

Optimization of Carbon Fiber Bonding Parameters

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Abstract: Application of carbon fiber reinforced polymer (CFRP) is growing in all industry branches, such as aeronautics, automotive, military, space industries, etc. An excellent ratio of weight to strength, high impact toughness and corrosion resistance, as well as possibility of bonding by gluing make CFRP suitable for various applications in the mentioned industries. Extensive application of CFRP requires continuous research into their improvements and properties, in order to exploit their potentials to the maximum. In the conducted experiment, there were various combinations of operating parameters varied for the purpose of analyzing their individual and joint influence on the properties of glued joints. By optimizing the input variables of the experiment (type of glue, mass and curing temperature), which have a significant role in changing the characteristics of the bond, it is possible to define the best combination that can deliver the best tensile-shear properties. Tensile-shear strength is selected as an output variable of the experiment and as desired property for optimization. The implementation of the central composite design and the statistical analysis of data result in mathematical models on the basis of which it is possible to influence the observed and desired properties of such bonds in a short time, i.e. it is possible to obtain concrete values of individual parameters that will ensure the best tensile-shear strength.

Keywords: bonding; carbon fiber; optimization; tensile-shear strength

1 INTRODUCTION

A composite is a material that consists of at least two components that have different properties and a clear boundary between them. Due to their exceptional mechanical properties, such materials are widely used in various industrial branches [1-5]. Even better properties can be obtained by optimizing the parameters of their use [6]. Composites are produced by artificial joining of materials, and the aim is to produce a new material, which retains the best properties of all components [7]. The need for such materials is increasing with the progress and development of numerous industrial branches. If manufacturers of certain components want to be competitive, they must follow trends and constantly improve the characteristics of their products. Such an example is the use of carbon fibers, whose use for commercial purposes began 50 years ago. But in the face of constant new market demands, already existing properties are being developed and improved. Graphite, i.e. carbon composites belong to composites with a polymer matrix (PMC) reinforced with carbon fibers (CFRPC - Carbon Fiber Reinforced Polymer Composite). The carbon fibers act as reinforcement, but they are useless without the matrix. Carbon is strong, hard and light, used when light-weight material of high performance is required. Composites with carbon fibers in a polymer matrix deliver high specific strength even at high temperatures. Carbon woven fabrics are used in the automotive industry to make car body by connecting laminates into prepregs [8-10]. Exterior car parts, such as bumpers, front and rear diffusers, engine bonnet cover, doors, mirrors, spoilers, roof and even rims are produced from carbon. As carbon is considered a premium material, it is often used in production of accessories for vehicle interiors. Being previously produced of polymers, battens, steering wheels, seat shells, cages for racing cars and many other parts are now made out of carbon [11, 12]. Carbon is used because of its aesthetics and contribution to the reduction of mass. It is increasingly used in electric cars, which aim to increase environmental protection; however, it finds application in other branches such as construction

[13]. Equally, in addition to the previously mentioned examples, these materials are widely used in the production of orthopedic aids. Most of such aids are made by individual production and for specific cases, i.e. they are precisely made for a specific application and a specific individual. Because of these manufacturing characteristics, it can be assumed that the price itself is quite expensive. On the trail of this, the topic of these experimental researches was elaborated. Namely, if the orthopedic aid breaks, the most common case is the manufacture of a new one, which again requires considerable material resources. However, the desire of this work was to see how much this case can be avoided by applying glue, i.e. how practical is the application of glue with which the orthopedic aid would return to its original position and once again be functional. Research of this type is justified because the application of glue already has its application in certain cases [14, 15]. However, what is particularly specific to the theses presented here has not yet been defined. That is, it is not defined how the commercially available glues observed here behave depending on certain input parameters that are defined as input variables of the planned experiment. With this aim, experimental tests were carried out, with the help of which it was possible to optimize all observed parameters in order to obtain the best possible output properties, i.e. so that prostheses made of the observed materials could be safely and functionally applied again.

