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The Influence of Fiber Treatment and Matrix Type on the Impregnation Quality of Carbon Fiber Reinforced Thermoplastics

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Abstract
One of the main issues with Carbon Fiber Reinforced Thermoplastics (CFRTP) is the poor impregnation quality of the matrix on carbon fiber due to the high viscosity of the thermoplastic. Impregnation quality can significantly affect the mechanical properties of the composite. This study aims to compare the impregnation abilities of various types of thermoplastic matrices on carbon fiber. The matrices used in this study are HDPE, PC, and PET. Three variations of carbon fiber treatment were employed: the first variation involved immersion in liquid nitrogen at -196°C, the second variation included heating in an electric furnace at 600°C followed by rapid cooling in liquid nitrogen, and the third variation utilized treatment with a silane coupling agent. The research findings demonstrate that composites comprising a Polycarbonate matrix and carbon fiber reinforcement, treated with a silane coupling agent, exhibit superior impregnation quality, as evidenced by an Interfacial Shear Strength (IFSS) value of 9.34 MPa.. The lowest impregnation quality was observed in HDPE reinforced with carbon fiber that had been heated and rapidly cooled, with an IFSS of 5.52 MPa.

INTRODUCTION

Carbon Fiber Reinforced Thermoplastics (CFRTP) is an engineering material that has properties such as high stiffness, strength, and recyclability, as well as a short processing time (Yao et al., 2019). The mechanical properties of CFRTP composites are affected by the interaction between the fiber and the matrix. However, carbon fibers and matrices have poor interfacial bonding because carbon fibers are nonpolar and have low adsorption when combined with thermoplastics, this results in a low interfacial shear strength in CRFTP (Wenzhong, 2015;Sharma et al., 2014). Another problem is that the thermoplastic matrix has a high melt viscosity (500-5000 Pa·s) which increases the difficulty of impregnation between the matrix and the fiber (Wenzhong, 2015). The quality of the impregnation can be improved by increasing the roughness and interaction at the fiber interface. Many methods can be used to increase the surface roughness of the fiber such as chemical treatment, electrochemical treatment, and plasma treatment (Dai et al., 2011;Wong et al., 2012;Vishkaei et al., 2011;Käppler et al., 2014).

Impregnation quality refers to how well a matrix material fills and surrounds the reinforcing fibers. It is a measure of how effectively the matrix material wets and adheres to the fibers, ensuring a strong bond and uniform distribution of the matrix material throughout the composite structure. The impregnation quality can be indicated by the IFSS (Koubaa et al., 2013). A composite with high impregnation quality tends to exhibit higher IFSS.

Lee and Kang (1997) conducted a study where they exposed carbon fiber to air oxidation and heat treatment at 700 °C in order to alter the physicochemical properties and morphology of the fiber.. Heat treatment has been shown to reduce the functional groups and increase the roughness of the fiber surface (Wang et al., 2006).

Wenzhong (2015) applied a silane coupling agent and plasma treatment to increase the interfacial shear strength between carbon fiber and thermoplastic matrix. The three-point bending test results showed that composite specimens using treated fiber had a 48.7% increase in interfacial strength compared to those using untreated carbon fiber.

Budiyantoro et al (2020) conducted a study on the effect of pultrusion extrusion parameters on the impregnation quality of thermoplastic composite filaments. High-impact polypropylene copolymer was chosen as the matrix and carbon fiber was used as reinforcement. Carbon fiber treatment was carried out with three variations, namely: vinytrimethoxysilane (VTMS), aminopropyltriethoxy silane (APTS), and liquid nitrogen. Treating the fiber with liquid nitrogen, with the right combination of melting temperature parameters and withdrawal speed, can increase Interfacial Shear Strength (IFSS). Liquid nitrogen can also affect the surface roughness of the fiber, which provides a better bond between the fiber and the matrix.

Zhang et al (2004) compared the effect of several carbon fiber treatments on fiber surface roughness and IFSS. Two types of treatment were given to carbon fiber, namely oxidation in hot air at 500 °C and 600 °C for 1 hour, and immersion in liquid nitrogen for 10 minutes and 20 minutes. Both types of treatments could increase the surface roughness of carbon fibers, therefore enhancing the IFSS of fiber and epoxy bonding due to the mechanical interlocking. Compared to oxidation treatment, cryogenic treatment has a shorter treatment time and is more environmentally friendly. It also leads to a higher improvement in strength and modulus (Shao et al., 2017).

Yao et al (2017) investigated the interfacial adhesion properties between carbon fibers and polycarbonate matrix using a single-filament fragmentation test. The study analyzed the influence of fiber surface treatment and matrix properties on interfacial adhesion strength. The authors found that surface treatment of the fiber can significantly improve interfacial adhesion strength, and the polycarbonate matrix also plays a crucial role in determining the interfacial properties.

