

Effect of annealing on tensile properties of carbon fiber reinforced PA 6 manufactured by fused deposition modeling

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Abstract: Development of fiber reinforced filaments for fused deposition modeling - FDM shifted this technology application towards load carrying applications. For polymer materials reinforced with carbon fibers, it is important to carry out annealing of printed products to improve the mechanical properties. In this paper ANOVA approach was used to evaluate the effect of temperature and time of the annealing treatment of PA 6 filament reinforced with short carbon fibers (PA 6 - CF). Results indicate that higher temperatures (between 110 °C – 170 °C) result in better effects on tensile properties while duration of the annealing effect was negligible in most cases. An increase of up to 16.7% in tensile strength and up to 35% in tensile modulus can be achieved with proper annealing parameters. In some cases, annealing results in a decrease in tensile strain at break up to 35%. The p-values for tensile strength, strain and modulus are 0.0038, 0.0054, 0.0168, respectively, which indicates that the selected model of the influence of annealing parameters is significant because the p-value must be less than 0.05. The highest improvement in tensile strength and modulus was observed at a temperature of 170 °C, but this temperature is close to the softening temperature of PA 6 - CF, which is approximately 180 °C before annealing, which risks deformation of products.

Keywords: annealing; carbon fiber; fused deposition modeling - FDM; poly(amide) 6 - PA6; tensile properties.

1. Introduction

Additive technology opens the possibilities of design and functionality due to its material adding working principle. Until recently its application was common in areas where mechanical performance of printed parts was secondary or irrelevant such as visualization models, everyday usage products, customized parts or art pieces. Development of fiber reinforced polymer filament in fused deposition modeling enhances the load carrying capabilities of printed products. A better understanding of the impact of process parameters on mechanical properties has led to an optimized printing of various polymer materials [1]. One of the main setbacks of printed products is bad interlayer adhesion caused by rapid temperature crystallization and cooling of the previously printed layers. [2] This effect is enhanced in fiber reinforced polymers since the conductivity of fibers results in heat dissipation through the layer in longitudinal direction, rather through the thickness of the layer and reaching the layer underneath. Thus, poor adhesion is result of insufficient heating of the previously laid material. [3] Annealing at

temperatures higher than glass transition temperature increases the interfacial bond contact which strengthens the material. [4] The influence of annealing has been research for commonly used printing materials such as PLA [5], ABS [3] and PETG [6]. In most cases annealing increased mechanical properties of materials and reinforcement made them more suitable for annealing. The influence of annealing parameters (pressure and temperature) [7] on the mechanical properties is most often observed in the papers, but in this paper the influence of temperature and time on the tensile properties will be observed. This selection of input parameters is because most 3D printers already have only time and temperature included in their program, and these two parameters can be easily checked and compared because it is possible to do it in ordinary ovens as well. Furthermore, this selection of parameters was chosen primarily due to the industrial application itself, because time is economically important for the product to reach the market as soon as possible, accordingly, it is necessary to determine whether time has an impact on the improvement of mechanical properties through post-processing heat

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treatment. Furthermore, it is important to mention that all tests were carried out on a *Makerbot* 3D printer, on which the manufacturer recommends annealing PA6 reinforced with carbon fibers for 5 hours at a temperature of 80 °C. [8] In previous literature, authors examined the effect of annealing at different temperatures, and some of the most commonly used materials in fused deposition modeling, such as PLA and PET-G, were tested at a temperature of 90 °C, or according to the material manufacturer, PA-CF at 80 °C. [9-14] However, preliminary tests have shown that such a temperature does not have much effect on the mechanical properties, primarily strength and modulus, and that it is necessary to conduct tests at higher temperatures.