2 EXPERIMENTAL RESEARCH

This experiment aims to examine the influence of selected parameters on the strength of bond obtained by gluing of two carbon plates. The experiment plan comprises 22 test samples made of carbon plates bonded with two types of glue in different amounts (glue masses) and cured at different temperatures. Prepared test samples were tested for their tensile-shear strength by a tensile testing machine. In the experiment, there were two types of 2-component glues used, one is LOCTITE EA 9466 [16] and the other is BISON POWER GLUE [17]. The test plates have been made of carbon cut into dimensions of $100 \times 25 \times 1,5$ mm. The carbon used was composed of

220 gram carbon fiber and epoxy resin. Before gluing, the plates are sanded, degreased and cleaned, all with the aim of achieving surface of the best adhesion properties. Two test plates were glued into one test sample, in a way to create a 9 mm wide overlap bond (Fig. 1). Bonded test plates were cured in different conditions.



Figure 1 Schematic presentation of a test sample

2.1 Preliminary Experiment

The test samples have been made of carbon fibers labelled as 220 – 2 × 2 Twill weave - 3K Tow. The label explains that 1 m² of material weighs 220 g, it is woven bidirectionally, which means that it has good tensile properties, both longitudinally and vertically. There was a total of 0,15 m² of carbon used in the experiment, which refers to 50 carbon plates cut into dimensions of 100 × 25 mm, 2 carbon plates of dimension 200 × 25 mm, and the remains. The price of a square meter of this carbon is approximately \$110.

The material has been cut into the mentioned size by using self-glue tape. The tape was used to prevent the carbon fibers from unraveling during cutting and to keep the required width of 25 mm. Special scissors used for cutting of carbon fibers were very sharp and have a shaped handle in order to provide leverage to the operator to cut the carbon as easily as possible without stretching the material. Cut carbon plates were then air-dried for about 24 hours. After drying, the plates have been inspected and the sharp edges were sanded, thus giving them their final shape. The surface to be glued must be prepared according to the rules in order to obtain an optimal bond. The surface treatment was carried out according to the instructions of the glue manufacturer, which states that all samples should be clean and degreased for the best effect of the glue. It is necessary to remove impurities such as paint, oil, dust and other impurities from the surface. All surfaces were treated with LOCTITE 7063 degreaser. The agent was applied to the stripe to be bonded, then wiped with a dry cloth. Plates were left for a few minutes to allow the surface to dry. After that, the surface was further sanded to enable better adhesion. The surfaces were cleaned and ready for bonding by glue.

There were two 2-component glues used in this experiment to bond the test plates, one was LOCTITE EA 9466, and the other was BISON POWER Glue. LOCTITE EA 9466 is a tough 2-component epoxy glue that cures for about 72 hours at room temperature, but the curing process can be accelerated by raising the temperature. Long curing time is a great advantage when the parts to be bonded have to be precisely adjusted and fixed so that they do not move. After application, the glue cures at room temperature and forms a solid, whitish bond line of high peel resistance and tensile-shear strength. Cured bonded material is resistant to different chemicals and solvents, and acts as an excellent electrical insulator. LOCTITE EA 946 provides high

strength for bonds of polymers, metals and ceramics. It is usually used for general industrial production when extended time of application is required because of alignment of parts during assembly. Resin and hardener are mixed in the ratio of 2:1. BISON POWER GLUE is a universal 2-component polyurethane glue. It bonds metal, wood, stone, marble, ceramics, rubber and glass. It is yellowish in color and quite viscous, which prevents dripping. Resin and hardener are mixed in the ratio of 2:1. The glue is applied to a clean surface and it cures for 15 minutes. The service temperature of cured glue is from –30 °C to 100 °C. The prepared plates were glued into the test samples.