Chandran and Padmanaban (2019) evaluated the interfacial adhesion in self-reinforced polyethylene and polypropylene composites using the microbond fiber bundle pullout technique. The study aimed to investigate the effect of fiber surface treatment and matrix melt flow rate on the interfacial adhesion of the composites. The results showed that the fiber surface treatment significantly improved the interfacial adhesion, and the melt flow rate of the matrix had a marginal effect on the adhesion. The authors concluded that the microbond fiber bundle pullout technique is a reliable and efficient method for evaluating interfacial adhesion in self-reinforced composites.

Shonaike et al (1996) investigated the effect of fiber loading on the interfacial properties of polyethylene terephthalate (PET)-glass fiber composites. They used a pull-out test to measure the IFSS of the composites and found that increasing the fiber loading resulted in a higher IFSS. They also used scanning electron microscopy (SEM) to examine the fracture surfaces of the composites and observed improved interfacial adhesion with increasing fiber loading. The authors concluded that fiber loading plays a crucial role in determining the interfacial properties of PET-glass fiber composites and increasing the fiber loading can lead to improved interfacial adhesion and mechanical properties. This study provides valuable insights into the design and optimization of PET-glass fiber composites for various engineering applications.

From the above background, it can be concluded that the impregnation quality on the surface of CFRTP is still very low, thus further research is needed. This study aims to obtain a combination of matrix and fiber that has high and consistent impregnation quality, resulting in high interfacial bonding between the matrix and the fiber. In this study, thermoplastic materials of HDPE, PC, and PET were used, and the fiber was treated with cryogenic, heat treatment, cryogenic and silane coupling agent treatments, which will be molded using an injection molding machine.

EXPERIMENTAL METHOD

Materials and tools

The tool used for specimen fabrication in this study is the Meiki-70B injection molding machine. The thermoplastic materials, in the form of pellets, including PET, Polycarbonate (PC), and HDPE were obtained from SABIC Plastic (Sabic, 2022). Carbon fiber reinforcement was used, specifically T700SC 12K carbon fiber produced by Toray Composite Materials America, Inc (Torayca, 2018). The carbon fiber was cryogenically treated using liquid nitrogen at a temperature of -196 ˚C, and it was placed in a container for 10 minutes (Shao et al., 2017;Zhang et al., 2004). For heat treatment, the carbon fiber was heated in a furnace at a constant temperature of 600 ˚C for 10 minutes using a Nabertherm model N 11/H machine (Wang et al., 2006). Silane coupling agent treatment was conducted by immersing the carbon fiber in an APTS solution produced by Hangzhou Jessica Chemicals Co., Ltd, with a concentration of 0.1 wt% (weight concentration) or 1 gram of silane solution mixed with 1 liter of distilled water, and the solution was adjusted to pH 4.2 with an acetic acid solution, then stirred for 1 hour. The carbon fiber was then immersed in the solution for 20 minutes, followed by drying at room temperature (Budiyantoro et al., 2021).

Specimen Manufacturing

The treated carbon fiber is then placed into the injection mold and then by using a defined setting parameter, the melted thermoplastic injection covers the fibers and therefore the molded specimen can be produced. In this case, the specimens have dimensions of 75 x 10 x 4 mm. In the process of making specimens using an injection molding machine, injection pressure, and melt temperature are adjusted according to the type of material to be molded, Table 1 shows the processing parameter (Chen et al., 2023; Osarenmwinda & Olodu, 2018; Chang et al., 2000). After the specimens were molded, the matrix material surrounding the carbon fibers wasremoved from a portion of the specimen to leave a remaining bond length of 3-4 mm, as can be seen in Figure 1. The resulting specimen is then gripped by the exposed fiber end and pulled out at a constant rate of displacement using a universal testing machine (Jia et al., 2011; Kamps et al., 2018). The pull-out test is a commonly used method to determine the IFSS between the matrix material and the reinforcing fibers in a composite material. It involves applying a tensile force to a fiber that is embedded in the matrix material and measuring the force required to pull the fiber out of the matrix (Chandran & Padmanabhan, 2019). During the pull-out test, the load-displacement data is collected and used to calculate interfacial shear strength and other mechanical properties. The formula used to calculate the IFSS value is shown in Equation (1).

$$\tau = \frac{F}{\pi d \times L_b} \qquad (1)$$

Where F is the maximum load, d is the diameter of fiber bundles and L_b is the bonding length.