2. Materials and Methods

The manufacturing method for test specimens was fused deposition modeling - FDM. Tensile test specimens were printed on a *Makerbot Method X* 3D printer using original *Makerbot* PA6-CF material. Material was dried for 24 hours in 60 °C in oven (manufacturer *Falc*) before printing of test specimens. Test specimens dimension was 75 × 5 × 2 mm in accordance with standard HRN EN ISO 527:2019 type 1BA for tensile testing of plastics (ISO 527 Plastics — Determination of tensile properties). Printing parameters are set up in *Makerbot Print* slicer and shown in Table 1. The choice of processing parameters in fused deposition modeling plays a major role in mechanical properties, but as the paper aimed to show the influence of post-processing heat treatment, all parameters were taken according to the default printer parameters for this material.

Table 1. Printing parameters

Printing parameter	Value
Layer height [mm]	0.2
Number of shells	3
Infill density	30%
Infill pattern	Rectilinear
Nozzle temperature [°C]	250
Chamber temperature [°C]	80
Nozzle diameter [mm]	0.4
Raft	none

Statistical analysis was used for designing the parameters of annealing by Design Expert software (manufacturer *Stat Ease*). A central composite design of experiment was used where tensile strength, tensile strain and modulus were set up as response. Temperature and time were chosen as variables for the annealing treatment as those are the most influential parameters according to the literature review. Glass transition temperature of polyamide is between 45 °C – 70 °C [9] and common annealing times are from 6 up to 8 hours. Variables with 2

and 3 hours for the duration of the treatment are chosen to see how a shorter period in a combination with higher temperatures compares to other conditions. Instead of melt transition temperature, Vicat softening temperature according to standard HRN EN ISO 306:2022 was used to make sure the shape and dimensions are constant during the annealing. Vicat testing method B120 served as the purpose of defining the top temperature limit of the material for the annealing experiment. The Vicat softening temperature was measured on a Frank device, with a Tinius Olsen displacement gauge with a measuring range from 0 mm to 1.2 mm in 0.01 mm increments. Vicat test shown in Figure 1. resulted in temperatures 179.23 +/-4.1. °C without annealing and 188.13 +/-0.4 °C after annealing according to manufacturers guidelines (80 °C, 5 hours). First, a preliminary annealing experiment was made where the limits of the input factors for the temperature was 50 °C to 110 °C and the annealing time is from 2 h to 8 h. At these temperatures, the properties did not change at all, and it was decided to increase the annealing temperatures to the temperature close to Vicat softening temperature and set up a new experiment with limits for temperature from 50 °C to 170 °C and time from 2 h to 8 h.

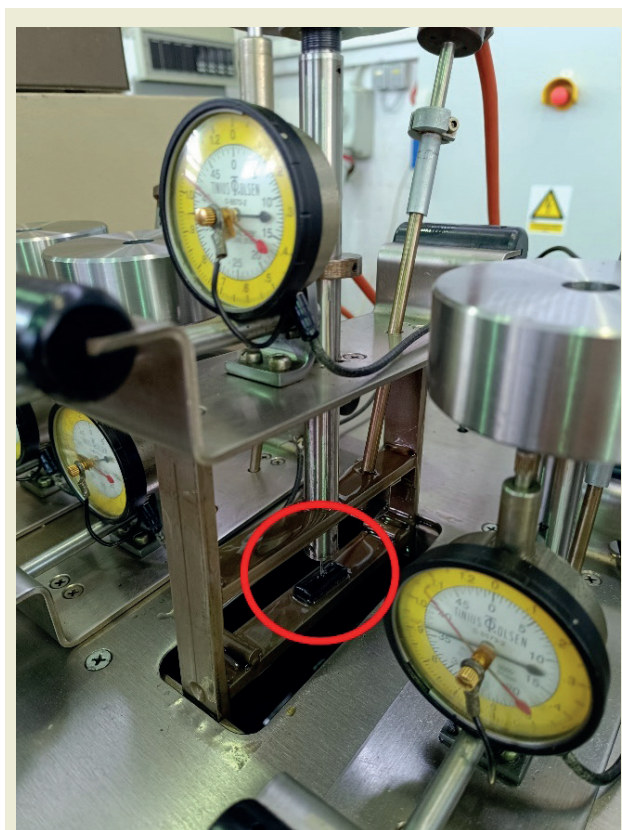


Figure 1. Vicat testing. The red circle in the figure shows the position of the test specimen during the Vicat softening temperature test. The test specimen is then lowered into the heated oil and when the test needle penetrates the specimen to a depth of 1 mm, the softening temperature value is recorded.

