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Research on the Durability of Adhesive Composites

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The paper presents research on the durability of adhesive composites, which may be used for the repair of technical objects. Durability of adhesive composites is understood in this paper as a material ability to transfer long lasting loads. Since that kind of information is not provided by manufacturers, the long-term strength and fatigue-life of selected adhesive composites are determined; especially with regard to their use in expedient (temporary) repairs of weapon systems. The results obtained show that safe values of the maximum long-term and fatigue loads of the adhesive composites investigated should be a half of the short-term strength obtained in a static tests.

Key words: durability of materials, adhesive composites features, long-term strength, fatigue life.

1. Introduction

A large number of failures in military equipment are especially visible during different kinds of combat operations. The most common battle failures of weapon systems are punctures, cracks, breaks, and lack of tightness. Any field repair actions of damaged weapon systems can be divided into two basic groups: standard (regular) repairs or expedient (temporary) repairs. Standard repairs are conducted by exchange of whole broken units or single spare parts, which are delivered by supply chains of logistic units or obtained from totally destroyed weapon systems (cannibalization). If possible, a standard repair is preferred, but it is very difficult to perform in the conditions of combat operations. An expedient (temporary) repair can be an alternative solution in many cases. An expedient repair means any improvised action which may lead to broken system being temporarily available in order to execute a task [12, 21–23]. There are many methods that can be used to execute expedient (temporary) repairs in field conditions [11, 25], and it seems to be that adhesive materials have a great potential of providing that actions. This is caused not only by the numerous advantages of adhesive joints, but also by the increasing capacities of the particular links of the repair system, such as, reducing work consumption, enabling to perform some repairs on a lower level, no necessity to use expensive and heavy equipment, creating possibility of an available soldier and becoming independent of spare parts [23, 26]. As a result, technical objects' repairs are more and more often taking advantage of structural bonding [1, 17, 19], as well as, quick chemically setting materials; so-called adhesive composites, which create new possibilities in the scope of temporary or permanent removal of different types of damages [2, 5]. Modern adhesive composites may provide solutions to many problems, which can occur as the result of intensive exploitation of military equipment, as well as, from the influence of the enemy's munitions.

Adhesive composites applied to field repairs of weapon systems must be able to transfer great loads and create solid construction joints; therefore, it is required of them to show high cohesion and adhesion. However, the adhesive materials are characterised with relatively low durability. These materials are very sensitive to repair conditions, as well as, that they have limited shear and long-term strength – the ability to transfer constant loads in time – and limited fatigue life – the ability to transfer changing loads [5]. Without familiarity with these properties, the application of adhesive materials is burdened with the risk of re-damage in a relatively short time after the performed repair.

2. Methodology of research

Currently, a wide range of specialist adhesives for different purposes is produced. There are many products of numerous companies, but taking the specificity of field repairs into account, it seems that the most useful are composites fulfilling the requirements of multi-purpose materials as "super metals" and those of the quick-setting "rapid" type [2, 24]. Therefore, the main objects of research are well-known and available in the Polish adhesive composites market of Belzona, Unirep and Chester Metal.

Application of adhesive composites to repairs requires, among others, determining their durability, which is broadly understood as a material ability to transfer long-lasting static load (static long-term strength) and their durability to changing load (fatigue life). Since that information is not provided by the manufacturers, the main aim of the work is to determine the aforementioned features of selected adhesive composites with regard to their use in expedient (temporary) repairs of technical objects.

Numerous experimental investigations were conducted in order to execute the planned research, in which specimens of the examined adhesive composites were used to determine their mechanical properties. Bonded specimens were used in order to determine the strength of joints made with the examined materials. It was decided to elaborate the results of experimental investigations with statistical methods. The arithmetic mean and the standard error of the mean value were calculated according to the following formulas [8]:

$$\overline{x} = \frac{\sum_{i=1}^{n} x_i}{n}, \tag{2.1}$$

$$\overline{S} = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}}, \tag{2.2}$$

where n is the number of specimens in a lot; x_i is the measured result for i of that specimen; and $\overline{x}$ is the arithmetic mean of n results.

Since $n < 30$, the confidence interval was defined by means of the Student-Fischer method for a confidence level of $1-\alpha = 0.95$:

$$\left(\overline{x} - \frac{t_\alpha \overline{S}}{\sqrt{n(n-1)}}; \quad \overline{x} + \frac{t_\alpha \overline{S}}{\sqrt{n(n-1)}} \right), \tag{2.3}$$

where t_α is a number satisfying the relation

$$P\left(|T_{n-1}| \geq t_\alpha\right) = \alpha \tag{2.4}$$

in which T_{n-1} means a random variable of a T-Student distribution of $n-1$ degrees of freedom.

2.1. Methodology of research on long-term strength

The research on static long-term strength is aimed at an estimation of the permissible value of loads, for which constant influence does not cause damage to adhesive joints for at least 500 hours at a temperature of 60°C. The operation time of a joint was assumed by considering the temporary nature of expedient repairs. The temperature was assumed by taking into consideration the possibility of the heating up of military equipment exposed to sunlight, as well as the possibility of repairing elements heating up during operation; i.e. engine blocks, with adhesive materials [16].

