spring. The measurement of strains' increase was conducted with a micrometric sensor. Creep tests were limited to 1000 h. The research was conducted in the temperature of 60 °C, using a lab drier with thermo-circulation. The specimens were put under load, causing a normal negative stress of 30 MPa.

#### Load capacity of lap joints

Load capacity of shear loaded lap joints was researched in order to select proper values of load in the durability test. Load capacity was determined with ZD-10 material testing machine in the ambient temperature, according to the methodology described in paper [11]. Determination was made using 6 specimens (the remaining specimens were used to investigate static long-lasting life). The results were verified by statistic methods at the confidence level $1 - \alpha = 0.95$.

#### Static long-lasting life of adhesive joints

The static long-lasting life was determined using devices presented in paper [13], able to maintain constant value of load. The testing of static long-lasting life of single-lap joints was carried out by loading them in the temperature of 60 °C with the constant force equal to 0.6 of their load capacity. In order to provide necessary temperature conditions, the devices with the investigated specimens were placed inside a lab drier with forced air circulation. The research was carried out according to the methodology described in paper [13]. The experiment was carried out with 5 specimens simultaneously. The scale of lap joints' durability was the time to the moment of the specimen's destruction. The assumed maximum time of test was 500 h.

#### Fatigue life of adhesive materials and joints

The research concerns comparison of fatigue life of joints made with Belzona 1111 and Epidian 57. Additionally, the durability research of materials was conducted, during which, after every 1000 cycles, the location of the moving traverse of the materials testing machine at the maximum and minimum load in the fatigue cycle was registered.

Testing of the fatigue life of joints was conducted using specimens identical to those prepared to determine the short-term strength. Load in the durability tests was of a from-zero pulsating cycle character in the range of 0.1—1.5 kN (the maximum load was 0.6 of the temporary durability of joints).

The specimens for testing were loaded with a from-zero pulsating cycle of the maximum load equal to 0.35 of the maximum load for Epidian 57/TETA and 0.5 for Belzona 1111, gained in the static compression test. In order to limit heating of the material the used load frequency was 20 Hz. The fatigue research was conducted with Instron testing machine.

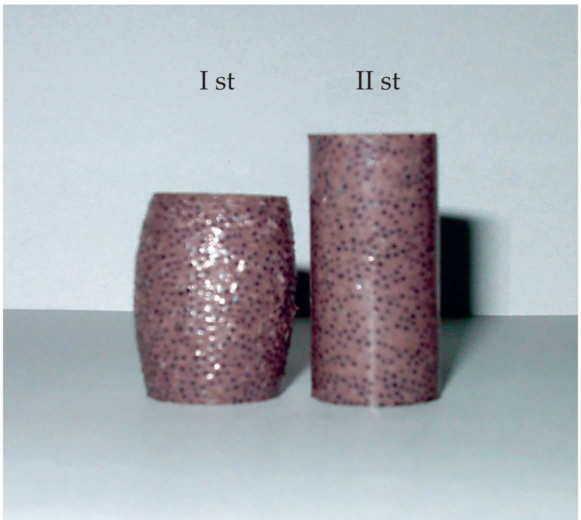


*Fig. 2. The comparison of stains of specimens cast from Belzona 1812 composite (single-stage hardening — I st and two-stage hardening — II st), investigated in the temperature of 60 °C under load, which caused normal negative stresses of 30 MPa*

## RESULTS AND DISCUSSION

### Creep curves

In the case of composites which specimens were hardened in the ambient temperature (a way recommended by the manufacturer) there were sudden increases of the strains registered in the research, which, in the case of Epidain 57/TETA, resulted in its destruction (Fig. 1), while in the case of Belzona 1812 Ceramic Carbide FP (a material with a ceramic filler) the specimen within 30 minutes from the beginning of the research became shorter by approximately 30 % (Fig. 2).

The creep curves of Belzona 1111 and Belzona 1812 adhesive composites, as well as Epidain 57/TETA material which were subjected to two-stage hardening are presented in Fig. 3.

As opposed to the typical course of creep of polymeric materials, the curves gained for the investigated materials did not have the third stage of creep, in which the velocity of creep increases rapidly causing material's destruction. The most considerable changes of strains in time were visible for Epidian 57/TETA. For the Belzona adhesive composites the changes of strains were less significant. The least increase of strains was visible in the case of Belzona 1812 material.