Test specimens for tensile properties were printed in plank face down position, so no support structures were

required. PVA glue was used to assure adhesion during printing, the residue of the glue was cleaned with a damp towel. Annealing treatment was done in *Falc* oven with closed air. The temperature range in the oven is up to a maximum of 300 °C, where air circulation can be turned on or off. The temperature can be adjusted in steps of 0.5 °C. Test specimens placed inside the oven chamber are shown in Figure 2.



Figure 2. Annealing of the tensile test specimens in the oven

A universal tensile testing machine was used for the test procedure (Shimadzu AGS-X max force 10 kN, manufacturer *Shimadzu*) with extensometer. The universal testing machine is in the measurement range from 10 N to 10 kN and is in the accuracy class of the measurement range 0.5 with an extensometer with a resolution of 0.003 mm, which meets all the requirements of the calibration standard HRN EN ISO 9513:2012. Testing was performed in accordance with standard HRN EN ISO 527:2019 at room temperature of 20 °C and 40% RH. The testing speed was 2 mm/min. Treated test specimens are compared to non-treated ones designated 0_1. The test was performed on 3 test specimens and

the mean value and standard deviation were then calculated.

3. Results and Discussions

Variables used in an annealing experiment with results are shown in Table 2. The effect of annealing procedure on the tensile properties of PA6-CF was tested according to the design of the experiment generated by software Design Expert, using response surface methodology. Data acquired by testing were processed by ANOVA (analysis of variance) linear modeling method with three center points.

Figure 3 shows tensile stress and strain across all variables. Untreated test specimen (designation 0_1) has 28.4 N/mm² tensile strength and the highest result was 34.1 N/mm² in case of 9_1 test specimen. The average increase of tensile strength at break was 7.5%. Modulus has increased on average 14.8% while strain at break was decreased in cases with higher temperature treatment (above 150 °C) and on average was 21.7%.

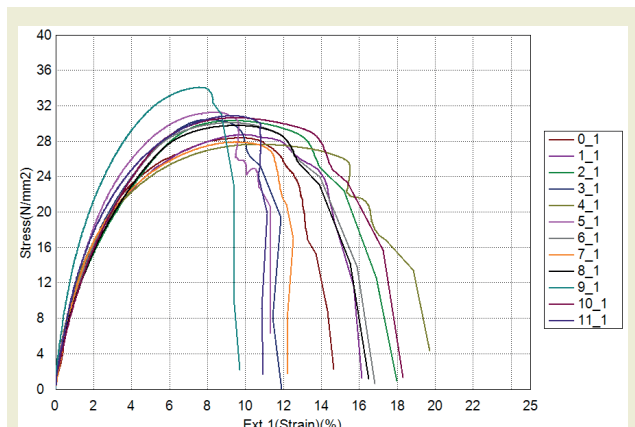


Figure 3. Diagram of tensile stress – strain for PA6-CF with different annealing factors. Each line represents the mean value obtained when measuring individual test specimens in each condition.

Table 2. Annealing factors (time and temperature) and results of the tensile testing

