COMPARATIVE ANALYSIS OF COMPRESSIVE STRENGTH OF REGENERATIVE MASSES MADE FROM EPOXY ADHESIVE COMPOUNDSRFACES ROUGHNESS OF FLAT CERAMIC ELEMENTS AFTER LAPPING

Analiza porównawcza wytrzymałości na ściskanie mas regeneracyjnych wykonanych z epoksydowych związków adhezyjnych

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Abstract: The aim of this paper is to analyse statistically the compressive strength of two epoxy regenerative masses with different properties, which were subjected to the same three seasoning conditions. The first epoxy adhesive compound contained Epidian 5 epoxy resin mixed in mass ratio 100:80 with PAC curing agent, while the second epoxy adhesive compound consisted of Epidian 5 epoxy resin mixed in mass ratio 100:10 with Z-1 curing agent. The epoxy adhesive compounds were subjected to three seasoning variants. The first seasoning variant was carried out under normal conditions, at a temperature of 29°C ± 1°C and at humidity of 20% ± 1%, for 1 week. The other variants of seasoning were performed in a climate chamber at a temperature of 80°C and humidity of 95%. The seasoning period in the climate chamber was continued for 4 or 6 weeks, depending on the variant. The obtained compressive strength results of both epoxy adhesive compounds were analysed statistically. The mean compressive strength values of the tested compounds differed significantly for particular variants. In order to obtain a precise statistical analysis, the ANOVA was used, which allowed to compare the results with regard to the seasoning variants for a given epoxy adhesive compound. The analysis showed a lack of similarity between variants of seasonings of epoxy adhesive compound consisting of Epidian 5 epoxy resin and PAC curing agent. In the case of compound made up of Epidian 5 epoxy resin and Z-1 curing agent, the ANOVA showed a very high similarity of compressive strength with respect to the compared seasoning variants.
Keywords: ....

Streszczenie: Celem niniejszej pracy jest analiza statystyczna wytrzymałości na ściskanie dwóch epoksydowych mas regeneracyjnych o różnych właściwościach, które poddano sezonowaniu w tych samych warunkach. Pierwsza epoksydowa masa klejowa składała się z żywicy epoksydowej Epidian 5 zmieszanej w stosunku masowym 100:80 z utwardzaczem PAC, natomiast druga masa składała się z żywicy epoksydowej Epidian 5 zmieszanej w stosunku masowym 100:10 z utwardzaczem Z-1. Epoksydowe masy klejowe poddano sezonowaniu. Pierwszy wariant sezonowania prowadzono w warunkach normalnych, w temperaturze 29°C ± 1°C i przy wilgotności 20% ± 1%, przez 1 tydzień. Pozostałe warianty sezonowania przeprowadzono w komorze klimatycznej w temperaturze 80°C i wilgotności 95%. Czas sezonowania w komorze klimatycznej kontynuowano w zależności od wariantu przez 4 lub 6 tygodni. Uzyskane wyniki wytrzymałości na ściskanie obu epoksydowych mas klejących poddano analizie statystycznej. Średnie wartości wytrzymałości na ściskanie badanych mieszanek różniły się istotnie dla poszczególnych wariantów. W celu uzyskania precyzyjnej analizy statystycznej zastosowano metodę ANOVA, która pozwoliła na porównanie wyników w zakresie wariantów sezonowania dla danej epoksydowej masy klejowej. Analiza wykazała brak podobieństwa pomiędzy wariantami sezonowania epoksydowej masy klejącej składającej się z żywicy epoksydowej Epidian 5 oraz utwardzacza PAC. W przypadku mieszanki złożonej z żywicy epoksydowej Epidian 5 i utwardzacza Z-1 analiza ANOVA wykazała bardzo duże podobieństwo wytrzymałości na ściskanie w odniesieniu do porównywanych wariantów sezonowania.
Słowa kluczowe: ....

