

# Üretim Parametrelerinin Termoplastik Kompozitlerin Mekanik Özelliklerine Etkisinin İncelenmesi

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## MAKALE BİLGİSİ

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## ÖZ

Üretim parametreleri, özellikle havacılık sınıfı polieter keton keton (PEKK)/karbon fiber (CF) kompozitlerde termoplastik kompozitlerin mekanik özelliklerinde önemli bir rol oynamaktadır. Sunulan bu çalışmada, farklı sıcaklık değerlerinde konsolide edilen PEKK/CF kompozitlerinin mekanik özellikleri değerlendirilmiştir. Benzer bir basınç profiliyle, kür sıcaklığının PEKK/CF kompozit laminatın gerilme, laminalar arası kesme dayanımı (ILSS) ve düzlem içi kesme dayanımı (IPSS) üzerindeki etkisini belirlemek için çeşitli kür sıcaklıkları uygulandı. Kompozit laminatlardaki gözeneklilik, delaminasyon ve boşluk içeriği, Ultrasonik Test yoluyla NDT yöntemi kullanılarak karakterize edildi. Bu makale, bir polieter keton keton (PEKK)/karbon fiber (CF) kompozitleri kullanılarak otoklav işlemiyle farklı kürlenme sıcaklığı numunelerinin karşılaştırılması için üretim kupon test numunelerinin bazı güncel sonuçlarını sunmaktadır. PEKK/CF'nin gerilme ve laminalar arası kayma mukavemeti özellikleri, sırasıyla 350°C, 375°C ve 400°C'de farklı kürlenme sıcaklık geçmişleri ile hazırlanacak şekilde çalışılmıştır. Yapılan testler ideal kürlenme sıcaklığını 400°C derece olarak belirlemiştir.

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## An Investigate of the Effect of Manufacturing Parameters on the Mechanical Properties of Thermoplastic Composites

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## ABSTRACT

Manufacturing parameters play an essential role in the mechanical properties of thermoplastic composites, especially in aerospace grade polyether ketone ketone (PEKK) / carbon fibre (CF) composites. In this presented study, the mechanical properties of PEKK/CF composites consolidated at different temperature values have been evaluated. Various cure temperatures were applied with an identical pressure profile to determine the effect of cure temperature on tensile, interlaminar shear strength (ILSS) and in-plane shear strength (IPSS) of PEKK/CF composite laminate. Porosity, delamination and void content within the composite laminates were characterized using the NDT method via Ultrasonic Testing. This paper presents some current manufacturing coupon test specimens for comparing different cure temperature specimens by autoclave process using polyether ketone ketone (PEKK) / carbon fibre (CF) composites. Effects of curing temperature on the mechanical properties and consolidation grade of PEKK/CF composite are studied. Tensile and interlaminar shear strength properties of PEKK/CF have been studied to prepared with different curing temperature histories at 350°C, 377°C, and 400°C, respectively. The tests were carried out have determined the ideal curing temperature as 400°C degrees.

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## 1. INTRODUCTION (GİRİŞ)

The aviation industry's rapid growth necessitates the quick advancement of new aircraft materials. The major goal is to save costs by reducing weight and extending the service life of aircraft parts. With the engineering materials with improved mechanical properties, lighter designs can be made in the frame and engines of the aircraft, thus increasing the efficiency of the aircraft and reducing the operating costs [1]. Therefore, the use of composite material in the field of engineering materials is overgrowing. The main reason for the spread of composite materials in the aviation field is that the material provides a combination of important properties such as hardness, toughness, lightness and corrosion resistance [2-6]. With the increasing interest in thermoplastic fibre composite materials in aviation, the investigation of the processing and production of these materials has increased [7].