Run	Designation	Factor 1: Time t, h	Factor 2: Temperature ϑ , °C	Tensile strength σ_m [N/mm ²]	Tensile strain at break ϵ_b [mm/ mm]	Tensile modulus E [N/mm ²]
0	0_1	-	-	28.41 +/- 1.45	13.25 +/- 2.38	1060.82 +/- 545.32
1	1_1	5	50	28.78 +/- 1.59	14.71 +/- 2.82	1317.92 +/- 220.99
2	2_1	5	110	30.33 +/- 0.94	13.95 +/- 1.16	1007.68 +/- 170.73
3	3_1	7	150	30.74 +/- 0.63	10.13 +/- 1.66	1196.46 +/- 272.86
4	4_1	7	70	27.69 +/- 1.59	17.54 +/- 1.77	1270.22 +/- 184.78
5	5_1	3	150	31.53 +/- 1.58	11.24 +/- 0.68	1330.02 +/- 203.92
6	6_1	5	110	30.08 +/- 1.71	12.61 +/- 0.99	1096.69 +/- 249.68
7	7_1	3	70	27.92 +/- 0.97	11.9 +/- 0.77	1127.96 +/- 110.93
8	8_1	2	110	29.78 +/- 1.26	12.76 +/- 0.62	1106.7 +/- 124.4
9	9_1	5	170	34.1 +/- 1.82	8.32 +/- 0.78	1640.9 +/- 229.1
10	10_1	8	110	30.72 +/- 1.19	14.53 +/- 0.79	943.19 +/- 251.37
11	11_1	5	110	30.88 +/- 1.54	10.68 +/- 2.04	1283.07 +/- 75.29

Table 3. Analysis of variance – influence of annealing time and temperature on tensile strength

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	25.24	2	12.62	13.65	0.0038	significant
A-Time	0.0179	1	0.0179	0.0194	0.8932	
B-Temperature	25.23	1	25.23	27.29	0.0012	
Residual	6.47	7	0.9245			
Lack of Fit	6.16	6	1.03	3.24	0.4011	not significant
Pure Error	0.3162	1	0.3162			
Cor Total	31.72	9				

3.1. Results for the tensile strength

ANOVA analysis of variance indicates that the linear interaction model best fits the influence of annealing procedures on the tensile strength. Tensile strength statistical results can be seen in Table 3 and Table 4.

The model F-value (variation between sample means) of 13.65 implies that the model is significant. There is only a 0.38% chance that an F-value this large could occur due to noise. p-values less than 0.05 indicate that model terms are significant. In this case, factor B, temperature shows a major role in the values of tensile strength. Factor A, time of annealing seems to have a negligible influence on tensile strength. The lack of fit F-value of 3.24 implies that the lack of fit is not significant relative to the pure error. There is a 40.11% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good because it means that the model fits.

Statistical data (mean value, standard deviation, and R^2) about the model are given in Table 4. The coefficient of determination R^2 is a measure of deviation from the arithmetic mean which is explained by the model. The closer R^2 is to 1, the better the model follows the data, that is, the phenomenon is better explained.

Table 4. Summary statistics about the model for tensile strength

Std. Dev.	0.9615	R^2	0.7960
Mean	30.22	Adjusted R^2	0.7377
C.V. %	3.18	Predicted R^2	0.5303
		Adeq Precision	9.8138

Tensile strength for PA6-CF can be described by Equation (1) in actual parameters:

$$\sigma_m = 25.36965 + 0.022956 \cdot t + 0.043069 \cdot \vartheta \quad (1)$$

where: σ_m (N/mm²) – tensile strength, ϑ (°C) – annealing temperature, t (h) – annealing time.

The obtained equation for tensile strength is determined by the annealing temperature and time in the range, which is shown in table 1, i.e. for an annealing

temperature of 50 °C – 170 °C and for an annealing time of 2 h to 7 hours.

Figure 4 shows the tensile strength dependence on the annealing parameters. The diagram shows that the highest tensile strength is a result of a temperature of 150 °C in both cases of shorten or longest annealing time periods. The diagram shows the lowest values of tensile strength in blue, and the highest values in green lines. From Table 3 itself, and thus from the diagram in Figure 4, it can be seen that changing the annealing time has no effect on the tensile strength, but rather an increase occurs only when the annealing temperature is increased.

