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Original Research

THE INFLUENCE OF AGING ON THE LOAD CAPACITY OF ADHESIVE LAP JOINTS MADE OF ALUMINUM ALLOY EN AW-2024-T3

WPŁYW STARZENIA NA NOŚNOŚĆ POŁĄCZEŃ KLEJOWYCH ZAKŁADKOWYCH ZE STOPU ALUMINIUM EN AW-2024-T3

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Abstract

The aim of the study was to assess the effect of environmental ageing on the load capacity of single-lap adhesive joints made of EN AW-2024-T3 aluminum alloy, subjected to and not subjected to pneumatic shot peening. Adhesive joints were made using two-component epoxy adhesive Loctite EA 3430. Half of the formed joints were strengthened by pneumatic shot peening of the overlap zone. The shot peening time was 120 s, the ball diameter was 1.0 mm, and the compressed air pressure was 0.5 MPa. The adhesive joints were then subjected to different variants of ageing in natural conditions. Individual variants differed in the length of ageing. The next step was to examine the load capacity of the adhesive joints using a static tensile test. The results were subjected to statistical analyzes, including analysis of variance (ANOVA), regression and correlation analysis, and Student's t test. Based on the analyzes, it was found that for the assumed range of input factor variability, ageing of joints not subjected to shot peening increased their load capacity by 38%. Aging of joints strengthened by shot peening resulted in a 62% decrease in their load capacity. The ageing length had a significant effect on the load capacity of the adhesive joints.

Keywords: adhesive joints, aging, shot peening, strengthening of adhesive joints

Streszczenie

Celem pracy była ocena wpływu starzenia środowiskowego na nośność połączeń klejowych jednozakładkowych ze stopu aluminium EN AW-2024-T3, które poddano i nie poddano procesowi pneumokulkowania. Połączenia klejowe wykonano z użyciem dwuskładnikowego kleju epoksydowego Loctite EA 3430. Połowę utworzonych połączeń umocniono za pomocą pneumokulkowania strefy zakładki. Czas pneumokulkowania wynosił 120 s, średnica kulek 1,0 mm, a ciśnienie sprężonego powietrza 0,5 MPa. Następnie, połączenia klejowe poddano różnym wariantom starzenia w warunkach naturalnych. Poszczególne warianty różniły się długością starzenia. Kolejnym krokiem było zbadanie nośności połączeń klejowych za pomocą statycznej próby rozciągania. Uzyskane wyniki poddano analizom statystycznym, które obejmowały przeprowadzenie jednoczynnikowej analizy wariancji ANOVA, analizy regresji, korelacji i testu t-Studenta. Na podstawie przeprowadzonych analiz stwierdzono, że dla przyjętego zakresu zmienności czynników wejściowych, starzenie połączeń niepoddanych pneumokulkowaniu, zwiększyło ich nośność o 38%. Starzenie połączeń umocnionych w procesie pneumokulkowania spowodowało natomiast zmniejszenie ich nośności o 62%. Długość starzenia miała istotny wpływ na nośność połączeń klejowych.

Słowa kluczowe: połączenia klejowe, starzenie, pneumokulkowanie, umacnianie połączeń klejowych

1. Introduction

Adhesive joints are exposed to many factors that can affect their strength and durability. Awareness of the existence of such factors and knowledge of the potential effects of their impact are crucial from the point of view of designing safe adhesive joints.

The mechanical properties of adhesive joints can be significantly affected by daily and annual fluctuations in temperature and air humidity. Water can cause changes in the physical and mechanical properties of the adhesive as well as damage the bonds between the adhesive and the adherend (Bowditch, 1996). The results of the analyzes that examine the effect of moisture on adhesive joints by immersing them in water indicate that the negative impact of the aquatic environment on the adhesive joint increases with increasing exposure time and water temperature (Hirulkar et al., 2020).