High-performance thermoplastic composites, which is carbon fibre/ Polyether-ketone-ketone (CF/PEKK), are recently studied with great interest by the aerospace industry for reinforced structural parts. PEKK matrix materials may also be consolidated out of the autoclave since they have a lower processing temperature than PEEK matrix materials [8]. Processing properties have an essential place in the mechanical properties of thermoplastic composite parts. The matrix properties and the bond between the matrix and carbon fibres can be regulated depending on the heat treatment cycle. PEKK composites are consolidated under high temperature (around 360°C), and in this way, the viscosity of the matrix will be decreased to the porosity is lowered. During this process, changes may occur in the molecular structure of the PEKK matrix owing to the chemical change of the macromolecular chains. With this change, the chemical and mechanical properties of the matrix material can change, and this change may ultimately affect the mechanical properties of the composite part [9-10].

There are significant differences between the processing of thermoplastic composites and thermoset composites. The main reason for this difference is; The processing of thermoplastic composites is based on the matrix melting and solidification. Which is generally based on energy transfer as heat [11-14]. The production of thermoplastic composites; offers the opportunity to combine the laying, melting and consolidation steps in a continuous process. Thus, manufacturing can be made without the need for the curing process required by thermosetting materials. In

addition, while the production of thick sectioned parts is limited in thermoset composites due to limits of exothermic reaction and heat diffusion, there is no conceptual thickness limitation in a consolidated thermoplastic composite [15].

Recently, many methods are used for the production of thermoplastic composites. One of these production techniques is an autoclave. Production of large and complex parts with autoclaves is more suitable than other production methods. There is a fundamental difference in the production of thermoplastic composites and thermoset composites. While a physical change occurs only in the matrix material in thermoplastic composites, a chemical reaction occurs in the matrix material of thermoset composites. Despite this fundamental difference, the experience gained from the production of thermoset composites is used in the autoclave production process. In the production of thermoplastic composites by autoclave, there is usually a need for processing above 300°C. Therefore, an autoclave that can reach high temperatures is required for production with this method [16].

The procedure comprises melting and softening the bonding area by heating the layer interface while applying pressure to achieve interfacial contact between the two surfaces. Thermoplastic composites go through a multitude of microstructural changes during processing that affect their characteristics [17-20]. Melting, degradation, crystallization, bonding, residual strain generation, and consolidation are examples of these changes, which must account for void consolidation and void expansion. The process parameters have a direct impact on all of these modifications.

The adhesion of thermoplastics, particularly thermoplastic composites, has been studied for many years. The composite surfaces are squeezed together for a period of time above the polymer matrix's processing temperature to permit adhesion. A construct of strength at the interface is then caused by a number of processes [21,23,24]. The adhesion process, according to Wool and O'Connor [22], consists of five steps: (1) surface reconfiguration, (2) surface approach, (3) wetting, (4) diffusion, and (5) randomization. The assembly has no mechanical strength during the first two phases because the initial interface is still there. To ensure contact at the microscopic scale, deformation of the surface roughness is required, which is driven by contact pressure and wetness. This is the so-called physical contact, which has been investigated and modelled in

a variety of industrial processes by different authors. Once intimate contact is established, the interface gradually fades away due to a healing process (steps (4) and (5)), and the mechanical strength of the interface develops to eventually match that of the bulk.

This article aims to determine the effect of autoclave cycle temperature in the production of CF/PEKK composite materials. For this purpose, plates were produced at three different consolidation temperatures, and these temperatures are 350°C, 377°C, 400°C, respectively. It is aimed to determine the effect of the temperature parameter on the mechanical properties of the material during production by performing Tensile, ILSS and IPSS tests on samples in different sequences.

## 2. MATERIAL AND METHODS (YÖNTEM VE TEKNİKLERİ)

The thermoplastic composite material used in this study was a unidirectional carbon fibre reinforced PEKK based polymer APC/AS4D 12K produced by Solvey S.A. Company. 305 mm wide UD tape was used for plate production in the autoclave. The tape is shown in Figure 1. The nominal prepreg fibre areal weight (FAW) and the initial prepreg matrix weight fractions were 145 g/m<sup>2</sup>(gsm) and 34% respectively according to product datasheet [25].