Experiment parameters applied on bonded plates were: 2 types of glues, 5 different curing temperatures and 3 different glue masses. The amount of glue was measured by a scale with a precision of 0,01 g, and glues were applied with provided tool. The test samples were cured in an oven. When gluing the test samples, it is important to note that the thickness of the connection was not analyzed, but that parameter was observed in the form of the mass of the added glue. In addition, it is important to note that no additional force was applied to the test samples during gluing. The application of additional force during gluing was avoided in this case so that it would not cause leakage of glue outside the area of the test samples, which would drastically affect the observed factor in the form of the mass of added glue.

The Shimadzu AGS-X 10 kN tensile testing machine was used in this experiment to test the tensile-shear strength of the bonded plates. The device holds a test piece attached from the lower side, and the upper device jaw pulls the upper part of a test piece until it breaks. In doing so, the device records applied force during the time of elongation, as well as the force reached at the moment of breaking the bond. The recorded force should not deviate from the actual applied force by more than 1%. The main characteristics of the tensile testing machine are provision of a constant load and ability to maintain it. The test samples are placed in the machine jaws and pulled until they brake. The test speed was 2 mm/min. All samples broke after the test in the same way, i.e. the fracture of the test tubes occurred in the very zone of gluing the test plates. The TRAPEZIUM X software records the results and processes them into a diagram of tensile strength elongation. Fig. 2 shows the test sample after breaking in the tensile testing machine.



Figure 2 Test sample after being tested in a tensile testing machine

2.2 Experimental Testing and Analysis of Obtained Results

The experiment was carried out with two different types of glues, i.e. glues. The experiment objective was to define the optimal parameters for the strength of carbon plates bonded by glue. Coded values of experiment factors are presented in the Tab. 1.

Table 1 Coded values for the experiment design

Coded values	Factor 1 - Curing temperature / °C (numerical)	Factor 2 - Glue mass / g (numerical)	Factor 3 - Glue type (categorical)	
-1,4	22	0,2	Glue 1	Glue 2
-1	32	0,2		
0	56	0,3		
1	80	0,3		
1,4	90	0,4		

All experiment runs and results obtained by measuring the tensile-shear strength are presented in Tab. 2. These results refer to the mean value of three repeated measurements for each experimental sample.

Table 2 Results of measured tensile-shear strength

Experimental sample	Experiment run	Factor 1	Factor 2	Factor 3	Tensile-shear strength / MPa
		Curing temperature / °C	Glue mass / g	Glue type	
1	11	32	0,2	Glue 1	2,9
2	18	80	0,2	Glue 1	1,9
3	13	32	0,3	Glue 1	1,9
4	7	80	0,3	Glue 1	2,1
5	19	22	0,3	Glue 1	3,4
6	8	90	0,3	Glue 1	2,6
7	9	56	0,2	Glue 1	3,1
8	16	56	0,4	Glue 1	2,5
9	22	56	0,3	Glue 1	1,4
10	12	56	0,3	Glue 1	1,6
11	21	56	0,3	Glue 1	2,6
12	17	32	0,2	Glue 2	4,6
13	10	80	0,2	Glue 2	1,2
14	2	32	0,3	Glue 2	4,5
15	1	80	0,3	Glue 2	3,4
16	4	22	0,3	Glue 2	3,6
17	3	90	0,3	Glue 2	1,6
18	5	56	0,2	Glue 2	5,0
19	15	56	0,4	Glue 2	5,6
20	6	56	0,3	Glue 2	1,6
21	20	56	0,3	Glue 2	4,3
22	14	56	0,3	Glue 2	1,9

2.3 Overview of Results Referring to the Measurement of Tensile-Shear Strength

Experiment results were obtained by calculating the arithmetic mean of three repeated measurements. The principle of randomization was applied in the experiment, so the samples were tested in a random order, as presented in Tab. 2. Analysis of the obtained results shows that the minimum response value (tensile-shear strength) is 1,2 MPa, while the maximum one is 5,6 MPa.