Another degrading factor may be the ambient temperature. As a result of long-term exposure to elevated temperatures (e.g., in the summer season), the adhesive undergoes the so-called thermal degradation. In the first stage of degradation, the mechanical strength of the joints increases as a result of the additional crosslinking of the adhesive structure. However, in the later phase of the process, excessive crosslinking of the adhesive structure occurs or its molecular weight is reduced. As a result, the strength of the joints decreases (Rojek, 2011).

The phenomenon of increasing the strength of the joints in the first phase of the degradation process can be used to strengthen the adhesive joints. It has been shown that the additional heating of joints during curing has a beneficial effect on their static strength at both ambient and elevated temperature (Szabelski et al., 2016). It has been proven that the strength of adhesive joints increases as a result of their aging at high temperature (150-200˚C). The authors of the study associated the improvement of strength properties with additional crosslinking of the adhesive structure and reduction of stress concentration resulting from uneven polymerization shrinkage on the bonded surfaces (Morcos et al., 2022). The improvement in strength properties was also obtained as a result of the aging of adhesive joints subjected to cyclic daily and annual temperature fluctuations in natural conditions or in a thermal shock chamber (Zielecki et al., 2021).

Another factor that should be considered when designing adhesive joints is the possibility of using additional procedures to increase their strength. Methods of strengthening adhesive lap joints include, among others: rounding the edge zone of the lap (Çalık, 2016, Zhao, 2011), narrowing the ends of the lap (Belingardi, 2002), creating steps in the lap area (Durmuş, 2019), making an undercut along the edge of the adhesive joint (Bahrami, 2019), sandblasting (Godzimirski, 2018) or pneumatic shot peening of the lap zone (Zielecki, 2008, Ozga, 2023).

Pneumatic shot peening of the lap zone is one of the simplest and most affordable methods of strengthening adhesive joints. As a result of this procedure, the adherend is deformed, and compressive stresses are created in the bond line. The introduced stresses are added to the stresses coming from the external (shearing) load. As a result, the degree of stress concentration in the edge zone of the bond line is reduced and the strength of the joint is increased. It has been shown that as a result of pneumatic shot peening, the strength of adhesive overlap joints made of S235JR steel can be increased by as much as 93-112% (Zielecki, 2008).

To sum up, the literature analysis indicates that exposing adhesive joints to elevated temperatures may have a beneficial effect on their strength properties. The load capacity of the adhesive joints can also be increased by pneumatic shot peening of the overlap zone. However, the impact of ageing on the load capacity of adhesive joints subjected to pneumatic shot peening has not been investigated so far. Therefore, the objective of the research presented in the article is to determine the effect of environmental ageing (daily and annual fluctuations in temperature and air humidity) on the load capacity of adhesive joints subjected to and not subjected to the pneumatic shot peening process.

2. Material and methods

2.1. Preparation of adhesive joints

The subject of the study was single-lap adhesive joint made of the EN AW-2024-T3 aluminum alloy designated according to PN-EN 485-2+A1:2018-12 standard. Due to its good fatigue resistance, this alloy is used, among others, in the aviation and military industries, for example, in landing gear structures and wing tensioning elements. This alloy is not suitable for welding and anodizing. It is characterized by low corrosion resistance and good workability (Dobrzański, 2012, PN-EN, 2018-12).

The adhesive joints were made using the two-component epoxy adhesive Loctite EA 3430 (Loctite, Düsseldorf, Germany). This is a general-purpose adhesive. It cures quickly at room temperature. Increasing the ambient temperature also shortens the curing time (Technical Data Sheet, 2024). Table 1 presents the physical properties of the Loctite EA 3430 adhesive.