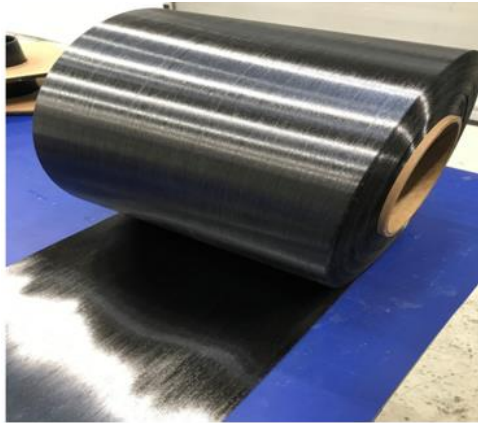


Figure 1. UD PEKK/CF Tape (*Tek yönlü karbon fiber PEKK kompozit bant*)

For this study, 12 symmetrical laminate plates in four different configurations were produced at three different curing temperatures.



Figure 2. Welding Process (*Kaynak işlemi*)

The stacking sequence and number of plies are given in Table 1 according to the curing temperatures of all laminates manufactured. All autoclave consolidated laminates were hand-laid, with each ply ensured a welding point of a tiny size utilizing handbuilt welding. The welding process is given in Figure 2.

### 2.1 Specimen Manufacturing (*Test Kuponu Üretimi*)

Composite structures prepared with four different laying directions were produced using the autoclave process. Each sample was given a reference number for the distinction of sample sets and is shown in Table 2. In the cycle used in the autoclave, the same pressure value was used for all samples, and while all variables were kept the same, only the curing temperature (dwell temperature) was changed. The autoclave cycle is given in Figure 3.

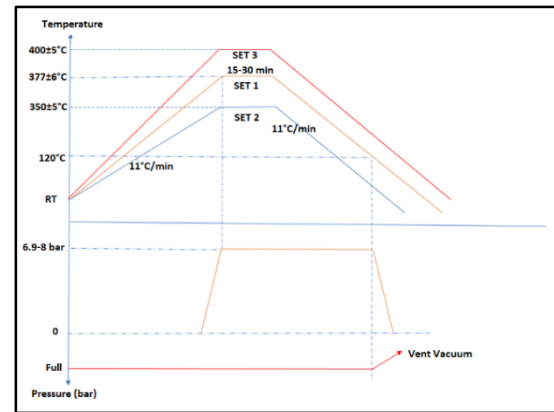


Figure 3. Autoclave Cycle (*Otoklav Döngüsü*)

After processing, the data from the autoclave cycle was analyzed to ensure that the production requirements were met.



Figure 4. NDT Control (NDT kontrolü)

### 2.2 Ultrasonic NDT (Ultrasonik Tahribatsız Numune)

Using automated Doppler Type Phascan devices, ultrasonic scanning using through-transmission was used to examine all of the laminates. NDI was performed by wetting the plate surface with water. A-scans, B-scans, and C-scans for all of the laminates were evaluated and the results via Visual Scan software, which enables processing with attenuation and amplitude modes. For the examination of all specimens, a standard frequency of 10 MHz was selected. Using PAUT image analysis software, the mean ultrasonic attenuation (in dB) values of each

CF/ PEKK laminate were computed. NDT control is shown in Figure 4.

### 2.3 Void Content (Boşluk İçeriği)

The void content of the CF/PEKK specimens was estimated using methodologies immersion for this analysis. For void content analysis, a minimum of three 20\*10 mm coupons from each plate were utilized. The samples are kept in 23°C and 50% humidity for at least 2 hours before testing. The test sample is stored under these conditions or conditioned before testing.