The research on long-term strength was executed with three simultaneous stages. The first stage consisted in determining the specimens strains loaded at increasing temperature (creep curves). In order to determine creep curves, specially designed and executed devices were used in which the specimens were loaded with compression coil springs. The characteristics of the springs used were linear; therefore, the load on the specimen could be selected (with the sufficient accuracy) on the basis of measurement of spring deflection [13]. The measurement of strain values of the examined specimens was conducted by means of a mechanical micrometric sensor mounted to the moving component of the device. The device together with the specimen was placed into a temperature chamber (Fig. 1). Subsequently, the strain value of the examined adhesives was noted at the specific time intervals. The research was continued until the increase of strain stopped, or until the strain increase became negligible. The specimens used to determine creep curves were made in the form of a cylinder of 12.5 mm diameter and 25 mm length (Fig. 2a).

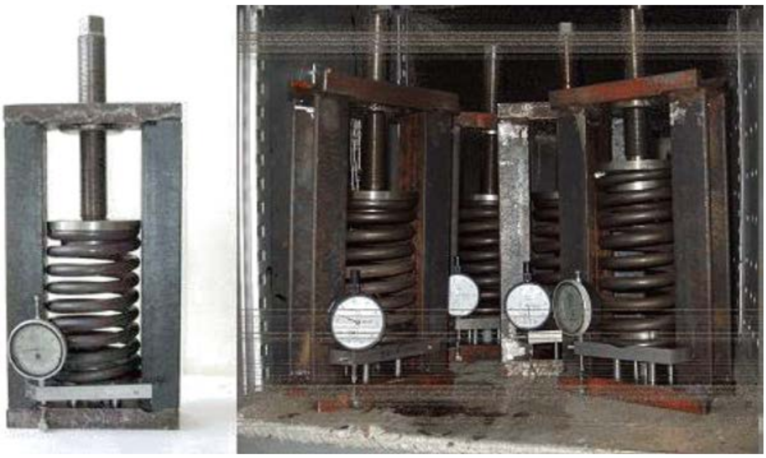

FIG. 1. View of the device used to determine creep curves with mounted and loaded specimens.

a) b) c)

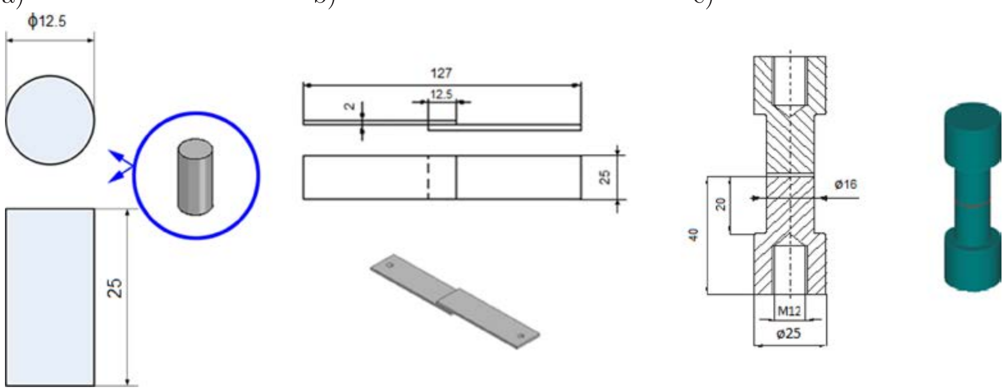

FIG. 2. Shape and dimensions of specimens used to determine properties of adhesive composites.

The subsequent stage of research allowed determination of the shear strength of adhesive composites joints. The determined strength was the basis for load selection to long-term tests. In order to determine shear strength, single-lap joints were used (Fig. 2b), which were made of S235Jr steel. The tests were conducted according to PN-EN 2243-1:1999, with use of a ZD-10 testing machine [14].

The last stage of research defined static long-term strength of the examined adhesive composites. The measured value was the time needed to destroy the joint subjected to certain load with increasing temperature. These tests were conducted by means of devices designed by Rośkowicz [13] (Fig. 3).

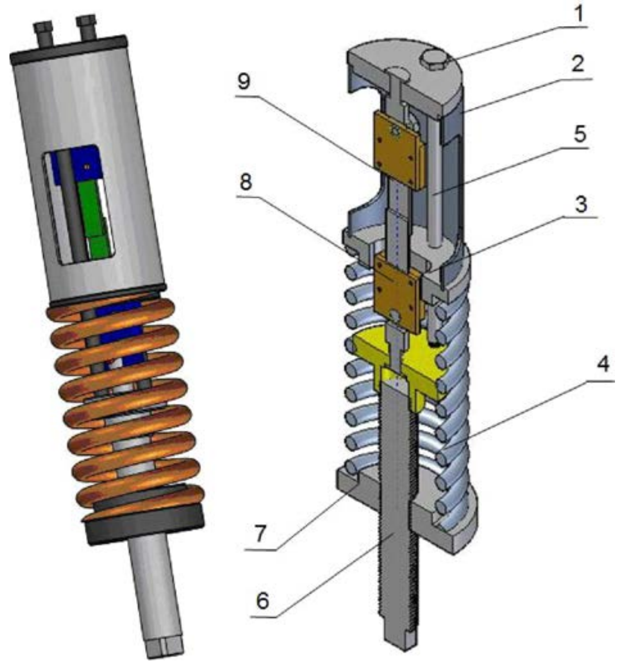

Fig. 3. The device used to determine static long-term strength of adhesive joints: 1, 3 – covers; 2 – spacing sleeve; 4 – spring; 5 – lock screws; 6 – screw tightening the spring; 7 – resistance sleeve; 8, 10 – grips for specimen mounting; 9 – tested specimen [13].