Introduction

The long-term operation of machines used in the technical field is associated with a wear and tear process of parts used in a given machine, this results in disruptions of the proper operation of the machine. Parts such as gears, crankshafts or housings are often exposed to various types of material damage. Examples of such damage are cracks, scratches or losses of material.

There are several types of wear and tear processes, they include [4]:

- mechanical-chemical wear,
- abrasive wear,
- corrosive-mechanical wear,
- erosive wear,
- adhesive wear,
- fatigue wear.

Typically, defective parts are replaced with new ones. A part can be purchased or manufactured by designing an appropriate technological process. However, this involves costs and machine downtime. A defective part can also be reconditioned. This process reduces operating costs and shortens downtime of the machine containing the faulty part [15].

The reconditioning process is a universal method and is widely used in technology. Its purpose is to restore the functional properties of a given part of a technological machine. However, in this process it is very important to determine the degree of wear and tear of a given part. The damage may be too serious to undergo the reconditioning process. This applies mainly to cracks and material loss. Therefore, before proceeding with regeneration of a part, it is necessary to check whether it is possible to restore the original properties of the damaged part as a result of the process.

In the regeneration process, various methods are used to ensure the reconstruction and restoration of the component to a renewed operation. The methods used in the generation process include [13]:

- welding,
- surfacing,
- spraying,
- application of regenerative coatings,
- application of regeneration masses.

Parts which undergo wear and tear process are usually reconditioned by welding, surfacing or spraying. During the regeneration process using the above methods, a material with similar structure is used. When applying regenerative coatings, the phenomenon of electrolysis is used, which changes the chemical structure of the surface layer of the material of the damaged part, the process is due to the diffusion of the elements which are regenerating and strengthening the structure of the material. The most commonly used regenerative coatings include iron coatings, chromium coatings and iron-nickel coatings [2, 3].

Currently, regenerative masses based on polymeric materials are more widely used in the process of regeneration of parts. The popularity of regenerative masses is caused by their good mechanical properties. Moreover, they are easy to use which means that operations of the technological process, such as preparation or application of polymer regeneration masses, do not require special equipment as it happens in, for example, welding. Other advantages of this method are significantly lower repair costs and shorter period of machine downtime.

Usually regenerative masses consist of 2 components, an adhesive substance and a curing agent. To create an adhesive compound, both components need to be mixed in the appropriate mass ratio, recommended by the manufacturer. Improper mixing of these ingredients can reduce the strength of the compound. Curing and seasoning processes which can be carried out in different climatic conditions are also important for strength of the regenerative masses. In some cases, the impact of higher temperature on cured regenerative masses may accelerate the aging process, which leads to a significant reduction in the strength of the regenerative mass [11].

This paper, focuses on strength studies of epoxy adhesive compounds, which can be used as regenerative masses. Compressive strength tests have been performed on the epoxy adhesive compounds tested. The type of strength test was chosen because of the forces that mainly affect the regenerated areas in a worn-out part. The ANOVA comparing both adhesive compounds with respect to compressive strength was also performed. For the comparison the STATISTICA program was used.

Research methodology – description of the research preparation

Two epoxy adhesive compounds were used in the study as regenerative masses. The contents of these compounds is shown in Tab. 1. The epoxy adhesive compounds were based on Epidian 5 epoxy resin. This resin is characterised by low shrinkage during the curing process and is resistant to chemical agents, such as greases and oils. It also has excellent adhesion to most materials such as glass, metals and wood, making it suitable for a range of industrial applications [7].