The densities of the cut plates are then determined using the "TS EN ISO 1183 Standard, Plastics-Non-porous Plastics- Density. Determination Methods - Part 3: Gas Pycnometer Process" standard. Then, using the "ASTM D792 Standard, Test Method for Determining Density and Specific Weight of Plastics by Displacement," the densities of the same plates are determined. By substituting the measured density data in the formula is given Eq.1, the void ratios of the plates can be determined.

$$V_0 = \frac{\rho_{pyc} - \rho_{immerson}}{\rho_{pyc}} * 100 \quad (1)$$

$V_0$  = Voit Content

Table 1. Laminate specification (Laminat özellikleri)

Laminates	Plate Code	Size (mm)	Stacking Sequence	Plies	Test Specimen Size (mm)
CT 350 °C	B1	200x200x5,2	[(0)2 (+/-45)16S (0)2]	36	250x15x1
	B2	210x265x2,3	[90]16	16	175*25*2,3
	B3	310x215x2,3	[(+/-45)16]s	16	200*25*2,3
	B4	330x180x1	[0]8	8	30*10*5,2
CT 377 °C	A1	200x200x5,2	[(0)2 (+/-45)16S (0)2]	36	250x15x1
	A2	210x265x2,3	[90]16	16	175*25*2,3
	A3	310x215x2,3	[(+/-45)16]s	16	200*25*2,3
	A4	330x180x1	[0]8	8	30*10*5,2
CT 400 °C	C1	200x200x5,2	[(0)2 (+/-45)16S (0)2]	36	250x15x1
	C2	210x265x2,3	[90]16	16	175*25*2,3
	C3	310x215x2,3	[(+/-45)16]s	16	200*25*2,3
	C4	330x180x1	[0]8	8	30*10*5,2

Table 2. Specimens reference numbers (Test kupon referans numaraları)

Stacking Sequence	Specimens					Cure Temperature	SET	Specimen Size (mm)
	1	2	3	4	5			
[0] <sub>8</sub>	1a	2a	3a	4a	5a	377°C	SET 1 (Varyant A)	250x15x1
[90] <sub>16</sub>	6a	7a	8a	9a	10a			175*25*2,3
[+/-45] <sub>16</sub> s	11a	12a	13a	14a	15a			200*25*2,3
[(0) <sub>2</sub> (+/-45) <sub>16</sub> s (0) <sub>2</sub> ]	16a	17a	18a	19a	20a			30*10*5,2
[0] <sub>8</sub>	1b	2b	3b	4b	5b	350°C	SET 2 (Varyant B)	250x15x1
[90] <sub>16</sub>	6b	7b	8b	9b	10b			175*25*2,3
[+/-45] <sub>16</sub> s	11b	12b	13b	14b	15b			200*25*2,3
[(0) <sub>2</sub> (+/-45) <sub>16</sub> s (0) <sub>2</sub> ]	16b	17b	18b	19b	20b			30*10*5,2
[0] <sub>8</sub>	1c	2c	3c	4c	5c	400°C	SET 3 (Varyant C)	250x15x1
[90] <sub>16</sub>	6c	7c	8c	9c	10c			175*25*2,3
[+/-45] <sub>16</sub> s	11c	12c	13c	14c	15c			200*25*2,3
[(0) <sub>2</sub> (+/-45) <sub>16</sub> s (0) <sub>2</sub> ]	16c	17c	18c	19c	20c			30*10*5,2

### 3. RESULTS (SONUÇLAR)

#### 3.1. NDT Results (NDT Sonuçları)

Figure 5 displays ultrasonic C-scans corresponding to laminates made with an autoclave. C-scans are shown on a colour scale that indicates various degrees of attenuation (in dB).

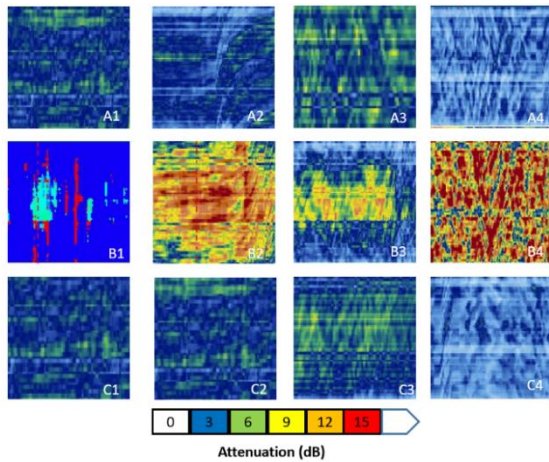


Figure 5. Ultrasonic C-Scan Result of Variant A,B and C plates (A,B ve C varyant plakaları için ultrasonik C-tarama sonuçları)

Because to defects in the laminate, such as voids, dispersed porosity, or poor layer consolidation, an ultrasonic beam traveling because of a plate can be attenuated. Attenuated areas are located in specimens with the most undesirable manufacturing conditions, suggesting that entrapped air was not adequately extracted and the plate's degree of consolidation might not be sufficient for industrial applications. In C-scan analysis, laminates with attenuation values less than 6 dB are deemed homogeneous and defect free.