The function of these devices was to apply the required constant load to the lap specimen bonded with the adhesive composite. The research involved simultaneously five devices, which were placed in a KC 100/200 thermal chamber of the EL-CON company (Fig. 4).

The load was imposed – similarly to determining of creep curves – by the spring compression of a value defined with a characteristic of each spring used (Fig. 5). Specimens were kept in the assumed temperature under constant load until their destruction. The particular time was measured to the specimen's destruction. The applied load caused normal negative stress in the specimen of

Fig. 4. The view of devices together with the mounted and loaded specimens used to determine long-term strength of adhesive joints.

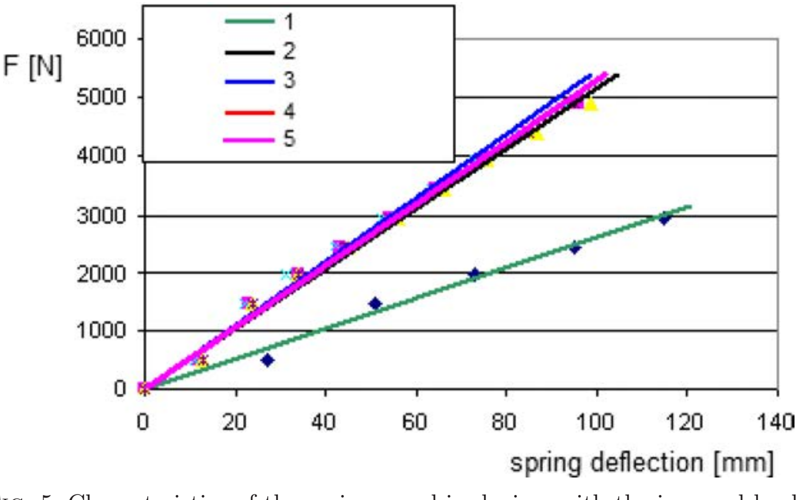

Fig. 5. Characteristics of the springs used in devices with the imposed loads.

30 MPa. The value of stress was selected on the basis of a compromise between the dimensions of the measuring devices (these were designed in such a way that it was possible to place them all in a thermal chamber at the same time) and the value of the load, with which the phenomenon of creep shows itself. The value of the load was in the range of 30–50% of the shear strength of the adhesive composites.

2.2. Methodology of research on fatigue life

The research on fatigue life of adhesive composites made it possible to estimate the permissible loads of joints loaded with changing forces. Thanks to the repeated experimental investigations, the conditions of the experiment were selected: frequency of loads, type of load cycle, value of the maximum load of cycle, and the types of specimen used in the research. With regard to the temporarily nature of field repairs of weapon systems, the fatigue life tests were terminated after 250,000 load cycles. In the first step, specimens used were those presented in Fig. 2a. In the research, adhesives composites of the "super metal" group were loaded with zero-start pulsating compression stresses of 55 MPa, that is, equal to a half of their compression strength. The adhesive composites of the "rapid" group were loaded in fatigue tests with zero-start pulsating compression stresses of 28.5 MPa, and that is a half of compression strength of the material of the lowest strength [5]. The tests were performed on an Instron 8501 testing machine with a frequency of 20 Hz.

Further research on fatigue life was carried out for adhesive joints with use of tensile loaded cylindrical specimens (Fig. 2c). The elements of these specimens were made with a 2024TR aluminium alloy. The method of preparing the specimens' surface for bonding was by sand blasting using aloxite, followed by cleaning with extraction petrol. The specimens were subsequently evaporated in a laboratory dryer at 60°C for ten minutes. In this fatigue test, the chosen adhesive composites were loaded with zero-start pulsating compression stresses of 55 MPa. The tests were performed on an Instron 8501 testing machine with a frequency of 20 Hz. The research concerned adhesive composites whose destruction in static tests was typically cohesive [7]. This was done in order to find out what would be the destruction of joints made of this material in a fatigue test, and whether its fatigue life would be longer than that of joints undergoing adhesive destruction, e.g. those made of Epidian 57/Zl. Apart from that, s correlation was sought between the fatigue life of the materials themselves and the joints made of them.

Subsequently, the materials were subjected to a fatigue life test in lap joints (Fig. 2b). The specimens were loaded with zero-start pulsating shear stresses in the range from 0.1–1.5 kN, that is, about a half of the shear stress. The tests were performed on an Instron 8501 testing machine with a frequency of 20 Hz.

3. Research on the durability of adhesive composites

3.1. Long-term strength

In the literature there are no all-purpose prognostic methods concerning long-term strength that can be applied in practice. Searching for a direct relation between the short-term and long-term strength of adhesive composites is fallible. The example can be the research results presented in the information material [4], where long-term strength of Araldit AV 138 adhesive in 10000 h test is 75% of the short-term strength and for Araldit AW 106 adhesive it is only 25%.

The manufacturers of adhesives hardly ever define durability of their products to long-term loads. A credible method of determining the longterm strength of adhesive joints is by conducting an experimental analysis; and still, it involves considerable cost and time-consuming research [10, 13, 18].