Table 3 Simulation of four models in the Design-Expert software-tensile-shear strength

Model	<i>p</i> value for the model	<i>p</i> value for the deviation from the model	Adjusted coefficient of determination	Predicted coefficient of determination
Linear	0,0848	0,6699	0,2058	-0,0303
Linear with 2FI (two factor interaction)	0,0239	0,9430	0,5157	0,3751
Quadratic	0,4169	0,9538	0,5118	0,2989
Cubic	0,9629	0,6593	0,2183	-2,1113

The arithmetic mean of the response in this case is 2,73 MPa. Referring to the results (Tab. 2) obtained for the tensile-shear strength, the linear model with second-order

interaction 2FI proved to be the best (after testing the linear model, the linear model with second-order interaction 2FI, the quadratic and the cubic model; Tab. 3. In addition to the coefficient of determination, simulation of the models have been done in relation to the *p* value for each model (mean square deviation), *p* value for deviation from the model, and adjusted and predicted coefficients of determination.

Tab. 4 presents a report obtained from the Design-Expert software for the selected model. The selected model was used to describe the dependence of tensile-shear strength on the input variables applied in the experiment.

Table 4 Analysis of variance of the regression model for tensile-shear strength

Model	Sum of squares	Number of degrees of freedom	Mean square deviation	Value of <i>F</i> -test	<i>p</i> -value
Model	17,44	6	2,91	4,37	0,0124
A-Curing temperature	2,45	1	2,45	3,68	0,0773
B-Glue mass	3,99	1	3,99	6,01	0,0291
C-Glue type	0,7624	1	0,7624	1,15	0,3036
AB	0,0994	1	0,0994	0,1495	0,7053
AC	0,4727	1	0,4727	0,7112	0,4143
BC	6,83	1	6,83	10,28	0,0069
Residual	8,64	13	0,6646		
Lack of fit	3,41	9	0,3793	0,2902	0,9430
Error	5,23	4	1,31		
Total	26,08	19			

The coefficient of determination R^2 is the share of the explained variability (deviation of the regression \hat{y} from the arithmetic mean) in the total variability (deviation of the actual y from the arithmetic mean), and when calculated as shown by expression (1), it is 0,6687. It is important to mention that bonding of carbon is extremely complex. Its fibrous structure and complex surface preparation can cause significant problems, so it is difficult to achieve a repeatable result. Therefore, applying a coefficient of determination of a slightly smaller value is understandable.

$$R^2 = \frac{SS_{\text{model}}}{SS_{\text{total}}} = \frac{SKO_{\text{model}}}{SKO_{\text{uk}}} = 1 - \frac{SS_{\text{residual}}}{SS_{\text{total}}} \quad (1)$$

Nevertheless, a higher value of the coefficient of determination does not necessarily mean that the regression model is good. If new members are added to the model, then R^2 will increase, regardless of whether the added member is statistically significant or not. Therefore, there is a possibility that models with a high coefficient of determination will poorly express the estimated new value or the arithmetic mean of the response. Since the value of R^2 shall increase by adding new independent variables to the model, an adjusted coefficient of determination R^2_{adj} is to be used in order to adjust to the number of new model members. Adjusted coefficient of determination R^2_{adj} is 0,5157. Predicted coefficient of determination R^2_{pred} is 0,3751. The expression 2 describes regression model for the dependence of tensile-shear strength on the input variables. Values of the mentioned variables are coded for high factor levels as +1, and for low factor levels as -1. The expression 3 describes regression model with actual factor values, where the factor *C* (glue type) is Glue 1. The

expression 4 refers to the regression model with actual factor values, where the C is Glue 2.

$$\text{Tensile strength} = 2,61 - 0,4187 \times A + 0,6229 \times B + 0,2106 \times C + 0,1268 \times AB - 0,1841 \times AC + 0,8144 \times BC \quad (2)$$

$$\begin{aligned} \text{Tensile strength} = & 5,01459 - 0,032130 \times \text{Curing} \\ & \text{temperature} - 6,88490 \times \text{Glue mass} + 0,074556 \times \\ & \times \text{Curing temperature} \times \text{Glue mass} \end{aligned} \quad (3)$$

$$\begin{aligned} \text{Tensile strength} = & -0,617317 - 0,047443 \times \text{Curing} \\ & \text{temperature} + 16,15084 \times \text{Glue mass} + 0,074556 \times \\ & \times \text{Curing temperature} \times \text{Glue mass} \end{aligned} \quad (4)$$