Anomalies, defined as areas with attenuation larger than 6 dB, must be studied. A 6 dB threshold is applied to thin laminates [26-27]. Variant A and Variant C plates display no discernible signal attenuation above 6 dB. As a result, keeping the autoclave processing temperature at 377°C and 400°C for 15-30 minutes had no impact on the NDI of CF/PEKK laminates. The curing temperature of 350°C was insufficient for the adhesion of the layers on the variant B plates.

#### 3.2. Void Content Results (Boşluk İçeriği Sonuçları)

Figure 6 summarizes the findings of the void material study. It includes average void content values for each laminate tested using the pycnometer and immersion methodology. It was possible to obtain a porosity range of 0.99% to 3.11%. The horizontal dotted line is set to 2% void material, a standard threshold in engineering applications. In the pycnometer and immersion method, the void ratio is mainly determined by calculating the density difference. As a result, the density approach is a quick way to evaluate something that needs prior knowledge of specific theoretical material properties and can result in slightly inaccurate porosity values [28]. Despite these minor errors, the process is widely used and accepted.

According to the NDI results, there is no problem with consolidation in the plates produced as Variant A and Variant C, so laminates cured at 377°C and 400°C have less than 2% void content. On the other hand, low temperatures significantly impact the presence of cavities during autoclave production. The curing temperature parameter tends to obey a strict sufficient degree of consolidation in autoclave manufacturing. Only Variant A and Variant C laminates have a void

content of less than 2%. The highest void content value (2.61%) was obtained at 350°C, as expected by C-scans of laminate B4, and these degradation effects suggest discontinuities in the matrix. Based on these observations, autoclave curing of CF / PEKK laminates necessitates processing temperatures near 400°C to drain the voids inherent in the prepreg raw material. The B1 laminate, on the other hand, has a void content of less than 2% in the tests conducted, which is due to its thin thickness.

The effect of temperature on the void ratio was investigated due to this study by holding the pressure constant in all cycles among the pressure and temperature parameters, which are the two most crucial output parameters in autoclave production. In laminates manufactured between 1mm and 5mm, the effect of the curing temperature on the void ratio tends to be more dominant as the thickness increases.

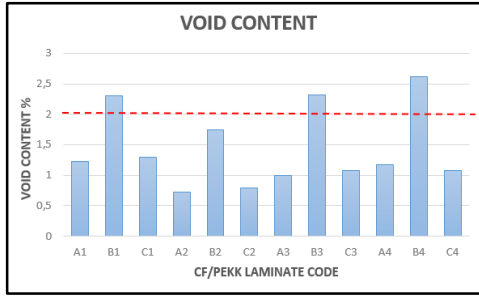


Figure 6. Void Content (Boşluk sonuçları)

The defects in the coupons were studied using electron microscope (SEM) images (100X) of the created plates. Figure 7 shows how SEM images of Variant A3, B3, and C3 coupons correlated well with ultrasonic performance. In the autoclave production process, it has been observed that a curing temperature of 350°C is insufficient to achieve layer adhesion (Figure 7).