As a result of the conducted research, it was stated that the hardening temperature of the composite had a considerable influence on material's susceptibility to creep. All the adhesive composites hardened in the ambient temperature were characterized by a sudden increase of strains, which in a short time (the first minutes and hours of the experiment) caused destruction of the single-stage hardened specimens. The exception was

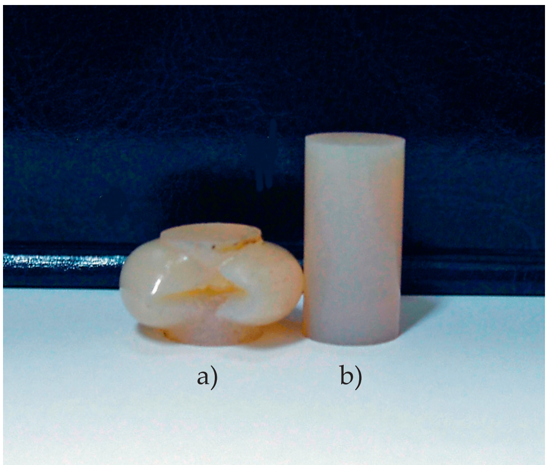


*Fig. 1. The strain of a specimen cast from Epidian 57/TETA hardened in the ambient temperature after 30 min of load (30 MPa) in the temp. of 60 °C: a) the specimen after investigation; b) the specimen before investigation*

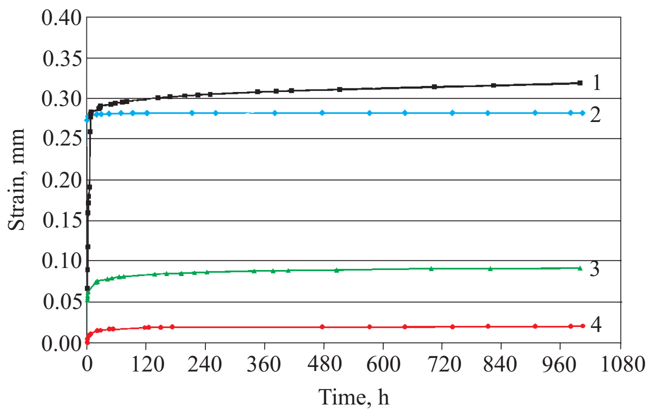


*Fig. 3. The creep curves of adhesive materials determined in the temperature of 60 °C for a load that caused normal negative stresses of 30 MPa in the investigated specimens. 1 — Epidian 57, two-stage hardening, 2 — Belzona 1812, single-stage hardening, 3 — Belzona 1111, two-stage hardening, 4 — Belzona 1812, two-stage hardening*

Belzona 1812 Ceramic Carbide FP adhesive composite, which is a material modified with a considerable amount of ceramic filler. The initial sudden strains of this material (single-stage hardened) connected to incomplete cross-linking of epoxy matrix of the composite, after a certain amount of time were stopped due to the adjoining particles of the ceramic filler (after the examination, the specimens had apparently rough back surfaces). Insufficiently cross-linked epoxy matrix of the composite underwent relatively fast cross-linking in the higher temperature of research (60 °C) limiting further displacement of the ceramic filler's particles in the epoxy saturant. This phenomenon appeared after several dozen hours from the beginning of the examination when the increase in the specimens' strain became significantly slower in the form of a considerable slowness of the increase of specimens' strains. In the case of this composite that was two-stage hardened, the initial strains were several times smaller than those in the single-stage hardened composite, what is more, the final strains were considerably smaller.

## Load capacity of adhesive joints

Load capacity of the investigated two-stage hardened adhesive joints was depicted in Fig. 4. The joints were prepared with the two-stage hardening method, since on the basis of the first stage of the experiment it was stated that the one-stage hardened material had low mechanical properties in higher temperatures. For the comparison purposes, there were also research conducted in which one-stage hardened Belzona 1812 was used.