Table 1. List of adhesive compounds

Compound No.	Contents		Stoichiometric ratio	Designation
	Resin	Curing agent		
1.	Epidian 5	PAC	100:80	E5/PAC/100:80
2.	Epidian 5	Z-1	100:10	E5/Z-1/100:10

The first adhesive compound was made of Epidian 5 epoxy resin mixed with PAC curing agent (E5/PAC/100:80). The structure of this compound was a homogeneous thick liquid which was dark yellow in colour. After being mixed with the epoxy resin, PAC curing agent required approximately 180 minutes to start the curing process. It is classified as a slow reacting curing agent. After 72 hours from the beginning of the curing process, the epoxy adhesive compound was pre-cured with PAC curing agent [16]. The curing process of the epoxy adhesive compound containing PAC curing agent was performed under conditions where the air humidity did not exceed 70% [17].

The second adhesive compound contained Epidian 5 epoxy resin mixed with Z-1 curing agent (E5/Z-1/100:10). The structure of this compound was a homogeneous liquid with a light yellow colour. When using Z-1 curing agent, the curing process started as soon as the curing agent was added to the epoxy resin. The time needed to pre-cure the epoxy adhesive compound with the applied Z-1 curing agent was 48 hours [9]. Z-1 curing agent is a chemical compound based on triethylenetetramine [17]. The excess of this curing agent in the epoxy adhesive compound causes a decrease of the physical properties of the created compound [8].

In case of both curing agents used in epoxy adhesive compounds, the period of full curing was 7 days. Comparing the properties of epoxy adhesive compounds, the ones containing PAC curing agent were characterized by higher elasticity, but had lower resistance to elevated temperature than epoxy adhesive compounds for which Z-1 curing agent was used [1, 10]. In the study, the epoxy adhesive compounds were prepared immediately before filling the moulds.

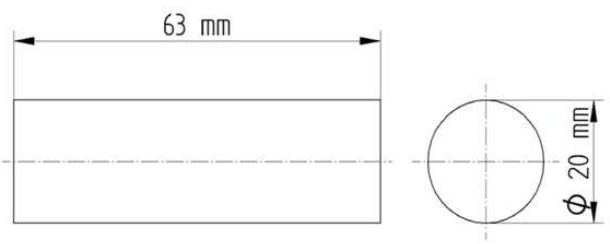

Fig. 1. Sample dimensions of epoxy adhesive compound

Samples of epoxy adhesive compounds were cylindrical in shape, their dimensions are shown in Fig. 1. Thirteen samples were made for each epoxy adhesive compound studied. During the preparation of the samples of the epoxy adhesive compounds the following conditions existed in the room : temperature 29°C ± 1°C, humidity 20% ± 1%.

During the curing process, the temperature and air humidity in the room were monitored. After 7 days, the cured samples of both epoxy adhesive compounds were subjected to 3 variants of seasoning.

The first seasoning variant involved seasoning of the samples for 7 days in room conditions at an ambient temperature of 29°C ± 1°C and humidity of 20% ± 1%. For the second and third variants of sample seasoning, the Espec SH 661 climate chamber was used, which allowed for adjusting seasoning conditions, such as temperature and humidity. For both variants seasoned in the climatic chamber, identical seasoning conditions were applied. In the climatic chamber, the following conditions were created : temperature 80°C, humidity 95%. The temperature 80°C is the border temperature of resistance of this epoxy resin [18]. The difference between the seasoning variants was the period of curingthe samples in the climatic chamber. Table 2 summarises the adhesive compounds, the number of prepared samples and the seasoning parameters.

After seasoning the samples according to individual variants, the samples were subjected to compressive strength tests according to PN-EN ISO 604:2006 [5] with the following parameters: initial force equal to 10 N, test speed 10 mm/min. The strength tests were performed using Zwick/Roel Z150 testing machine. The tested specimens were fixed in a compressive test device, which consists of two parallel flat plates, between which the samples were fixed. Fig. 2 shows the way of fixing the specimens in the compressive test device.