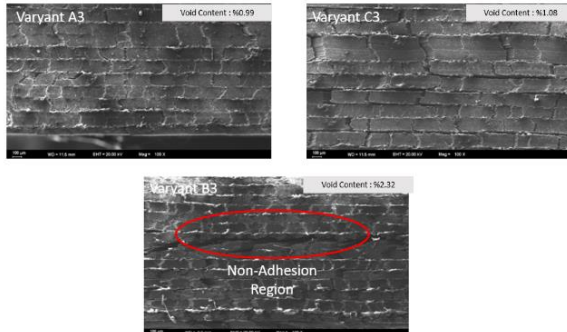


Figure 7. 100X SEM Images (100X Elektron mikroskobu görüntüsü)

### 3.3. Mechanical Testing Results (Mekanik Test Sonuçları)

Many authors have stated that the void content of material harms CFRP mechanical properties controlled by the matrix [29,30,31]. Similarly, the void quality of composite structures is directly related to manufacturing parameters. The results of tensile tests at 0 and 90 degrees and In-plane shear strength (IPSS) and Interlaminar shear strength (ILSS) mechanical tests on CF/PEEK laminates are discussed in this section. Test specimens detail is given Figure 8.

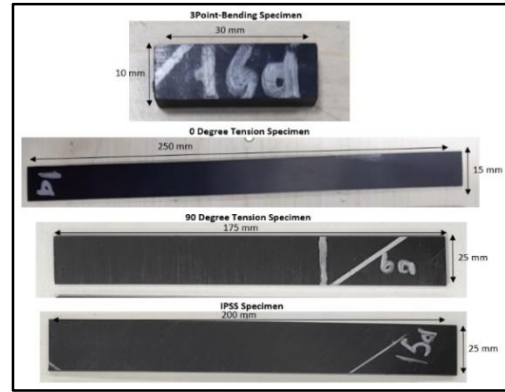


Figure 8. Test specimen details (Test numunelerin detayları)

#### 3.3.1. IPSS test result (IPSS test sonuçları)

ASTM 3518 standard was used to research test coupons made with three different curing temperature variations. Table 3 shows the mechanical properties calculated based on the test results for all variants. The comparison of IPSS test results according to in-plane shear strength values is given in Figure 9. According to this comparison, the weakness of the fibre-matrix bond in low-temperature curing was confirmed by the test results. Figure 2 shows SEM images magnified 500 times. Low temperature cured B variant plates displayed voids and matrix-fibre adhesion weakness, as seen in SEM images. According to test results, the material's mechanical properties are affected by this void and bond weakness.

Table 3. IPSS test result (IPSS test sonuçları)

TEST COMPONENT	In-Plane Shear Strength (MPa)	In-Plane Shear Modulus (GPa)	Poisson Ratio
A3-IPSS Test	103	5,13	0,633
B3-IPSS Test	98,9	3,76	0,719
C3-IPSS Test	154	5,05	0,656

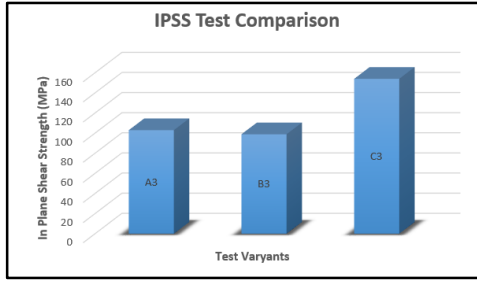


Figure 9. In-plane shear strength comparison (*Düzlem içi kesme dayanımı karşılaştırması*)

The highest mechanical strengths were observed in C variant plates cured at 400°C. However, the lowest void ratio in the C variant and the success in matrix-fibre adhesion confirms the reason for their high mechanical strength.

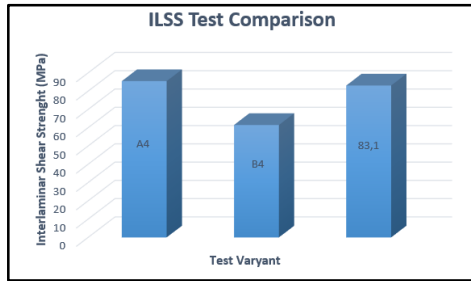


Figure 10. IPSS test plates SEM Images 500X (*500X IPSS test plakası elektron mikroskobu görüntüsü*)

### 3.3.2. ILSS test result (*ILSS test sonuçları*)

Three-point bending tests were used to assess interlaminar shear strength. All of the coupon tests were completed in compliance with ASTM D 2344. Tests were carried out with 20 mm between two support points and 6 mm contact point radius according to ASTM 2344. Table 4 shows the mechanical test results of coupons made with three different variants. The mechanical values of the Variant B coupons are significantly lower than the others, as shown in the test results. The main explanation for this is that in Variant B, the curing temperature is too close to the material's melting temperature, preventing a sufficient fibre-matrix bond.