The first stage of the process was to prepare the adherend surfaces. In order to properly develop the geometric structure, the surfaces were subjected to abrasive blasting with 95A electrocorundum with a grain size of 0.27 mm using a New-Tech sandblasting cabinet (New-Tech, Wrocław, Poland). The values of the roughness parameters, measured using a Taylor Hobson SURTRONIC 25 contact profilometer (Taylor Hobson Ltd, Leicester, England) were: Rz = 25.95 μm, Ra = 4.53 μm, Rq = 5.67 μm, RSm = 0.141 mm, Rku = 2.99 (average values determined based on the measurements carried out for 6 randomly selected samples). The measurements were carried out in accordance with the standard PN-EN ISO 21920-2:2022-06. After mechanical processing, the bonded surfaces were degreased with acetone.

Table 1. Selected Physical properties of Loctite EA 3430 adhesive

Physical properties[1]		
Property	**Unit of measurement**	**Value**
Coefficient of thermal conductivity	W/(m·K)	0.3
Tensile strength	N/mm^2	36
Tensile modulus	N/mm^2	3 210
Compressive strength	N/mm^2	65
Elongation	%	2
Shore hardness, (durometer D)		70
Glass transition temperature	°C	58

[1] Cured for 7 days at a temperature of 22 °C; 1.2 mm thick samples.

The next stage was to create adhesive joints. The adhesive components were mixed manually in a 1:1 ratio, in accordance with the manufacturer's recommendations. After the components were mixed, a small amount of the adhesive composition was applied to adherend surfaces and spread using a triangular comb device. This made it possible to evenly distribute the composition on the surface and remove excess of the composition to obtain the appropriate thickness of the bond line. The elements with the applied layer of adhesive were placed in a fixing device. The device ensured the achievement of the appropriate dimensional and shape accuracy of the adhesive joints, prevented the movement of the elements, and allowed for exerting even pressure on the constituted adhesive joint using one-kilogram weights. The application of pressure allowed for better filling of micropores and micro-irregularities of the surface, facilitating the spreading of the adhesive and the fixing of the joined elements. When selecting the weight of the weights, care was taken to avoid excessive leakage of the adhesive between the bonded surfaces or deformation of the structure. The constant amount of adhesive, its viscosity, and the constant pressure force determined the thickness of the adhesive joint, which was 0.09 ± 0.01 mm. A simplified scheme of adhesive joints is shown in Fig. 1.

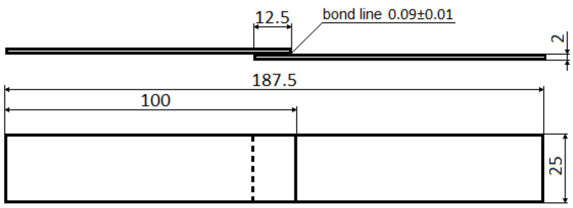

Fig. 1. Simplified scheme of an adhesive joint

The samples were subjected to a single-stage cold curing process for 72 hours (3 days) at a temperature of 22±1°C.

2.2. Pneumatic shot peening

Half of the adhesive joints were subjected to the pneumatic shot peening process to increase their load capacity. The pneumatic shot peening time was 120 s, the ball diameter was 1.0 mm, and the compressed air pressure was 0.5 MPa. Pneumatic shot peening was applied only to the surface of the joint overlap. The remaining part of the sample was covered. Both sides of the joint overlap were treated. The scheme of the adhesive joint prepared for pneumatic shot peening is shown in Fig. 2.

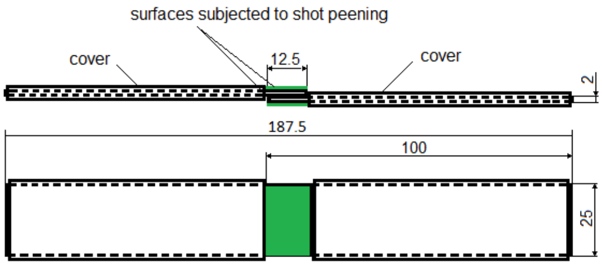

Fig. 2. Scheme of an adhesive joint prepared for pneumatic shot peening

2.3. Aging

The adhesive joints subjected to and not subjected to shot peening were aged in natural conditions. The aim of ageing was to determine the effect of long-term exposure to atmospheric conditions (daily and annual fluctuations in temperature and air humidity) on the load capacity of the adhesive joints subjected and not subjected to shot peening. The analyzes were carried out for 5 aging variants, which differed in length and months in which they were carried out (Table 2).