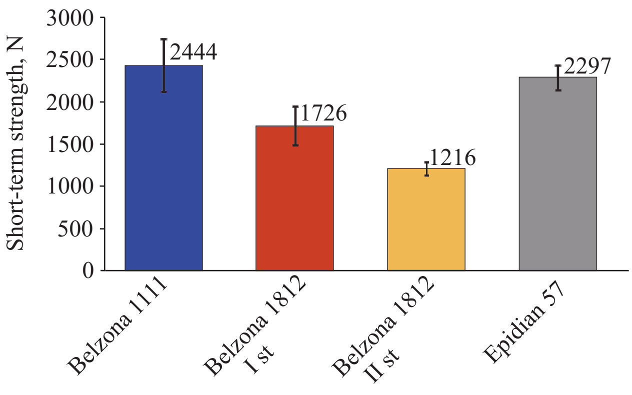


*Fig. 4. Short-term strength of lap joints made using investigated adhesive materials (single-stage hardening — I st and two-stage hardening — II st)*

The least considerable load capacity was gained for the joints prepared using Belzona 1812. The achieved results for these joints differed considerably depending on the method of material's hardening. The joints which were two-stage hardened were characterized by load capacity less by approximately 30 % than these joints in which Belzona 1812 hardened in the ambient temperature (according to the manufacturer's recommendation) was used. It seems to be connected with the level of the particular material's cross-linking. The material hardened in the higher temperature (precisely, its epoxy matrix) has a higher level of cross-linking and, in connection with that, it has different physical-mechanical properties. Probably, at it was shown by research results gained for Belzona 1111 composite [14], Belzona 1812 hardened in the conditions of ambient temperature shows lesser longitudinal elasticity modulus in comparison to the two-stage hardened composite (this conclusion could be drawn on the basis of the creep curves — different values of strain in the moment of load application, *cf.* Fig. 2). According to a relation concerning shear loaded adhesive joints, together with reduction of longitudinal elasticity modulus of an adhesive, its load capacity in the lap joints increases [15]:

$$P = A\frac{\sigma_n}{\sqrt{E_k}} \tag{1}$$

where: $P$ — *destructive force*, $\sigma_n$ — *destructive stress*, $E_k$ — *longitudinal elasticity modulus*, $A$ — *constant that takes into account, among others, dimensions of the adhesive joint, thickness of joined elements and their chemical properties*.

## Static long-lasting life of adhesive joints

The static long-lasting life of adhesive joints is depicted in Fig. 5 in a form of a bar chart, where x axis is the time till destruction of a particular specimens.

In the temperature of 60 °C the two-stage hardened Belzona 1111 and Belzona 1812 adhesive composites were characterized by joint's durability at the assumed level of 500 h. However, the joints made of Epidian 57 and single-stage hardened Belzona 1812 were characterized by low durability since all of the examined specimens were destroyed within the first hours of the experiment. What is more, the fact that values of load for durability tests were selected on the basis of load capability is also

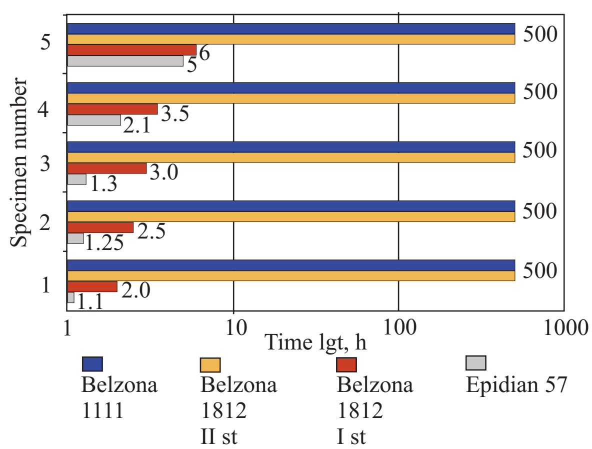


*Fig. 5. Static temporal durability of a lap joint made using the investigated adhesive materials (loaded with 60 % of destructive force in the temperature of 60 °C)*

essential, because this means that joints made using Belzona 1111 carried long-lasting loads at the level of 1.5 kN — $R_t$ = 6 MPa, for single-stage Belzona 1812 at the level of 1 kN – $R_t$ = 4 MPa, while for double-stage Belzona 1812 at the level of 0.7 kN – $R_t$ = 2.8 MPa (this value is only approximately 50 % of the load for Belzona 1111 composites).

Comparing the values of static long-lasting life of joints made using the investigated materials with their creep curves, it was stated that there was a clear relation between the character of creep curve of the composite and its ability to carry long-lasting shearing stresses in joints made of it.

**Fatigue life**

During determining the fatigue life of the specimens cast from Epidian 57 and Belzona 1111 materials, the measurements of changes in the specimens' height were conducted. The results of the conducted research are depicted in Fig. 6.