Table 4. ILSS test result (*ILSS test sonuçları*)

Test Component	Interlaminar Shear Strength (MPa)	Interlaminar Shear Peak Load (kN)
A4-ILSS Test	85,5	6163
B4-ILSS Test	61,5	4442,7
C4-ILSS Test	83,1	6010,6

The comparison of interlaminar shear strength of test coupons produced with three different variants is shown in Figure 11. The highest void ratio was detected in ILSS test coupons delivered with Variant B. One reason for this can be predicted as the high pressure and temperature conditions in the autoclave production method, which becomes difficult to control by increasing the thickness of the part. SEM images for all variants are given in Figure 12.

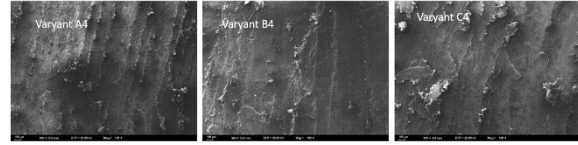


Figure 11. Interlaminar shear strength comparison (*Tabaka içi kayma dayanımı karşılaştırması*)

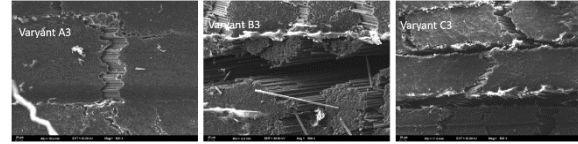


Figure 12. ILSS test plates SEM Images 100X (*100X ILSS test plakası elektron mikroskobu görüntüsü*)

### 3.3.3. 0 degree tensile test result (*0 derece çekme testi sonuçları*)

Tensile strengths for 0-degree tensile test specimens are given in Table 5, and tests were carried out according to ASTM D 3039 standards. In these results, it is seen that the B variant, which has the lowest mechanical strength in general, shows higher strength than the A variant. This can be predicted as the expected lack of adhesion between the bonds due to a vacuum loss during the autoclave cycle. Furthermore, since the tensile direction is applied as the UD band's 0 direction, the primary strength factor occurs in the fibres' orientation. In this case, fibre strength is the primary determinant of mechanical strength value, with the matrix acting as secondary support. SEM representations of the fracture surfaces are shown in Figure 13. The stripping of the fibers from the matrix in the A variant test coupons, as seen in the SEM photos, explains the reduction in mechanical strength. 0-degree test results comparison diagram is given in Figure 14.

### 3.3.4. 90 degree tensile test result (*90 derece çekme testi sonuçları*)

Tests were carried out according to ASTM D 3039 standards. Table 6 tests revealed a linear relationship between void ratios, and it was seen that the B version test coupons had the lowest tensile strength and the highest void ratio.

Table 5. 0 degree tensile test result (0 derece çekme test sonuçları)

Test Component	Tensile Strength (MPa)	Tensile Modulus (GPa)	Poisson Ratio
A1-0 Degree Tensile Test	1950	132	0,355
B1-0 Degree Tensile Test	2130	128	0,324
C1-0 Degree Tensile Test	2260	132	0,34

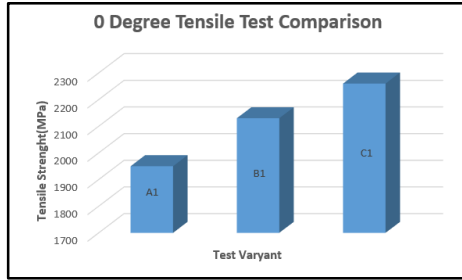


Figure 13. 0 degree tensile test specimen SEM Images 1500X (1500X 0 derece çekme test numunesi elektron mikroskobu görüntüsü)