The samples were aged in a sunny location at ground level. Ageing conditions, including air temperature and relative humidity over 24 months (from 12/07/2021 to 12/07/2023), are shown in Figure 3. The measurements were taken in southern Poland.

Table 2. Aging variants

Start date	End date	Length	Marking (not shot peened)	Marking (shot peened)
12/07/2021	12/01/2022	6 months	6NP	6P
12/01/2022	12/07/2022	6 months	6NPW	6PW
12/07/2021	12/03/2022	9 months	9NP	9P
12/07/2021	12/07/2022	12 months	12NP	12P
12/07/2021	12/07/2023	24 months	24NP	24P
Not subject to aging			N	P

2.4. Static tensile test

The load capacity of the adhesive joints was determined using a static tensile test. The tests were carried out according to the methodology described in the PN-EN 1465:2009 standard using a ZWICK/ ROELL Z100 test machine (Zwick/Roell, Ulm, Germany). In order to reduce the influence of tearing on the test results, the machine jaws were moved relative to each other. The force at which the adhesive joint was broken was considered as its load capacity and designated as P_t.

3. Results and discussion

The experimentally determined load capacity values of the adhesive joints are presented in Table 3.

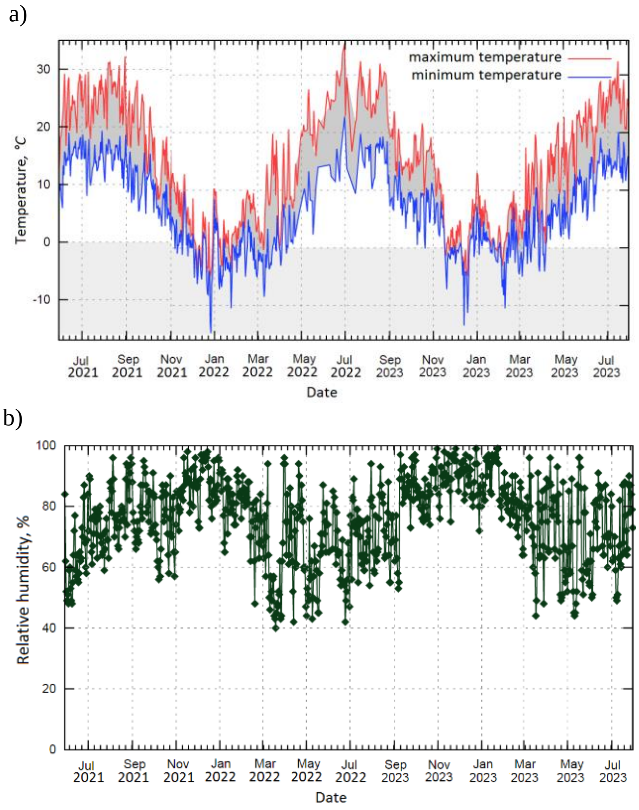

Fig. 3. Air temperature and humidity in the period July 2021 - July 2023 (24 months)

Table 3. Load capacity of adhesive joints

Variant			Load capacity, N							Average load capacity, N	Standard deviation, N
			P_{t1}	P_{t2}	P_{t3}	P_{t4}	P_{t5}	P_{t6}	P_{t7}	P_t	σ
Not subjected to shot peening	Not aging	N	8163	6164	6110	6535	7959	8396	7938	7324	932
	Subjected to aging	6NP	8874	8489	10328	7128	7821	8024	8634	8471	931
		6NPW	5324	7362	5059	6610	7036	5854	4051	5899	1095
		9NP	9916	6889	5071	7272	8652	6710	7458	7424	1418
		12NP	9938	8315	10873	10437	10584	10613	9805	10081	802
		24NP	7160	7876	7374	7614	7215	7559	7411	7458	229
Subjected to shot peening	Not aging	P	9553	9024	9273	9653	9237	10507	10815	9723	629
	Subjected to aging	6P	4573	5116	3889	4072	4284	4826	4978	4534	433
		6PW	4062	3716	5299	5515	4869	4474	4385	4617	601
		9P	7668	5030	4574	4953	5998	4653	2980	5122	1331
		12P	4150	4058	4868	4591	3955	4953	3017	4227	614
		24P	4490	3048	3863	3819	3514	3714	3583	3722	403