It was decided to estimate the permissible values of loads of the adhesive composites with regard to their static long-term strength. On the basis of the methodology used in the work [13], creep curves for the selected adhesive composites were determined. On the basis of previous research it seemed that ambient temperature was too low to gain high long-term strength of adhesive composites made of epoxy resins [14]. Therefore, the curing process was carried out by a single stage (at ambient temperature) and by a double stage (at increased temperature) in order to define the influence of curing conditions of adhesive composites on their long term strength [15]. It seems that this state of facts is related to a level of cross-linking of an adhesive. The adhesive cured in the increased temperature is characterized by a higher level of cross-linking, and therefore, it has got different physics and mechanical properties. The research showed that some adhesive composites were characterized by a rapid increase in strains in the increased temperature that caused permanent deformations or destruction of the examined specimen within several or several dozen minutes (Fig. 6). Therefore, these materials were excluded from the further examination.

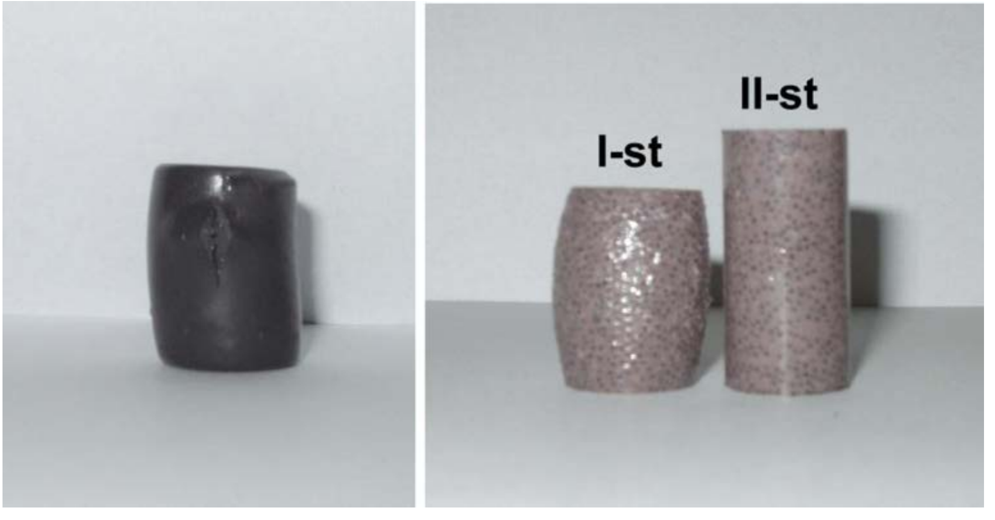

Fig. 6. Unirep 1 and Belzona 1812 (after single-stage stage curing) adhesive composites after a few minutes from the moment of 30 MPa load at a temperature of 60°C.

Creep curves for other materials were presented in Fig. 7. For the purposes of comparative analysis, the creep curves for an adhesive based on physically unmodified epoxy resin Epidian 57 were also plotted on the graph. From among the adhesive composites discussed, the least increase in deformation was visible for Belzona 1812 (double-stage curing) adhesive composite. Unirep 3 and Belzona 1111 also had a good resistance to creep, whereas Epidian 57 had a rather bad resistance.

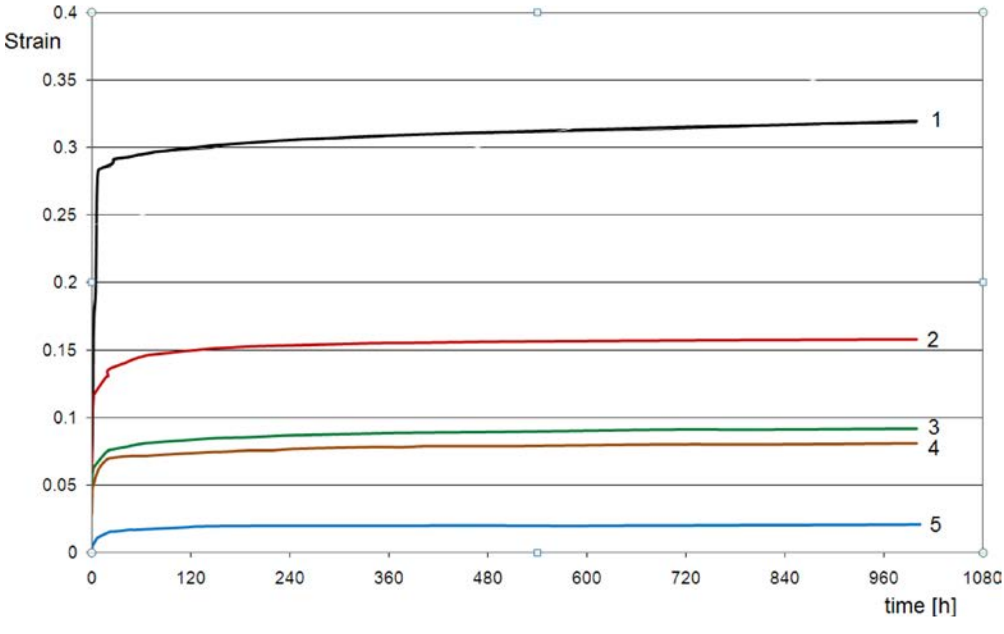

Fig. 7. Creep curves of the adhesive composites in the temperature of 60°C with load causing normal stresses of 30 MPa in the examined specimens (1 – Epidian 57, 2 – Chester Metal Super, 3 – Belzona 1111, 4 – Unirep 3, 5 – Belzona 1812 – II st.).