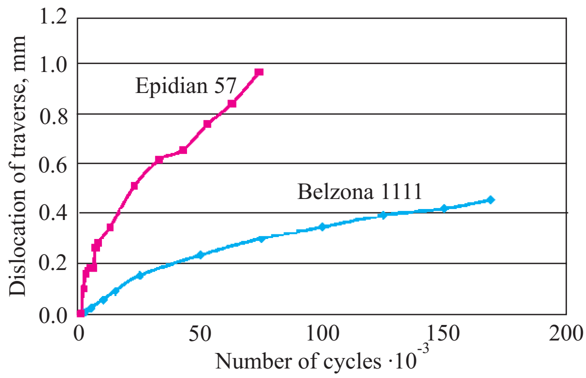


*Fig. 6. Dislocations of the traverse head (a change of the specimen's height) depending on the number of cycles for Epidain 57 and Belzona 1111 adhesive materials*

During the research, the constant increase of strains in the researched specimens under fatigue load was stated, that is specific for the scope of indefinite creep, and the range of height changes for Epidian 57/TETA was approximately threefold greater than for Belzona 1111. The fatigue life of single-lap joints in which the investigated materials were used is presented in Fig. 7.

On the basis of the results of adhesive joints' testing, it was stated that the fatigue life of joints made of Belzona 1111, loaded with a from-zero pulsating cycle is more than three-times longer than the fatigue life of joints made using Epidian 57/TETA.

The comparison of examinations of the fatigue life carried out for both the material itself, and lap joints makes it possible to suppose that the mechanism of fatigue destruction of the investigated materials, and thus, of adhesive joints made of them as well, is significantly

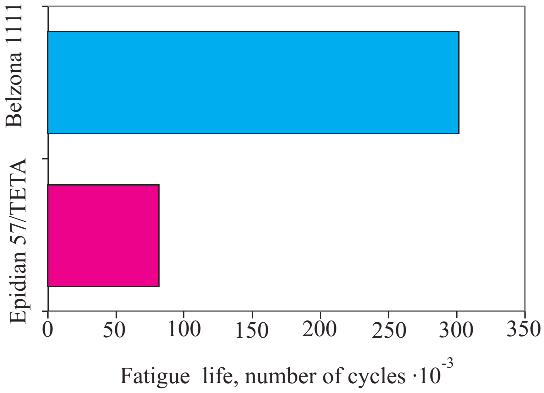


*Fig. 7. Comparison of fatigue life of single-lap joints, made with Epidian 57/TETA and Belzona 1111, with the maximum load of fatigue cycle equal to 0.6 of the destructive force*

connected with the material's creep under the load of the fatigue cycle.

A confirmation of this thesis could be the results gained during the research of fatigue load of the lap specimen (glued with adhesive Belzona 1111), where the location of the moving head of the materials testing machine was recorded (Fig. 8). In the first cycles of load, the change of strains was similar to the creep curve of the material (despite the fact that the curve presents summary strains of the joint and glued elements, it seems that the strains of the joint had a predominant importance).

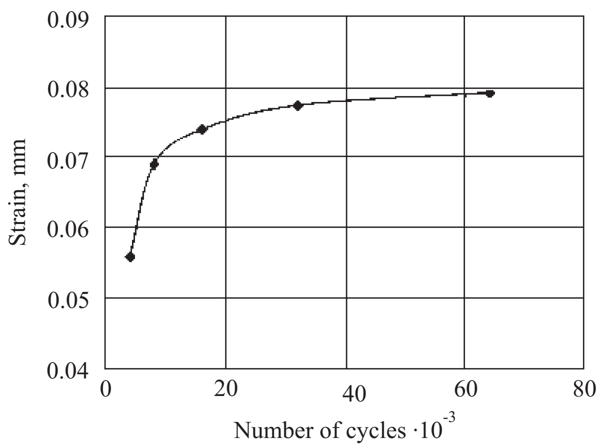


*Fig. 8. The change in the scope of strains in a fatigue loaded lap joint in the initial phase of the fatigue investigation [2]*

On the basis of the experimental research it was evaluated that both static long-lasting life and fatigue life of shear loaded lap joints are mainly connected to the phenomenon of creep of adhesive joint made with adhesive materials — increase of strains, and not to *e.g.* the phenomenon of adhesive material ageing. In order to verify this thesis a numerical model of a lap joint was built and analysis of changes in stresses and strains in the joint was conducted using the Finite Element Method.