Figure 4 shows the average values of the load capacity and the standard deviation for 12 sample variants. The average values were obtained based on 7 measurement samples.

Based on the results, it can be concluded that aging of adhesive joints not subjected to pneumatic shot peening contributes to increasing their load capacity by 38% (maximum). The increase in the load capacity of joints due to aging may be the effect of additional crosslinking of the adhesive structure under the influence of elevated temperature in the summer season. Polymer materials (including adhesives) exposed to long-term high temperature undergo thermal degradation which consists in the breakdown of macromolecules into smaller fragments. As a result of degradation, the crosslinking of the adhesive structure increases, which in the initial phase of the process can contribute to increasing the load capacity of the joints. However, in a later phase, the adhesive structure may undergo excessive crosslinking, the molecular weight may decrease, and, as a result, the strength properties of the adhesive joint may deteriorate (Rojek, 2011, Szabelski et al., 2019). According to the research results, the highest load is achieved by joints aged 12 months. After 24 months, the load capacity is already lower. The reason for the lower load capacity of the connections aged 24 months may be excessive crosslinking of the adhesive structure. Aging for six months from July increased the load capacity of the adhesive joints. In turn, the six-month ageing starting in January decreased the load capacity of the connections. Low winter temperatures did not allow additional crosslinking of the adhesive structure, but could have contributed to damage of the adhesive and cohesive bonds.

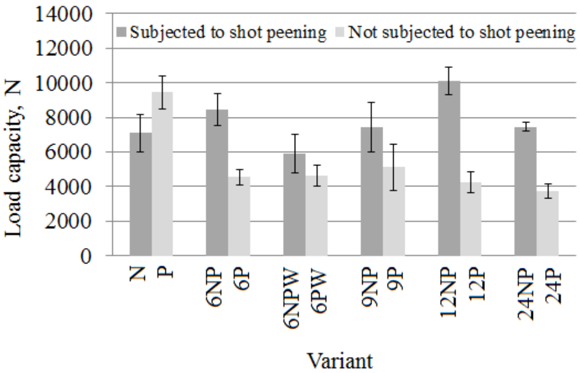

Fig. 4. Average load capacity of adhesive joints with standard deviation

Before aging, the load capacity of the joints subjected to shot peening is significantly higher than the load capacity of the joints that were not subjected to this treatment. As a result of shot peening, the elements are deformed, the edge of the overlap is pressed against the adherend, and compressive stresses are established in the bond line. Therefore, the joint load capacity increases. However, aging of joints previously subjected to shot peening reduces their load capacity by 62%. The reduction in the load capacity of the joints after aging may be caused by adhesive and cohesive bonds damage resulting from too intensive shot peening, degradation of the adhesive structure and the accumulation of stresses introduced during shot peening and resulting from temperature changes during aging. In contrast to connections not subjected to shot peening, six-month aging of shot-peened connections had a similar effect, regardless of when it was started (in summer or winter).

The failure patterns of adhesive joints were analyzed according to the PN-EN ISO 10365:2022-07 standard. The aim of the failure patterns analysis was to check whether the use of the shot peening process had an impact on the nature of the destruction of adhesive joints. The classification of the failure patterns was made on the basis of visual inspection. Figures 5-6 show the view of the deteriorated adhesive joints subjected and not subjected to shot peening and aged for 6 months from July to January.