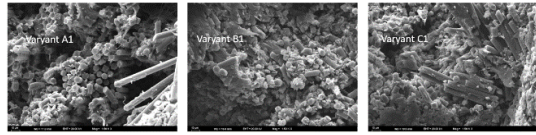


Figure 14. 0 degree tensile test results comparison (0 derece çekme testi sonuçları karşılaştırması)

Table 6. 90 degree tensile test result (90 derece çekme test sonuçları)

Test Component	Tensile Strength (MPa)	Tensile Modulus (GPa)	Poisson Ratio
A2-90 Degree Tensile Test	62,6	9,50	0,0321
B2-90 Degree Tensile Test	34,6	8,07	0,0374
C2-90 Degree Tensile Test	72,5	9,58	0,0330

SEM images of the tests are given in Figure 15. According to SEM visuals, the B variant test coupons had the weakest fiber-matrix bond of the three variants.

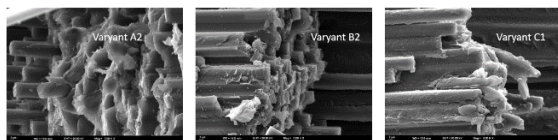


Figure 15. 90 degree tensile test specimen SEM Images 3000X (3000X 90 derece çekme test numunesi elektron mikroskobu görüntüsü)

The comparison of the tensile strength tests performed for the three variants is given in Figure 16.

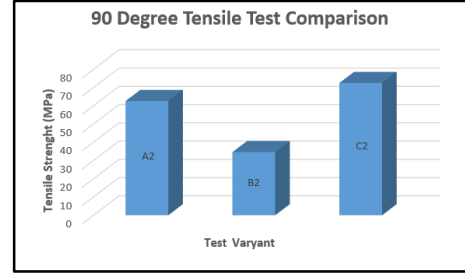


Figure 16. 90 degree tensile test results comparison (90 derece çekme testi sonuçları karşılaştırması)

#### 4. CONCLUSION (SONUÇ)

In this work, 12 thermoplastic CF/PEKK composite laminate plates were produced at three different curing temperatures. While the pressure and dwell time were kept constant on the autoclave cycle, a controlled production was realized by changing only the temperature parameter. All of the experiments have been carried out in compliance with the ASTM standard. The void ratios and mechanical strengths in the plates are inversely proportional to the test results. Generally, the lowest void ratios and the highest mechanical forces were seen in Variant C plates produced at 400°C. The curing temperature is very close to the melting temperature of the matrix material, which results in high void ratios and low mechanical strengths, particularly in plates produced at 350°C (Variant B). Due to its proximity to the melting point, the fibre-matrix adhesion was not achieved at the desired degree, resulting in low strength.

According to SEM images, the adhesion between fiber and matrix was greater in plates produced at 377 degrees and 400 degrees than in plates produced at 350 degrees. Due to the proximity of the prepreg material to the melting temperature of 343 degrees, complete adhesion could not be created, particularly in the plate generated at 350 degrees. During the experiments, the fibre began to peel away from the matrix due to this condition. The SEM images also revealed that pressure was not the sole cause of the material's void ratio. Although all plates are made under the same pressure and vacuum, plates with a low-temperature curing value have higher void ratios than plates with a higher temperature curing value. The temperature needed for the matrix to melt and warp the fibres is the key reasons for this. Since this matrix melted at low temperatures and did not display a sufficiently and homogeneous distribution, the void ratios of the plates formed at 350 degrees were higher than the others. The increased void ratios are one of the factors that directly cause the decrease in mechanical strength. While the strength of the



samples produced at 377°C is close to that of the samples produced at 400°C in some tests, the resistance of the samples produced at 400°C is higher. Another finding of these experiments is that manufacturing at temperatures very similar to the melting temperature of the matrix harms mechanical strength. As a result of the experimental study, the ideal production temperature was determined as 400°C.

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#### CONFLICT OF INTEREST STATEMENT (ÇIKAR ÇATIŞMASI BİLDİRİMİ)

The authors declare that no potential conflict of interest.

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