Subsequently, the long-term strength of adhesive composites was determined. The measure of long-term durability was the lapse of time between the commencement of the experiment and specimen destruction. With regard to the assumed expediency of repairs, 500 hours was the given maximum time of a test. The research was conducted in temperatures of 60, 80 and 100°C taking the possible operational temperature of the repaired elements under consideration. The research results are presented in a form of column charts where time of the certain specimen destruction was marked.

Joints made of Belzona 1111, Chester Metal Super, Unirep 3, and double-stage cured Belzona 1812 were characterized with a durability of 500 h at a temperature of 60°C (none of the examined specimen were destroyed); see Fig. 8.

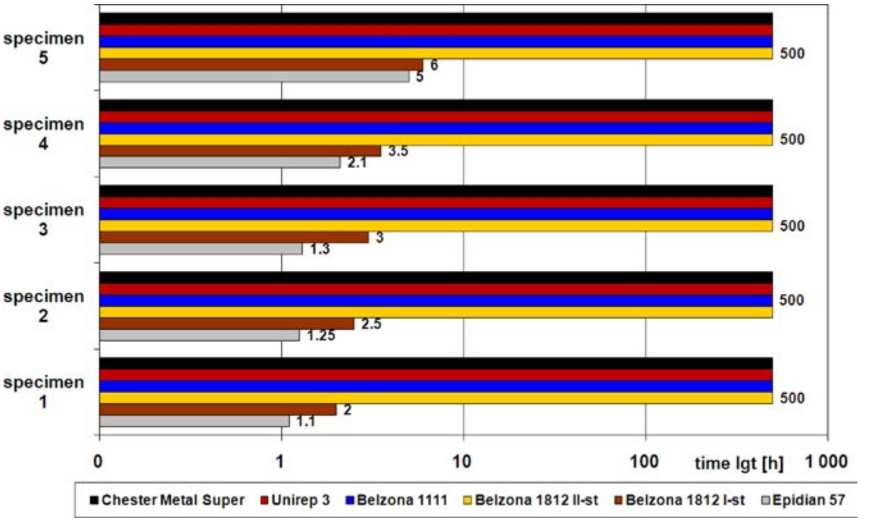

FIG. 8. Static long-term strength of lap joints made of adhesive composites (loaded with a power of 60% F_n and at a temperature of 60°C).

It was stated that even a preliminary estimation of adhesive creep curve shapes (Fig. 7) enabled the prediction of their ability to transfer long-term loads (Fig. 8), since there is a clear relation between the nature of a composite's creep curve and its ability to long-term load transfer in adhesive joints. Since the durability of adhesive joints did not change considerably at a temperature of 80°C, further research was conducted in temperature increased to 100°C (Fig. 9).

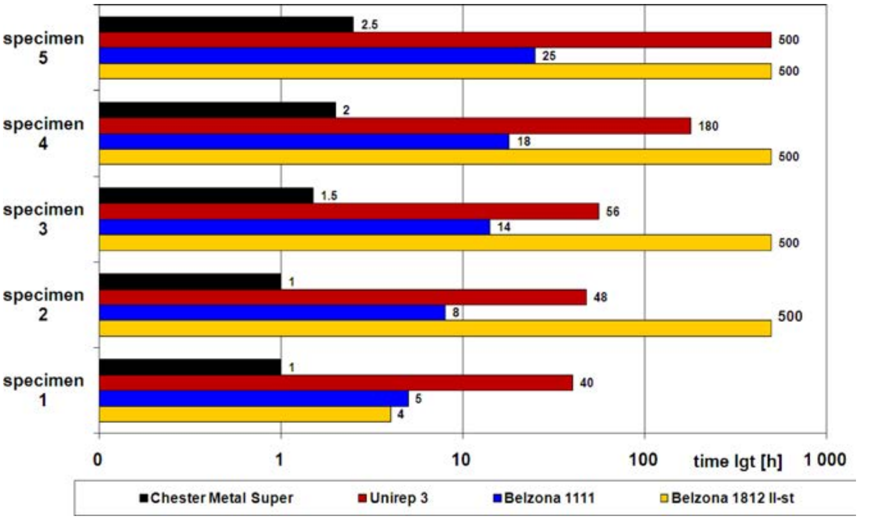

FIG. 9. Static long-term strength of lap joints made of the adhesive composites (load with power of 60% F_n and temperature of 100°C).

At a temperature of 100°C, most of the examined specimens were destroyed before the assumed time of 500 h passed. The specimens first destroyed were made of Chester Metal Super. Specimens made of Belzona 1111 were destroyed within the first day and night. Better durability was shown by specimens made of Unirep 3, since it lasted from several dozen to several hundred hours; and one of the examined specimens was not destroyed within the assumed 500 h. The best durability was shown by specimens made of double-stage cured Belzona 1812, which is the material designed for operation at increased temperature. However, specimen one showed considerably worse durability than the rest of them. This was probably caused by some fault in the preparation of the specimen (e.g. air bubble inside).