Fig. 5. Adhesive joints not subjected to shot peening and aged for 6 months from July to January

Based on Figures 5-6, it can be concluded that the nature of the destruction of the joints subjected to and not subjected to shot peening differs significantly. In both cases, however, adhesive and cohesive failure mechanisms can be observed.

In the case of joints not subjected to shot peening (Fig. 5), an adhesive failure (AF) is visible at the ends, while an adhesive-cohesive failure (AF and CF) occurs in the middle of the bond line. The failure patterns change with the distance from the edge of the bond line. Adhesive failure, visible at the ends of the bond line, may indicate that the maximum stresses in joints subjected to tension are located at the ends of the bond line.

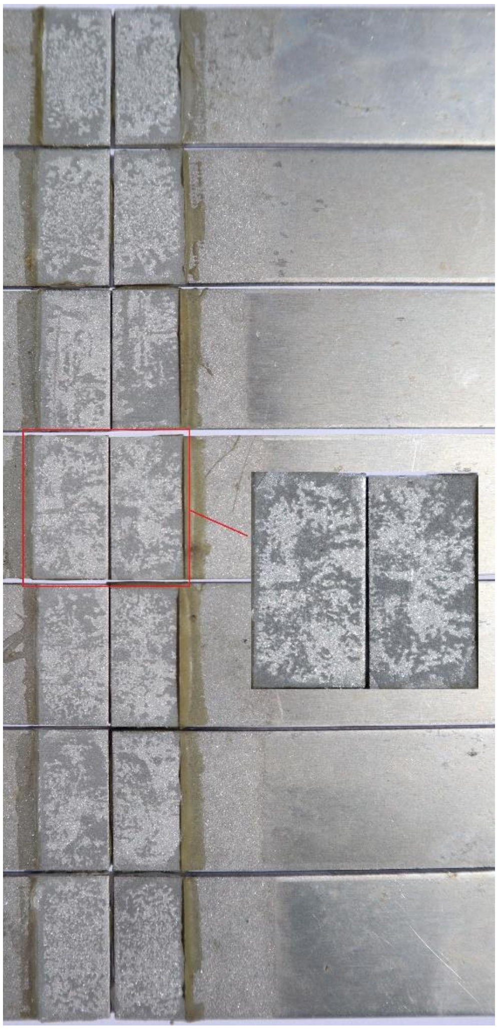

Fig. 6. Adhesive joints subjected to shot peening and aged for 6 months from July to January

In the case of joints subjected to shot peening, an adhesive-cohesive failure (AF and CF) can be observed along the entire length of the bond line. There is no visible adhesive failure zone at the ends of the bond line. Therefore, it can be assumed that, in the case of joints subjected to shot peening, the stress distribution in the bond line of the joint subjected to tension was more uniform than in the case of joints not subjected to shot peening. On the basis of the analysis of failure patterns, it can also be concluded that shot peening of the joint overlap could have affected the strength of adhesive and cohesive bonds.

The load capacity values obtained experimentally were subjected to statistical analyzes. Hypotheses in the statistical analyzes were tested for a significance level of $\alpha = 0.05$. This level of significance is most often used in research in the field of mechanical engineering. The analyzes assumed the normality of the data distribution and the homogeneity of variance. The analyzes were performed using the Minitab and MS Excel programs.

In the first step, an analysis of variance (ANOVA) was performed. The purpose of this analysis was to determine whether the differences between the mean load capacity values of the comparative groups were the result of chance or a deliberate change in the level of the input factor, which is the length of aging. The analysis was performed separately for joints subjected to and not subjected to shot peening. The results of the tests obtained for joints aged 6 months from January (variants 6NPW and 6PW) were not subjected to statistical analyzes, because these joints were characterized by a lower load capacity than joints aged 6 months from July. The results of the analyzes are presented in Tables 4-5.