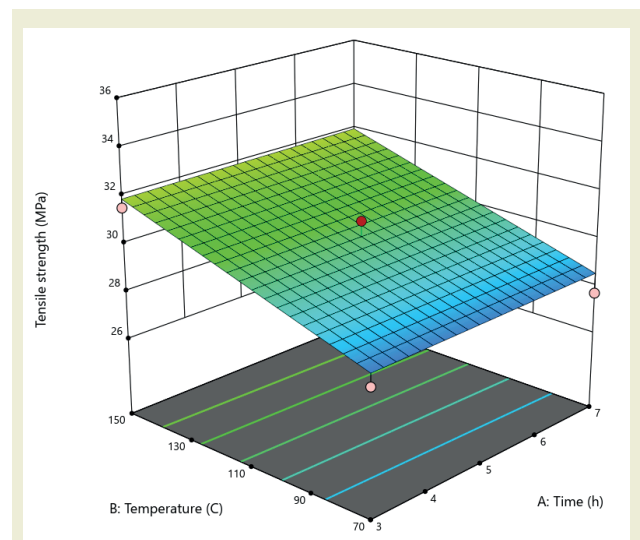


Figure 4. Dependence of annealing parameters (temperature and time) on the tensile strength. Blue fields show the lowest tensile strength values, and green and yellow fields show the highest values.

3.2. Results for the tensile strain at break

ANOVA analysis of variance indicates that the 2-factor interaction (2FI) model best fits the influence of annealing factors on the tensile strain at break. The details of analysis are shown in Table 5. The model F-value (variation between sample means) of 12.49 implies that the model is significant. There is only a 0.54% chance that an F-value this large could occur due to noise. P-values less than 0.05 suggest that temperature (factor B) has a

Table 5. Analysis of variance – influence of annealing time and temperature on tensile strain at break

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	54.13	3	18.04	12.49	0.0054	significant
A-Time	6.04	1	6.04	4.18	0.0868	
B-Temperature	36.70	1	36.70	25.41	0.0024	
AB	11.38	1	11.38	7.88	0.0309	
Residual	8.67	6	1.44			
Lack of Fit	6.81	5	1.36	0.7354	0.7038	not significant
Pure Error	1.85	1	1.85			
Cor Total	62.79	9				

prevalent effect on tensile strain at break and also interaction of factors AB which can clearly be also seen on Table 5 and Figure 3. The lack of fit F-value of 0.7354 implies that the lack of fit is not significant relative to the pure error. There is a 70.38% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good because it means that the model fits which we want.

Table 6. Summary statistics about the model for tensile strain at break

Std. Dev.	1.20	R ²	0.8620
Mean	12.44	Adjusted R ²	0.7929
C.V. %	9.66	Predicted R ²	0.6873
		Adeq Precision	10.1619

From Table 6, it can be concluded that the model followed the data very well since the coefficient of determination is $R^2 = 0.8620$. The predicted R^2 of 0.6873 is in reasonable agreement with the adjusted R^2 of 0.7929; i.e., the difference is less than 0.2. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 10.1619 indicates an adequate signal.

Tensile strain at break for PA6-CF can be described by Equation (2) in actual parameters:

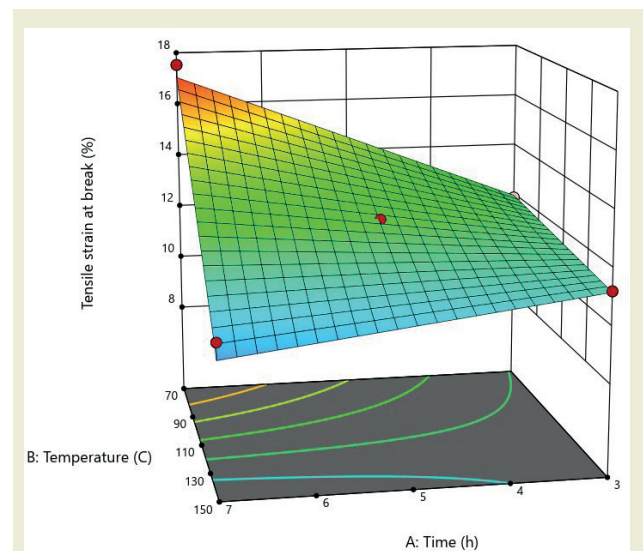
$$\varepsilon = 4.45197 + 2.7408 \cdot t + 0.053468 \cdot \vartheta \quad (2)$$

where: ε (mm/mm) – tensile strain, ϑ (°C) – annealing temperature, t (h) – annealing time.