The conducted research proved the thesis that the shortterm strength of adhesives cannot be the only criterion of its usefulness in executing repairs in crucial structures which will have to operate at an increased temperature for quite a long time. On their basis it is hard to find an unambiguous quantitative correlation between creep curves determined for the adhesive composite itself (treated as a material) and the durability of adhesive bonds. Still, one can attempt to give some rough interpretation of this relation and state that if strain of adhesive composite subjected to load at increased temperature (in this case 60°C) does not exceed 20% of specimen's dimension in the assumed time, joints made of this composite should be characterized by durability of approximately 500 h for loads that are equal to a half of the short-term strength (in case of the research conducted here, that was 60% of the shear strength). Moreover, creep curves are valuable sources of information concerning conditions (and especially thermal conditions) in which an adhesive joint can be used for a long time. An example here can be the relation $\varepsilon = f(t)$, gained for Epidian 57, where the adhesive showed low durability in joints with great strain caused by creep.

3.2. Fatigue life

The research on fatigue life of different adhesive joints [1, 3, 9] has shown that fatigue life of these types of joints is much less considerable than their short-term strength. Predicting the fatigue life of adhesive joints is a complicated issue and requires numerous tests [5]. However, using adhesives for the repair of technical objects, one should take the possibility of fatigue loads under consideration. A lot of research has proven that fatigue life of adhesive joints is actually dependent on the maximum reduced stresses present in bonding with the maximum value of load of the fatigue cycle. This thesis has been proven true in the research, which compared the fatigue lives of joints that differed considerably from each other in shape and load method, and which had similar adhesive bonds – the same type of adhesive, identical method of adhesive surface preparation, and similar thickness of bonding (Fig. 10).

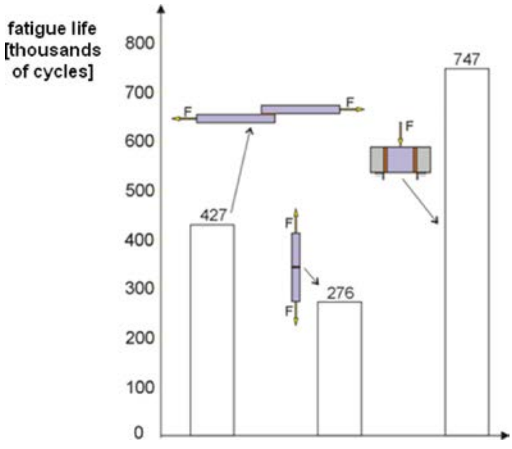

FIG. 10. Influence of the specimen's shape and load method on the average fatigue life of joints made of epoxy adhesive Epidian 57/Z1 [5].

During research on fatigue life the adhesive composites of the "super metal" group were loaded with zero-start pulsating compression stresses of 55 MPa, that is, equal to a half of compression strength. The research on Belzona 1111 and Unirep 3 was terminated after approximately 750,000 cycles. Specimens made of Chester Metal Super material were destructed after a min. of 2,785 and max. of 21,950 cycles (Fig. 11). The short fatigue life of Chester Metal Super adhesive composite has been confirmed by similar research, in which fatigue life was determined for slightly longer cylinder specimens of the same diameter ($l = 31.5$ and $\phi = 12.6$) with a zero-start pulsating load of 56 MPa at a frequency of 10 Hz. While Belzona 1111 and Unirep 3 adhesive composites reached the assumed value of fatigue life of 100,000 cycles, specimens made of Chester Metal Super composites were destroyed after 2,600 cycles on average [20].

a) b) c)

FIG. 11. View of specimens after fatigue tests: a) Chester Metal Super, b) Unirep 3, c) Belzona 1111.

The adhesive composites of the "rapid" group were loaded in fatigue tests with zero-start pulsating compression stresses of 28.5 MPa, that is, a half of the compression strength of adhesive composites of the lowest strength. After 1,483 cycles, the Unirep 1 specimen was subject to permanent deformation. The other was not destroyed after they were loaded by an approximate number of cycles of 1,000,000. The experiment was terminated and the load was increased to 41 MPa, that is, approximately 0.57 of the compression strength of Belzona 1221 and approximately 0.43 of Chester Metal Rapid. The Belzona 1221 specimen deformed plastically after 8,634 load cycles. The Chester Metal Rapid specimen transferred 210,110 load cycles without destruction (Fig. 12).

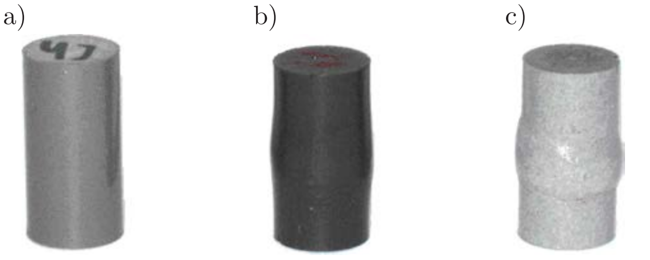

Fig. 12. View of specimens after fatigue tests: a) Chester Metal Rapid, b) Unirep 1, and c) Belzona 1221.