Contribution of the interacting member Curing temperature · Glue mass results from their synergic effect. The experiment shows that the glue mass significantly affects the bonding characteristics, and the same statement can be applied to the temperature of curing, if it is studied as a separate factor. However, when changing the mass of glue (e.g. increasing its quantity), it is to expect that the curing temperature shall be corrected in order to enable the best characteristics of the observed model. Glue as a viscous element will not exhibit the same characteristics at all temperature values [12-14].

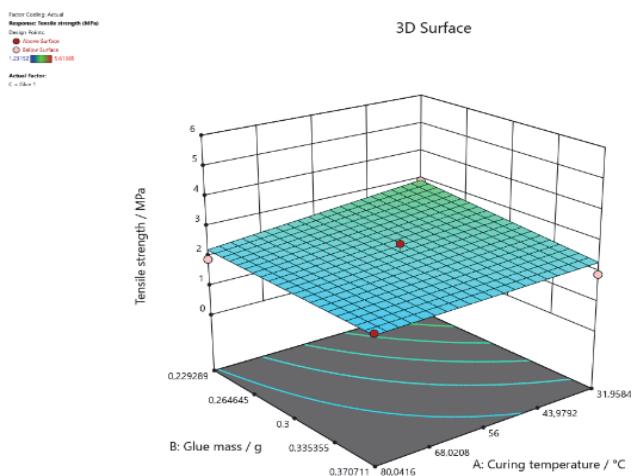


Figure 3 Response surface for regression model of dependence of tensile-shear strength for Glue 1

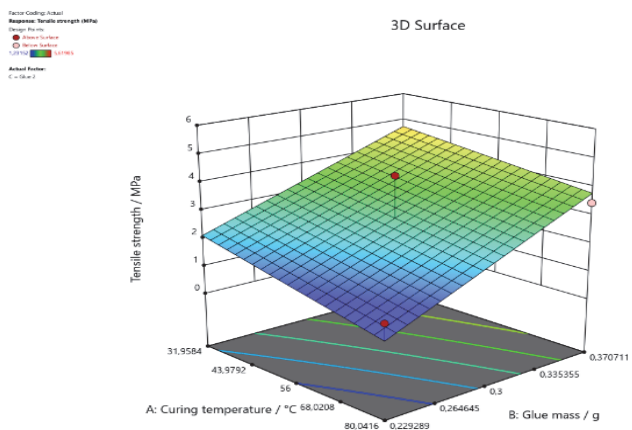


Figure 4 Response surface for regression model of dependence of tensile-shear strength for Glue 2

Fig. 3 and Fig. 4 give graphic presentations of the models. The figures below show how the tensile-shear strength behaves in dependence on the change of curing temperature and glue mass. Fig. 4 presents response surface for the regression model of the dependence of tensile-shear strength for Glue 1, while Fig. 5 presents response surface for the regression model of the dependence of tensile-shear strength for Glue 2.

2.4 Checking of Regression Model Adequacy

Model adequacy checking is an important part of the data analysis procedure in experiment design. It is necessary to test the estimated regression model in order to determine with certainty that the specified model is a high-quality approximation of the real system or process, and to check, i.e. to prove that all assumptions are valid when using the least squares method. When checking the adequacy of the regression model, one of the most important assumptions is that the errors in the model (residuals) should be independent and distributed normally, with expected zero and variance σ^2 . Fig. 6 presents the diagram used for checking of the model adequacy.