The obtained equation for tensile strain at break is determined by the annealing temperature and time in the range, which is shown in table 1, i.e. for an annealing temperature of 50 °C – 170 °C and for an annealing time of 2 h to 7 hours.

Figure 5. shows dependence of temperature and time on the tensile strain at break. The highest strain at break is measured in case of 7 hours of annealing time and 70 °C of annealing temperature. An increase in tensile strain

at break is observable with shorter annealing time in combination with lower annealing temperatures which can also be seen from table 2.

**Figure 5.** Dependence of annealing parameters (temperature and time) on the tensile strain at break. Blue fields and contour lines show the lowest strain at break values, while the highest are shown in red.

3.3. Results for the tensile modulus

A quadratic model best fits statistical analysis of the tensile modulus. P-values less than 0.05 indicate model terms are significant. In this case B and B² are significant model terms (Table 7). The lack of fit F-value of 1.59 implies that the lack of fit is not significant relative to the pure error. There is a 51.42% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good because it means that the model fits which we want.

From Table 8, it can be concluded that the model followed the data very well since the coefficient of determination is $R^2 = 0.9360$.

Tensile modulus for PA6-CF can be described by Equation (3) in actual parameters:

Table 7. Analysis of variance – influence of annealing time and temperature on tensile modulus

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.346E+05	5	66929.04	11.71	0.0168	significant
A-Time	6584.05	1	6584.05	1.15	0.3436	
B-Temperature	44174.95	1	44174.95	7.73	0.0498	
AB	19019.17	1	19019.17	3.33	0.1422	
A ²	764.90	1	764.90	0.1338	0.7330	
B ²	1.948E+05	1	1.948E+05	34.08	0.0043	
Residual	22865.89	4	5716.47			
Lack of Fit	18904.50	3	6301.50	1.59	0.5142	not significant
Pure Error	3961.39	1	3961.39			

$$E_t = 1812.6056 + 110.6715 \cdot t - 20.0234 \cdot \vartheta - 0.86194 \cdot \vartheta \cdot t - 2.9774 \cdot t^2 + 0.1188 \cdot \vartheta^2 \quad (3)$$

The obtained equation for tensile modulus is determined by the annealing temperature and time in the range, which is shown in table 1, i.e. for an annealing temperature of 50 °C – 170 °C and for an annealing time of 2 h to 7 hours.

where: E_t (N/mm²) – tensile modulus, ϑ (°C) – annealing temperature, t (h) – annealing time.

Table 8. Summary statistics about the model for tensile modulus

Std. Dev.	75.61	R ²	0.9360
Mean	1203.77	Adjusted R ²	0.8561
C.V. %	6.28	Predicted R ²	0.5739
		Adeq Precision	10.3193

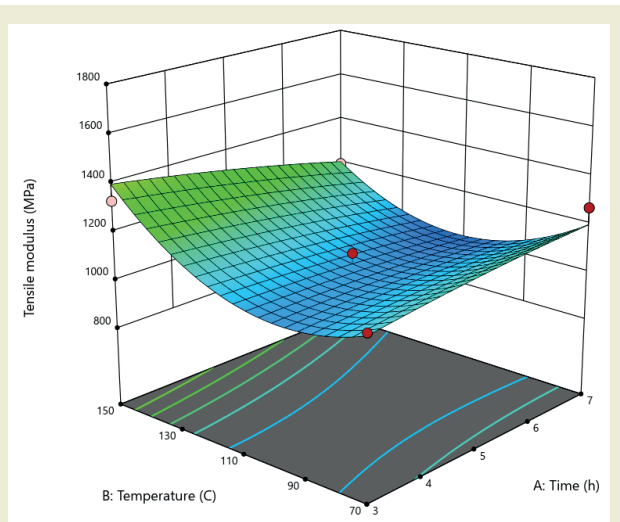


Figure 6. Dependence of annealing parameters (temperature and time) on the tensile modulus. Blue fields and contour lines show the lowest tensile modulus values, and green fields and contour lines show the highest values.