The research conducted here showed that the Chester Metal Super composite was characterized with a definite shorter fatigue life in comparison to the other materials examined in this group, while the longest fatigue life from the “rapid group” had a Chester Metal Rapid composite; and that is comparable to materials of “super metals” group (Fig. 13).

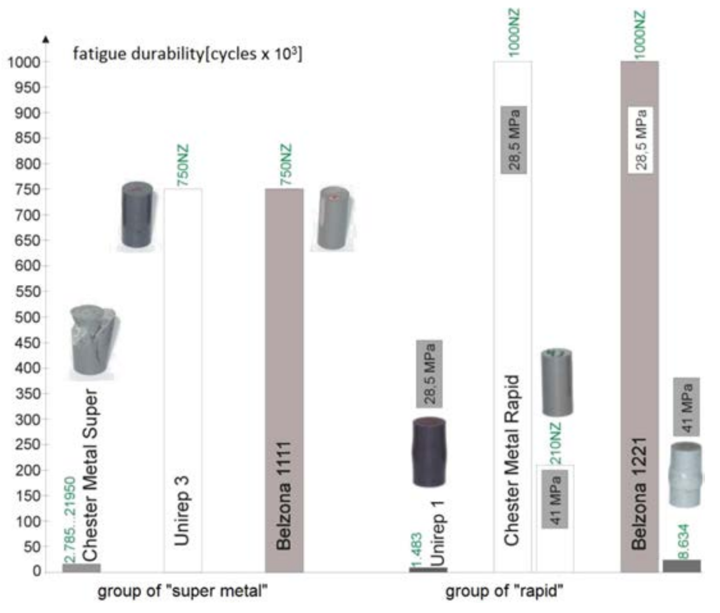

Fig. 13. Fatigue life of the adhesive composites examined (NZ – specimen was not damaged during the test).

Further research on fatigue life was carried out for adhesive joints made of the selected adhesive composites using tensile loaded cylindrical specimens (Fig. 2c) During the research destruction of the cohesive type was noticed in the case of every examined specimen made of Belzona 1111. What is more, it was stated that the examined composite was characterized by longer fatigue life in comparison with Epidian 57/Z1 subject to adhesive destruction. The examined joints made of Chester Metal Super composite showed long fatigue life, despite the fact that Chester Metal Super itself is characterized by a short fatigue life, and that that indicates no correlation between the fatigue life of the adhesive composite itself and tensile loaded bonds made with them. The most probable reason for this state of fact is the relatively high stress-strain strength of this material [6]. The research conducted also showed that the fatigue life of the Chester Metal Super composite examined in bonds was rapidly decreasing with an increase of the maximum load of a fatigue cycle from approximately 05 to approximately 06 of the short-term strength. The Chester Metal Rapid composite showed in this test a shorter average fatigue life of joints in relation to the composites of the "super metal" group, that is, Belzona 1111 and Chester Metal Super. This fact was probably caused by adhesive destruction of joints.

Subsequently, the materials discussed were subjected to a fatigue life test in lap joints (Fig. 2b). The tests were executed in two stages. In the first stage the absolute shear fatigue life was determined, since the adhesive composites investigated were subjected to the same load of 13 kN. In the second stage the adhesive composites were subjected to different loads that had value of $0.66F_n$. As a result, a relative shear fatigue life was determined. The fatigue life of Belzona 1111 turned out in single lap joints to be longer than that of Chester Metal Super; contrary to the situation with tensile loaded joints. In the Fig. 14 fatigue lives of specimens loaded with identical cycles and joined with different adhesive composites are compared.

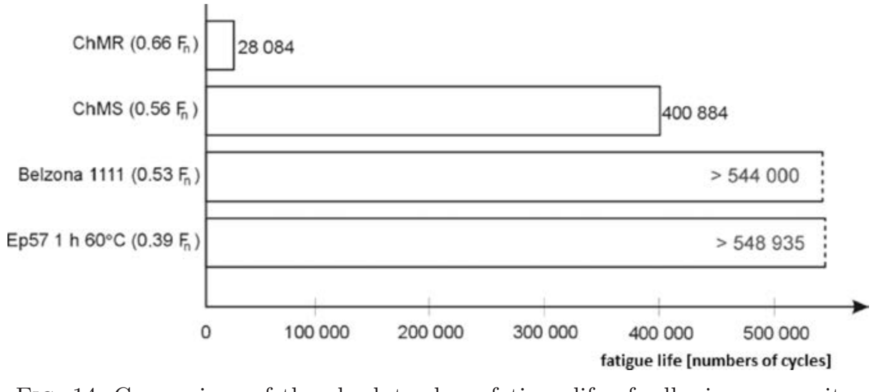

Fig. 14. Comparison of the absolute shear fatigue life of adhesive composites (the maximum load of a fatigue cycle was 1.3 kN for all adhesives).

The shortest fatigue life was visible for specimens joined with Chester Metal Rapid; and still, their relative load was the greatest. Belzona 1111 and Epidian 57/Z1 showed a long absolute fatigue life with the applied load.

The relative fatigue life of specimens joined with Belzona 1111 turned out to be longer than that of specimens joined with Epidian 57/Z1. It was stated that fatigue load of joints made with Chester Metal Rapid and Epidian 57/Z1 at the level of 066 of shear strength was unacceptable with regard to low durability of joints loaded in this way (Fig. 15).