Table 4. Results of analysis of variance (ANOVA) - adhesive joints not subjected to shot peening

Source	**Degrees of freedom**	**Sum of squares**	**Mean sum of squares**	**F statistic**	**Probability (*Pv1*)**
Regression	3	32634905	10878302	10.44	<0.001
Residual Error	24	25010438	1042102		
Total	27	57645343			

Table 5. Results of analysis of variance (ANOVA) - adhesive joints subjected to shot peening

Source	**Degrees of freedom**	**Sum of squares**	**Mean sum of squares**	**F statistic**	**Probability (*Pv1*)**
Regression	3	7235076	2411692	3.31	0.037
Residual Error	24	17488549	728690		
Total	27	24723625			

Based on the results of the analysis of variance (ANOVA), it can be stated that for the assumed range of input factor variability, the aging time has a significant effect on the load capacity of adhesive joints not subjected to pneumatic shot peening (Table 4) and subjected to pneumatic shot peening (Table 5). This is confirmed by the probability *Pv1* values, which in both cases are less than 0.05.

The conclusions of the analysis of variance are confirmed by the box plots presented in Figure 7b.

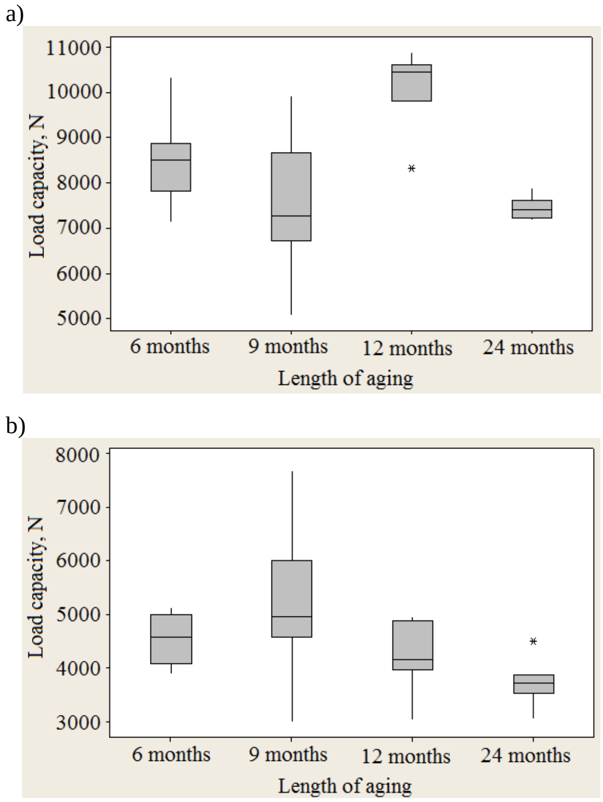

Fig. 7. Box plots: a) adhesive joints not subjected to shot peening, b) subjected to shot peening

Based on the box plots, it can be concluded that the smallest interquartile range is observed for connections aged for 24 months, which indicates the smallest variation in the load capacity of the connections belonging to this variant. In this case, ageing leads to stabilization of the strength properties of the connections. The greatest variation in the load capacity values is observed for the connections aged 9 months. The symbol (*) in the figures indicates the upper extreme value (value greater than the sum of the third quartile value and the interquartile range multiplied by 3) and the lower extreme value (value less than the difference of the third quartile value and the interquartile range multiplied by 3).

Analysis of variance (ANOVA) allowed to state that at least one of the analyzed groups differed significantly from another group. To check which groups differed statistically significantly, the Student's t test was performed. Student's t test was used to compare the mean values of load capacity between two groups. The results of the test are presented in Tables 6-7.