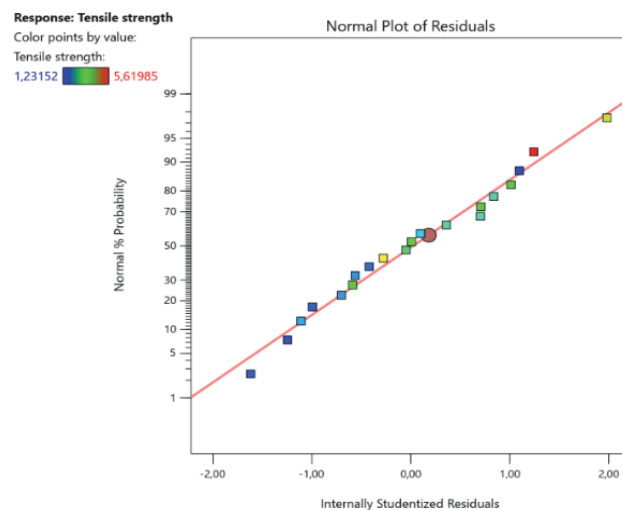


Figure 5 Checking the assumption on normal distribution - tensile-shear strength

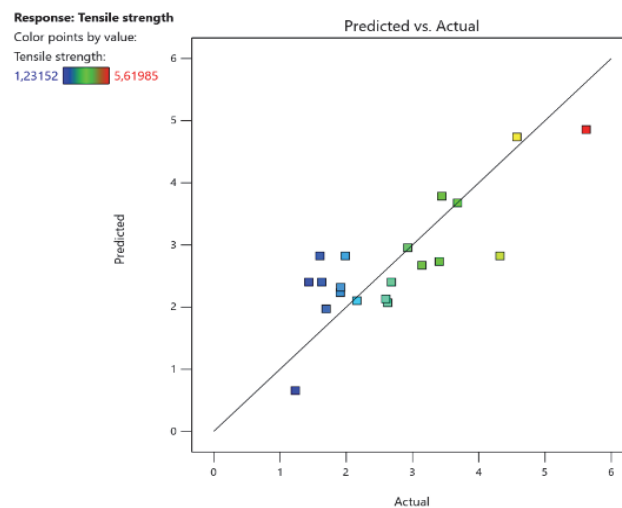


Figure 6 Actual and predicted value - tensile-shear strength

Fig. 5 suggests normal distribution of residuals (i.e. internally studentized residuals - transformed or scaled residuals), since there were no significant deviations from the line. Fig. 6 compares actually measured and predicted values by the model.

Following the above-presented experiment results, it was possible to optimize the input parameters in order to get desired output value (tensile-shear strength) for each of the studied glues. Tab. 5 contains data on limitations set for each of the input parameters, as well as the target value for the tensile-shear strength. Tab. 6 presents optimal values of input parameters needed to achieve the mentioned goal, i.e. tensile-shear strength.

Optimal solution, i.e. optimal values of the input parameters for achieving the target tensile-shear strength are shown by Fig. 7.

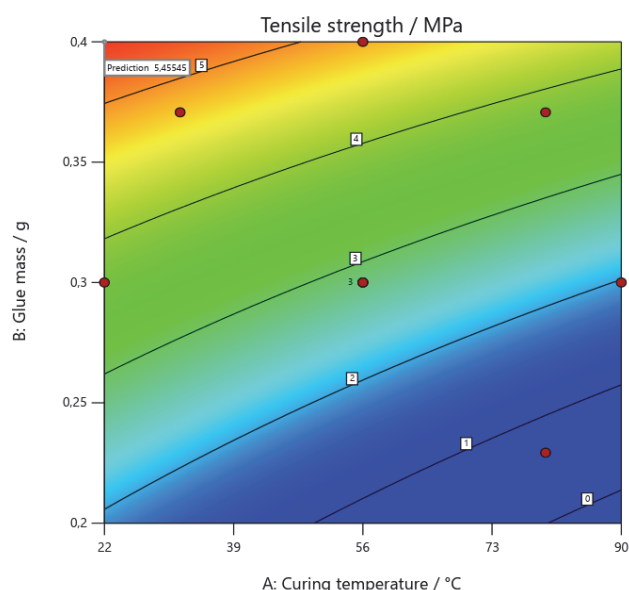


Figure 7 Optimal values of input parameters for achievement of maximum value of tensile-shear strength