### The analysis of stresses and strains in the joint

To conduct numerical calculation one should have assumed a proper model of the joint. Taking the discussed problems under consideration within the area of durability one should have taken into account the viscoelastic properties of adhesive materials.

To determine viscoelastic properties of an adhesive joint there was used the four-parameters model of Burgers body, which represents, in a qualitative way, all the properties of viscoelastic bodies (Fig. 9).

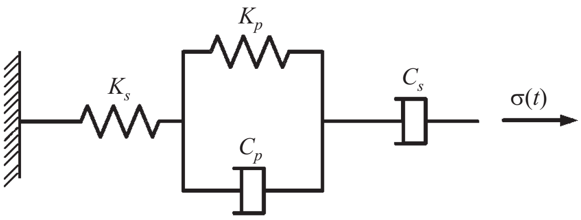


*Fig. 9. Model of physical properties of adhesive joint's material used in NASTRAN program to calculate the creep phenomenon*

For this model the following coefficients' values were declared: $K_s$, $K_p$, $C_s$ i $C_p$, which were estimated according to the methodology presented in paper [16] on the basis of creep curves determined experimentally in the temperature of 60 °C [17]. For Belzona 1111 in the temperature of 60 °C these had the following values: $K_s$ = 4545 MPa· s, $C_s = 21.65 \cdot 10^9$ MPa· s, $K_p$ = 2368 MPa, $C_p = 50.43 \cdot 10^9$. The model of the joint in which for discretization of the glued metal parts there were used 1300 elements, while for the adhesive joint there were used 500 elements, was constructed on the basis of elements of Hex type. The model was elaborated according to the directives included in paper [16] taking the marginal conditions occurring in the experimental research under consideration. The discrete model of the adhesive joint is presented in Fig. 10.

Taking the clear relation between stability of the gained solutions and amount of the selected time frame under consideration, the stresses and strains for a time that guaranteed stable numerical solutions were determined (up to 120 h).

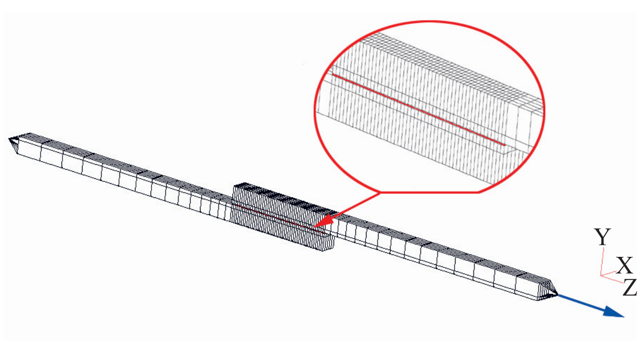


*Fig. 10. The discrete model of the adhesive shear loaded lap joint*

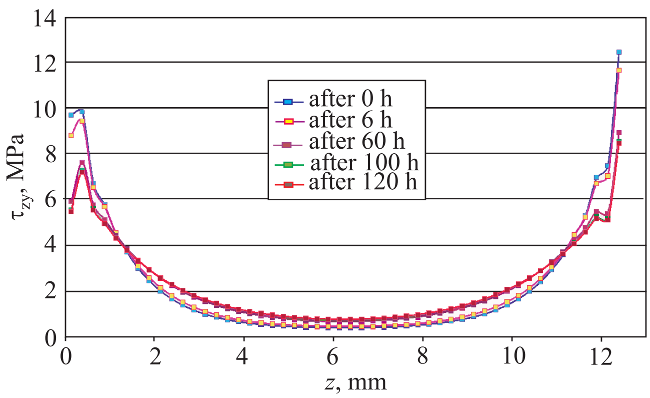


*Fig. 11. A change in time of tangential stresses ($\tau_{zy}$) along (z) the adhesive joint for Belzona 1111*