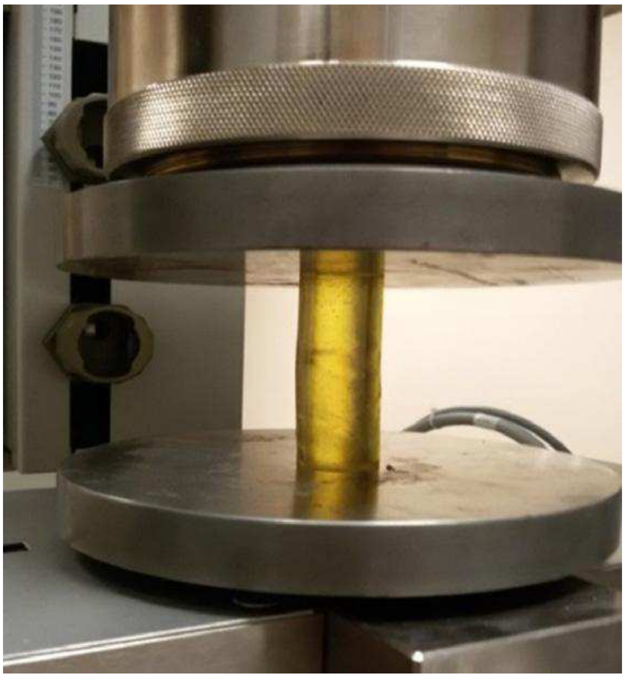

Fig. 2. System of the forced feed of abrasive compound

During the compression test, some slight buckling of the samples was observed. The obtained compressive strength results were then subjected to statistical analysis to compare the strength of the two adhesive compounds with regard to different seasoning options.

The statistical analysis used in this paper was ANOVA. This analysis allows for comparison of multiple mean results of tests coming from different groups, in this case seasoning variants [16,17]. The analysis of variance was applied only within a given adhesive compound,

Table 2: Summary of sample seasoning options

		Sampling seasoning variants		
		Variant 1	**Variant 2**	**Variant 3**
Number of cylindrical forms	**E5/PAC/100:80**	5	5	3
	E5/Z-1/100:10	5	5	3
Type of curing		Without climatic chamber	Climatic chamber	Climatic chamber
Seasoning period [week]		1	4	6
Temperature [°C]		29°C	80°C	80°C
Humidity		20%	95%	95%

comparing the obtained compressive strength results with regard to the applied seasoning variant.

Results of studies

Table 3 shows the compressive strength results of epoxy adhesive compounds. The obtained results were subjected to a descriptive statistical analysis.

Analysing the obtained values by comparing the descriptive statistics in Tab. 3, it is possible to conclude that the mean compressive strength values in the different variants between the two adhesive compounds differ significantly. For the first variant this difference is 34%, for the second variant 57% and for the third variant 63%. These differences show a significant discrepancy between the compressive strength of the analysed adhesive compounds. Similar differences are also visible in the case of the median, which is the middle value of the set. Due to significant differences of average strength results of both adhesive compounds with regard to one seasoning variant, no further statistical tests were performed to check the convergence of mean results of adhesive compounds with respect to the seasoning variant.

On the basis of the data from descriptive statistics, it is also possible to compare

the results of compressive strength of a given adhesive compound with respect to the seasoning variant. The average values of compressive strength of Epidian 5 epoxy resin with PAC curing agent clearly decrease with a longer time of curing the compound. The difference between the average strength results of variant 1 and variant 3 is 42%. In the case of compound with Z-1 curing agent, the mean result of compressive strength of the seasoning variants is similar.

The ANOVA was used for further comparative analysis of the obtained compressive strength values of the tested specimens. Equality of the obtained mean strength results was assumed as hypothesis H0. The test probability level was $\alpha=0.05$. One-way ANOVA was chosen as the type of analysis of variance.

Fig. 3 shows a comparative graph of the ANOVA of 3 seasoning variants of the E5/PAC/100:80 adhesive compound. The confidence interval of this test was $p=0.0003$. This means that the H0 hypothesis should be rejected and the alternative hypothesis accepted [12, 14]. On the basis of the result of this statistical analysis, a significant difference can be seen between the results of the seasoning variants of the adhesive compound of Epidian 5 epoxy resin with PAC curing agent.