Table 5 Limitations for optimization of parameters

Input parameter	Goal	Lower Limit	Upper Limit
A:Curing temperature / °C	in range	22	90
B:Glue mass / g	in range	0,2	0,4
C:Glue type	in range	Glue 1	Glue 2
Tensile-shear strength / MPa	maximized	1,23152	5,61985

Table 6 Optimal solution for achieving the maximum value of tensile-shear strength

Curing temperature / °C	Glue mass / g	Glue type	Tensile-shear strength / MPa
22	0,4	Glue 2	5,455

3 ANALYSIS OF RESULTS

Upon having analyzed the obtained experiment results, it is confirmed that each of the three observed input variables (curing temperature, glue mass and glue type) affects the response value, i.e. the tensile-shear strength. Different values of tensile-shear strength for all experiment runs are overviewed in Tab. 2, and presented in graphs of response surfaces for regression models of tensile-shear strength dependence on both observed glues. Fig. 4 shows that the highest value of tensile-shear strength can be achieved with Glue 1 applied in lower mass (0,23 grams)

and cured at lower temperature (32 °C). Accordingly, bonds made with Glue 1 require lower curing temperatures than the maximum set ones. It is assumed that the epoxy resin in the carbon would melt during curing at high temperatures, thus preventing penetration of glue into the material and weakening the bond itself. However, it is observed that bonds made with Glue 1 are not drastically decreasing their tensile-shear strength with the increase of curing temperature and the glue mass. On the contrary, when applied in less amount, the Glue 2, as shown in Fig. 4, drastically affects the reduction of achieved tensile-shear strength. Unlike the Glue 1, the increase of curing temperature for Glue 2 significantly affects the tensile-shear strength. Referring to the Glue 2, the highest values of tensile-shear strength can be achieved with applying larger amount of glue and curing it at a lower temperature. Further processing of data obtained in this experiment leads to determination of optimal input values that provide the required tensile-shear strength. If aiming at achievement of the maximum value of tensile-shear strength, and setting the upper and lower limits of the input parameters, the Glue 2 applied in 0,4 grams and cured at 22 °C will provide the best result, i.e. tensile-shear strength of 5.455 MPa.

4 CONCLUSION

Bonding of materials by gluing is widely used in industry mostly because of: small mass, the possibility of bonding various materials, the possibility of bonding most types of materials, bonding of materials of different thickness, bonding of large surfaces, achieving resistance to corrosion. There are also disadvantages of bonding, such as aging of glue, demanding surface preparation and low strength of bonds. It is because of all the advantages and disadvantages that one should be careful when choosing all the parameters of the procedure itself, starting from the compatible type of glue and ending with parameters such as the mass of addition and the drying temperature. This experiment is conducted with the aim to compare different parameters of gluing and curing and their influence on obtained tensile-shear strength. Based on the obtained results, it is concluded that the glue type, curing temperature and glue mass have significant effect on the bonding result. Moreover, it is important to emphasize that proper surface preparation also plays significant role in bonding of such materials. The complexity of carbon as a material should be also considered as a factor that affects quality preparation. It is important to obey all required and prescribed procedures when gluing carbon materials in order to achieve the desired outcome. In such undertakings, software for statistical data processing can be of help in analyzing specific glues and specific operating parameters in order to determine those that will deliver bonds of required properties. The results of the conducted experimental tests showed that by using such tools, optimal values of the observed properties can be obtained, with which the highest value of the output variable will be obtained. Specifically, in this case, the type of glue, mass and curing temperature were observed, but depending on specific cases, they can be changed arbitrarily. The generated optimal values gave the best values of the observed response, which in this case is the tensile-shear

strength. On the basis of the data thus obtained, experts in specialized companies can determine whether to approach this method of joining two materials made of carbon fibers. If the obtained properties correspond to the required ones, sometimes a certain construction or element can be repaired in this way in order to achieve savings by avoiding making a completely new part.

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