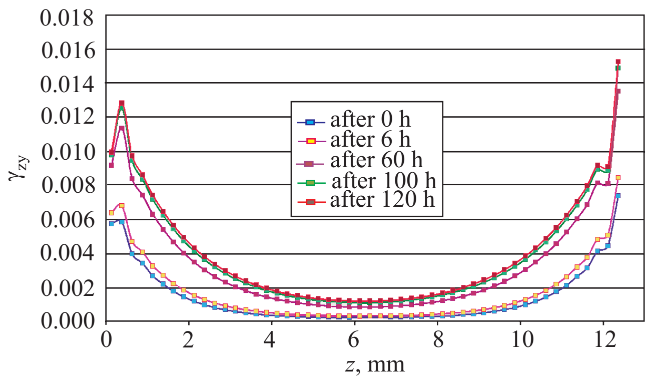


*Fig. 12. A change in time of non-dilatational strain in ($\gamma_{zy}$) along (z) the adhesive joint for Belzona 1111*

In order to observe changes that occur in the assumed time frame, determining stresses and strains for intermediate times became necessary. It was assumed that the numerical analysis would be conducted for load application

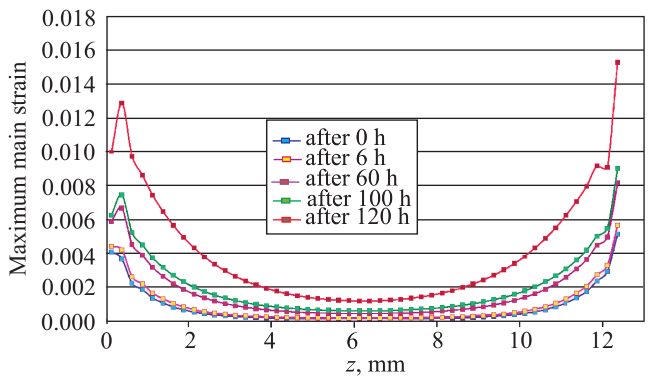


*Fig. 13. A change in time of the maximum main strain along (z) the adhesive joint for Belzona 1111*

(time $t = 0$), as well as, for the time of 6, 60, 100 and 120 h. The distribution of tangential stresses ($\tau_{zy}$) along the adhesive joint is depicted in Fig. 11, while the distribution of non-dilatational strains ($\gamma_{zy}$) and maximum main strains is presented in Fig. 12 and 13.

The changes of tangential stresses (decreasing the coefficient of stresses' concentration) as a function of time were observed at the ends of the lap — in the most strenuous area of the adhesive joint. Comparing the area of stresses' changes with the whole length of a joint lap one could state that the area of changes is still significantly limited.

The more significant are changes connected with strains. The value of strains increases along the time of load application in the whole volume of the joint — especially intensively at the ends of the lap (where the level of stresses is also significantly greater).

From the conducted calculation it follows that the growing strains in the joint (with slightly changing stresses) could significantly limit the durability of joints and it concerns both static long-lasting life and fatigue life.

## CONCLUSIONS

The conducted experimental research and numerical analysis enabled to draw the following conclusions:

1. Adhesive materials — existing both in the form of cast elements, as well as, in the form of an adhesive joint — when subjected to long-lasting static load or changing load undergo the phenomenon of creep. In connection to the above-mentioned it seems that an important element of destruction mechanism of the long-lasting loaded (statically and variably) adhesive materials (including adhesive joints) is connected to this phenomenon.

2. The research shows that the joints, in which adhesive materials resistant to creep are applied, are characterized by longer lasting static life and longer fatique life. And thanks to the nature of the creep curve of the adhesive composite one could predict joints' ability to long-lasting carrying of the load.

3. The phenomenon of adhesive materials' creep could be considerably limited by adding fillers (metallic or ceramic) to the materials. The example of this kind of operation are materials of Belzona Company. Depending on the type of filler, this kind of physical modification could cause changes in mechanical properties of the material — Young model of the material itself is changed, as well as, load capacity of adhesive joints.

4. The phenomenon of adhesive composites' creep could be considerably limited also by hardening them in the temperature higher than the ambient temperature, which would increase durability of the material itself and joints made of this material. It seems that this phenomenon is connected with the level of cross-linking of the epoxy matrix.

5. Taking the results of experimental research under consideration one must state that the static long-lasting life of adhesive joints, in which Belzona 1812 and Belzona 1111 are applied, could be at least 500 h in the temperature of 60 °C, with the load not greater than 0.5 of their load capacity. Taking into consideration the results, it seems that the researched composites can be successfully used in expedient repairs of military equipment conducted in field conditions. For instance, required working life for military planes, which are repaired is 100 h.

6. Fatigue life of the joints in which Belzona 1111 composite is applied should exceed 300 thousand cycles if the maximum load of the fatigue cycle does not exceed 0.5 of its temporary durability.

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