Table 6. Student's t test results for joints not subjected to shot peening

***Pv2* [%]**	**N**	**6NP**	**9NP**	**12NP**	**24NP**
N	-				
6NP	2.706	-			
9NP	44.375	8.018	-		
12NP	0.007	0.385	0.141	-	
24NP	37.055	1.866	47.750	0.006	-

Table 7. Student's t test results for joints subjected to shot peening

***Pv2* [%]**	**P**	**6P**	**9P**	**12P**	**24P**
P	-				
6P	<0.001	-			
9P	0.002	16.812	-		
12P	<0.001	16.991	8.559	-	
24P	<0.001	0.278	2.112	5.986	-

In the case of connections not subjected to shot peening (Table 6), a statistically significant difference was obtained in the average load capacity for connections aged for 12 months. Statistically significant differences in the average load capacity also occur between connections that were not aged (N) and aged for 6 months (6N) and between connections that were not aged (N) and aged for 24 months (24N). In the case of connections subjected to shot peening, the average load capacity of connections that were aged and connections that were not aged, connections that were aged for 6 months (6P) and 24 months (24P), as well as, connections that were aged 9 months (9P) and 24 months (24P) are statistically significantly different. The results indicate that ageing can significantly increase the load capacity of connections that were not previously strengthened by shot peening. The best effect is observed in the case of ageing for 6 and 12 months. Aging of shot-peened joints significantly reduces their load capacity (irrespective of the ageing length).

In order to check the strength of the linear relationship between the input variable (ageing length) and the output variable (load capacity of adhesive joints), the values of the Pearson linear correlation coefficient $r_{y|x}$. were calculated. The linear correlation coefficient can range from -1 to 1. The closer the coefficient value is to 0, the weaker the linear relationship between the variables. As part of the statistical analyzes, linear regression equations were also determined. The equations describe the effect of the length of ageing on the load capacity of adhesive joints. The results of the regression and correlation analyzes are presented in Table 8.

Table 8. Results of regression and correlation analysis

Output variable	Input variable	Regression equation	*Pv3*[2]	Coefficient of determination	Correlation coefficient $r_{y\|x}$
Load capacity (joints not subjected to shot peening) P_{tNP}	Length of aging T_s	$P_{tNP} = 8951 - 46.4T_s$	0.258	1.23%	-0.22
Load capacity (joints subjected to shot peening) P_{tP}	Length of aging T_s	$P_{tP} = 5162 - 59.7T_s$	0.021	18.72%	-0.43

[2] *Pv3* - probability level for an independent variable in regression analysis

Based on the regression equations and the Pearson linear coefficients, it can be concluded that the load capacity of the joints decreases with increasing aging length. However, in the case of regression analysis for joints not subjected to shot peening, the probability value *Pv3* is greater than 0.05. Therefore, the regression equation cannot be used to describe the relationship between the load capacity of the joints and the length of aging. The values of the coefficient of determination indicate that the changes in load capacity are the result of the change in aging length by approximately 2% for joints not subjected to shot peening and by approximately 19% for shot peened joints.

4. Conclusions

Based on the analyzes, it can be stated that for the assumed range of input factor variability, the ageing of the joints not subjected to shot peening contributed to an increase in their load capacity by 38%. The greatest increase in load capacity was observed in the case of ageing for 12 months. Extending the ageing time to 24 months resulted in a slight decrease in load capacity. However, samples aged for 24 months are characterized by the smallest dispersion of load capacity values, which may indicate stabilization of strength properties as a result of ageing. The six-month ageing that began in July increased the load capacity of the joints. In turn, six-month ageing started in January decreased the load capacity of connections.

Aging of joints subjected to shot peening reduced their load capacity by 62%. The lowest load capacity was observed for joints aged 24 months.

Statistical analyzes have shown that the length of ageing can have a significant effect on the load capacity of adhesive joints.

It has been shown that there is a weak linear negative correlation between the extension of the ageing time and the load capacity of the adhesive joints.

In summary, the results prove that aging of adhesive joints is justified, unless they were previously subjected to strengthening in the pneumatic shot peening process. It is also worth noting that aging has a positive effect on the strength properties of joints when it is carried out in the summer months (not winter).

Further research could be directed towards determining the effect of ageing on the load capacity of joints shot peened with lower intensity. It is also justified to investigate the effect of thermal shocks on the load capacity of joints subjected to and not subjected to shot peening.

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