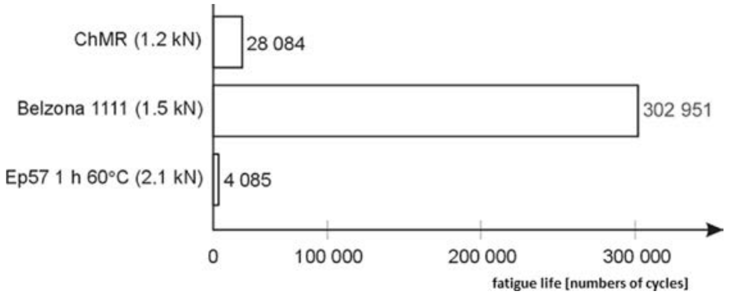

Fig. 15. Comparison of the relative shear fatigue life of adhesive composites (the maximum load of fatigue cycle was $\sim 0.66 F_n$).

During the fatigue tests, the strains of adhesive composites were determined which were exemplified by cylinder specimens cast from the examined adhesive composite (Fig. 2a). The specimens were subjected to compression stress for which the maximum load of fatigue life was $\sim$0.5 of short-term compression strength. A position of the moving traverse of testing machine was measured after every thousand cycles. The conducted test allowed for the determination of the strain amplitude of the samples, as well as the change in height resulting from movement of the traverse. The exemplary result of this research is presented in Fig. 16.

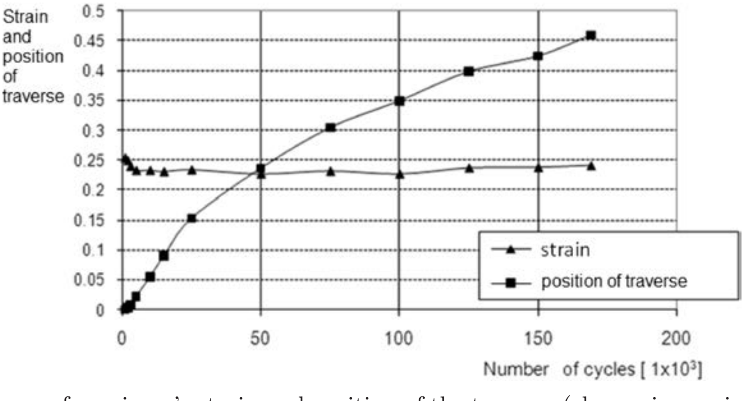

Fig. 16. Change of specimen's strain and position of the traverse (change in specimen's height) during fatigue tests of Belzona 1111 adhesive composite.

It was noticed that after several thousand load cycles, the strain amplitude of the specimen was defined on practically a constant level. Simultaneously, a constant increase of permanent strain of the examined specimen was noticed (by a decrease in height). The nature of the observed strains explicitly shows that the examined material was subject to creep (cf. Fig. 7).

4. Conclusion

On the basis of the research presented here, the following conclusions can be drawn:

- There is a quality relation between the nature of the creep curve of the composite itself and the durability of joints made with this composite. The composites, after being loaded at an increased temperature, showed greater initial strains and a greater gradient of increase in strains in time, also showed lower durability at increased temperature (from several to several dozen hours).
- On the basis of the tests conducted so far, it is hard to find a direct quantitative correlation between creep curves and the analysed strength of adhesive bonds. Still, it can be stated that if strains of adhesive joints subjected to loads at an increased temperature (in this case 60°C) do not exceed 10% within several hundred hours, then the joints of this adhesive composite should show at least 500 h durability at a temperature of up to 80°C with load that is equal to a half of short-term strength in ambient temperature.
- It was stated that for a specific level of loads of adhesive joints (in this case it is 60% of the short-term strength), there is a temperature value near to which the durability of shear loaded bonds is rapidly decreasing. For bonds made of adhesive composites that was at a temperature of 100°C.
- The temperature and time of adhesive composites' curing have a significant influence not only on shortterm strength of adhesive composites, but also on their durability. The increased temperature increases the extent of cross-linking of epoxy matrix; thanks to which the material is more resistant to creep at an increased temperature.
- Fatigue destruction of adhesive bonds can be of an adhesive or cohesive nature, and a longer fatigue life is shown by bonds which undergo cohesive damage during shortterm strength tests. On the other hand, a fatigue life of composite adhesive bonds cannot be predicted on the basis of fatigue life of these materials themselves.
- Both long term strength and fatigue life of adhesive joints made with the use of adhesive composites can be rapidly reduced with a slight increase of their load.

- It seems that, in the mechanism of fatigue destruction of adhesive joints, the process of adhesive material creep is of great significance since, as it has been proved, this occurs even at ambient temperature.
- Value of adhesion's forces has a considerable influence on the shortterm strength of adhesive joints, and has a less considerable influence on their fatigue life. Therefore, using procedures that increase the strength of adhesive joints is always advisable; and yet, one should bear in mind that it will not have directly proportional influence on their fatigue life.
- With regard to the requirements of expedient (temporary) repairs executed in field conditions, the long-term strength of adhesive joints longer than 50 hours and a fatigue life of adhesive joints higher than 100,000 cycles, can be accepted as sufficient in most cases. Therefore, a safe value for the maximum long-lasting and fatigue loads of theadhesive composites investigated that ensures the required durability of joints made of these composites should be 0.5 of the short-term strength.

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