Results shown in Figure 6 show that there is a possibility in modifying the modulus of the material with the

combination of higher temperatures and longer duration or shorter duration and lower temperatures. Modulus results show that time does not significantly affect the modulus, but temperature has a range of effects. Or rather as the analysis showed in Table 7, the curve of the dependence of temperature and annealing time on the tensile modulus is quadratic and it can be seen that the higher the temperature, the greater the tensile modulus, but also at lower temperatures there is a smaller increase, while time has no effect.

4. Discussion

In previous research, the author Rhugdhriyva, R. attempted to determine the effect of annealing on reinforced ABS material, and result has shown improvement of tensile strength up to 12%. Combination of annealing and uniaxial pressure gives better results for tensile strength. [10]

In [11] author Sudin, M.N. tested ABS for the impact of annealing, and it was concluded that there is no linear relationship between annealing time and temperature for the best tensile strength results. Our results suggest that it does not seem to be the case with PA reinforced with carbon fibres since Figure 4 shows a strong linear dependence of temperature on tensile strength.

PA12 specimens reinforced with carbon fiber were annealed at a similar temperature range in [12] resulting in an increase of tensile strength (11%) and a decrease of tensile strain at break (29%) which is similar to our results. A trend of increased annealing temperatures towards higher tensile strength results is also observed in [13]. On the other hand, in [14] PLA, PLA-CF and PETG, PETG-CF were annealed at 90 °C and 120 °C for 6 and 8 hours. In the case of PETG and PETG-CF annealing did not result in any significant change in tensile strength. Annealing of PLA at 90 °C for 6 and 8 hours increased tensile strength for 13% and 17%. PLA-CF showed a decrease of interlayer tensile strength before annealing in comparison to PLA but after annealing at 90 °C for 6 and 8 hours a significant increase was recorded.

From the tests conducted it can be concluded that if one wants to increase the mechanical properties, primarily strength and modulus, it is necessary to go to much higher temperatures, and that temperatures of 80–120 °C as in these previous studies are not sufficient for improvement. The time of even 8 hours mentioned by the author Bhandari, S. et al. [14] is not necessary because time has no effect on the entire tensile properties what this research showed (all p-values > 0.5).

5. Conclusions

Bearing in mind the tensile strength results it can be said that higher temperatures and longer duration are both beneficial for tensile strength and modulus. Combination of temperature and duration of annealing gives various options for material modification in terms of desired modulus or strain at break. Results show that higher temperatures can increase tensile strength up to 16.7% and modulus up to 35%. Strain at break of the materials can be decreased up to 59%. Shorter periods of annealing can replace prolonged duration with the same or even better effect on tensile strength. As a dominant factor across all responses was temperature and we can say that higher temperature is in most cases the best way to modify PA6-CF properties by annealing. Depending on whether higher strength or higher flexibility is required, the appropriate annealing parameters can be selected from the results obtained or the balance between all mechanical properties can be determined.

The obtained equations (equations 1, 2 and 3) for calculating individual output values (strength, modulus and strain) can be of great help in industrial applications

because in the mentioned temperature range (50 °C to 170 °C) and time (2 h to 7 h), the values can be calculated immediately and there is no need for individuals in the industry to perform the tests themselves.

This work can serve as a starting point for all other fiber/particle reinforced thermoplastic material where annealing is required, but for each material it is necessary to know the glass transition and melting temperatures in order to determine the limits that must not be exceeded during post-processing heat treatment.

Research ethics

Not applicable.

Author contributions

Conceptualization: Mislav Tujmer · Methodology: Mislav Tujmer and Ana Pilipović · Formal Analysis: Mislav Tujmer and Ana Pilipović, Investigation: Mislav Tujmer · Data Curation: Mislav Tujmer · Writing - Original Draft Preparation: Mislav Tujmer · Writing - Review & Editing: Ana Pilipović · Visualization: Ana Pilipović · Supervision: Ana Pilipović · Funding Acquisition: Ana Pilipović

Competing interests

The authors state no conflict of interest.

Research funding

None declared.

Data availability

The raw data can be obtained on request from the corresponding author.

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