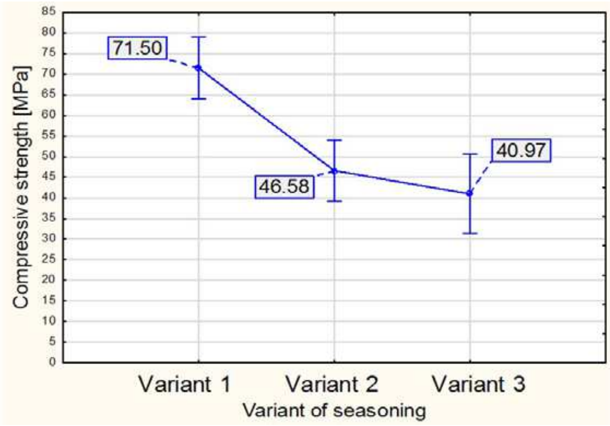

Fig. 3. The ANOVA of 3 seasoning variants of the E5/PAC/100:80

Fig. 4 presents the comparative ANOVA of 3 seasoning variants of E5/Z-1/100:10 adhesive compound. The presented graph shows the similarity of the average compressive strength results of this adhesive compound with regard to the seasoning variants. The confidence level of the test obtained by performing the analysis of variance was $p=0.999$. This means that the condition $\alpha<p$ was met. The confidence level of the test is close to 100%, which indicates a very high probability that the compressive strength of the adhesive compound of Epidian 5 epoxy resin with Z-1 curing agent for 3 different seasoning conditions is the same [11].

Table 3. Parameters of descriptive statistics for compounds compressive strength

Compound	Seasoning Variant	Number of samples	Mean [MPa]	Median [MPa]	Variance [MPa]	Standard deviation [MPa]	Skewness
E5/PAC	Variant 1	5	71.50	71.45	8.52	2.92	-1.03
	Variant 2	5	46.58	46.10	60.57	7.78	1.36
	Variant 3	3	40.97	35.40	140.96	11.87	1.65
E5/Z-1	Variant 1	5	107.54	106.00	181.06	13.46	0.32
	Variant 2	5	107.40	107.00	2.80	1.67	-0.51
	Variant 3	3	111.00	111.00	4.00	2.00	0

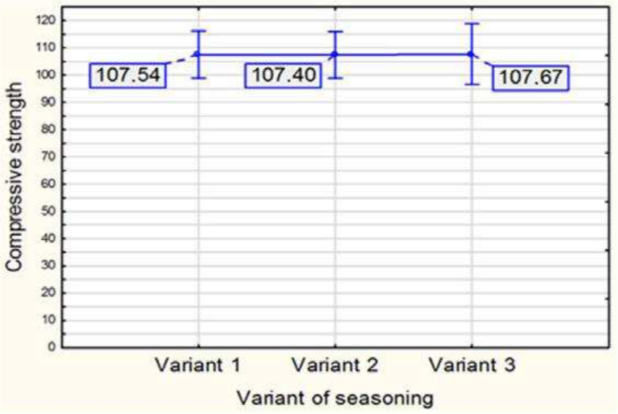

Fig. 4. The ANOVA of 3 seasoning variants of the E5/Z-1/100:10

Conclusion

After the test and statistical analysis, the following conclusions can be drawn:

- The type of curing agent and its amount in the adhesive compound, with different seasoning variants, clearly affects the value of compressive strength. In the case of the comparison of E5/Z-1/100:10 and E5/PAC/100:80 epoxy adhesive compounds with regard to the first variant, the difference was 34%, with regard to the second variant – 57%, and with regard to the third variant – 63%,
- Similar values of compressive strength of epoxy adhesive compounds in relation to different seasoning variants were obtained for E5/Z-1/100:10 compound. This means that the conditions of high temperature and humidity acting on the cured E5/Z-1/100-10 compound do not result in strength decrease. This compound can be used as a regeneration mass in machine parts,
- The change of temperature and humidity with regard to the conditions that prevailed during seasoning, negatively influenced E5/PAC/100:80 epoxy adhesive compound. Increasing the seasoning period in the climatic chamber by 2 weeks also contributed to a decrease in the compressive strength of the epoxy adhesive compound containing PAC curing agent. By increasing the seasoning period by 2 weeks, under the same conditions, a decrease of 19.08% in the compressive strength of variant 3 was observed in relation to variant 2 seasoning of the samples. This means that E5/PAC/100:80 epoxy adhesive compound is not a suitable regenerative compound to restore the usefulness of a broken machine part. This result also supports the finding that PAC curing agent is less heat resistant than Z-1 curing agent [8].

References

[1] Czub P., Bończa-Tomaszewski Z., Penczek P., Pielichowski J. 2002. Chemia i technologia żywic epoksydowych.Warszawa: Wydawnictwo WNT.

[2] Konarowska D. 2004. Powłoki ochronne. Radom: Wydawnictwo Politechniki Radomskiej.

[3] Mistur L. 1971. Spawanie gazowe i elektryczne. Warszawa: Państwowe Wydawnictwa Szkolnictwa Zawodowego,

[4] Mychajło P, Kindrachuk M. 2017. Tribologia. Lublin: Wydawnictwo Politechniki Lubelskiej.

[5] PN-EN ISO 604:2006 – Tworzywa sztuczne – Oznaczanie właściwości przy ściskaniu.

[6] Rabiej M. 2018. Analizy statystyczne z programami Statistica i Excel. Gliwice: Wydawnictwo Helion.

[7] Rudawska A. 2020. "The Impact of the Acidic Environment on the Mechanical Properties of Epoxy Compounds in Different Conditions". Polymers 12(12): 1–19.

[8] Rudawska A., Celejewski F., Miturska I., Kowalska B. 2018. „Wpływ warunków utwardzania i sezonowania na wytrzymałość połączeń klejowych doczołowych". Technologia i Automatyzacja Montażu 4: 48–52.

[9] Rudawska A., Głogowska K. 2014. „Analiza porównawcza wytrzymałości połączeń klejowych wykonanych przy użyciu klejów epoksydowych". Przetwórstwo Tworzyw 4: 320–325.

[10] Rudawska A., Semeniuk M. 2014. „Wpływ rodzaju żywicy epoksydowej i utwardzacza na wytrzymałość połączeń klejowych blach stalowych". Technologia i Automatyzacja Montażu 4: 65–68.

[11] Rudawska A., Wierzchowski A., Müller M., Petru J, Náprstková N. 2017. "The properties of regenerative polymer mass". Advances in Science and Technology Research Journal 11(3): 130–138.

[12] Seltman H. 2018. Experimental Design and Analysis. Pittsburg: Wydawnictwo Carnegie Mellon University.

[13] Tyra A., Świerczyński S., Poreda A. 1989. Regeneracja części maszyn i urządzeń. Radom: Wydawnictwo MCNEMT.

[14] Wasilewska E. 2015. Statystyka matematyczna w praktyce. Warszawa: Wydawnictwo Difin S.A.

[15] Wojdak J., Sędłak P., Wanke P., Stawicki T. 2009. „Analiza rozwoju metod regeneracji części maszyn w aspekcie przemian gospodarczych". Inżynieria Rolnicza 1: 347–353.

[16] https://www.zywicesarzyna.pl/epidian-5-utwardacz-pac/ (21.04.2021).

[17] https://www.zywicesarzyna.pl/epidian-5-utwardzacz-z1/ (21.04.2021).

[18] https://www.zywicesarzyna.pl/produkty/epidian-5/ (21.04.2021).

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