Table 1. Injection Molding Parameter Setting

Material	Parameter	Value	Unit
HDPE	Melt Temperature	160	˚C
	Injection pressure	133	bar
PET	Melt Temperature	265	˚C
	Injection pressure	140	bar
	Drying temperature	165	˚C

	Drying Time	4 - 6	hours
PC	Melt Temperature	267	˚C
	Injection pressure	140	bar
	Drying temperature	120	˚C
	Drying Time	2 - 3	hours

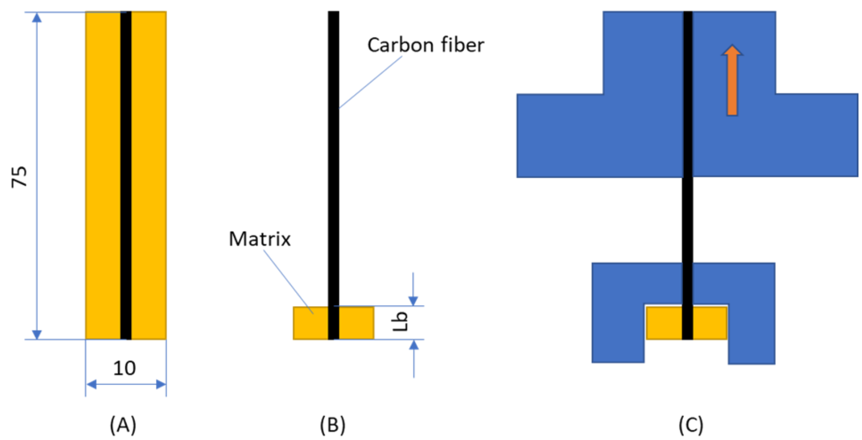


Figure 1. Specimen preparation: (A) Injection molded; (B) Partially removed matrix; (C) Pull-out test

Experimental Design

Taguchi's Design of experiment (DOE) was employed to statistically analyze the effect of thermoplastic material and fiber treatment type on IFSS. The two factors investigated were thermoplastic material (PET, PC, and HDPE) and fiber treatment type (cryogenic using liquid nitrogen, heat treatment followed by liquid nitrogen, and γ-aminopropyl triethoxysilane). These factors are listed in Table 2 and are expected to have a significant influence on IFSS.

Table 2. Factor and Levels for DOE

Factors	Levels		
Thermoplastic type	HDPE	PET	PC
Fiber treatment	Liquid nitrogen	The heat treatment followed by liquid nitrogen	γ-aminopropyl triethoxysilane

With the help of Minitab 14 software, an orthogonal array was selected based on the most optimal for designing experiments using L9 (3^2) as shown in Table 3. The experimental design means that the experiment is carried out 9 times to produce the final value. In each experiment, 3 specimens were produced and the interfacial shear strength values were obtained using the average value from three samples of each run

Table 3. Orthogonal Array L9 (3^2)

Run	Material	Fiber treatment
Run 1	HDPE	Liquid Nitrogen
Run 2	HDPE	The heat treatment followed by liquid nitrogen
Run 3	HDPE	γ-aminopropyl triethoxysilane
Run 4	PET	Liquid Nitrogen
Run 5	PET	The heat treatment followed by liquid nitrogen
Run 6	PET	γ-aminopropyl triethoxysilane
Run 7	PC	Liquid Nitrogen
Run 8	PC	The heat treatment followed by liquid nitrogen
Run 9	PC	γ-aminopropyl triethoxysilane

In the Taguchi method, performance characteristics are adjusted to optimization criteria and divided into three categories, namely larger is better, nominal is best, and smaller is better. The target of this research is to achieve the maximum value of IFSS, therefore the Taguchi method chosen is larger is better. This category can be calculated using equation (2).

$$S/N = -10log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Yi^2}\right) \quad (2)$$

Where i is the number of experiments, n is the total number of experiments (in this study, 9 experiments), and Yi is the average value.

Analysis of Variance (ANOVA) is a mathematical procedure used to compare responses with error data to determine whether independent variables or interactions are significant or not. When analyzing the DOE, ANOVA uses sums of squares to compare effects with error variances, thus determining statistical significance. By calculating the percentage contribution from ANOVA, significant factors that influence each response can be determined.

RESULTS AND DISCUSSION

Pull out test results

The combination of thermoplastic matrix types PET, HDPE, and PC with variations of carbon fibers treated with liquid nitrogen, APTS, oxidation, and liquid nitrogen. The results of the average IFSS can be seen in Table 4. Table 4 shows the results of the IFSS of specimens. Before testing, it is necessary to ensure that the specimens have no traces of plastic matrix on the carbon fibers, as using defective specimens may risk fiber breakage during testing.

Table 4. IFSS from the pullout test

Run	Thermoplastic type	Fiber treatment	Average IFSS (MPa)	Standard deviation	SNR
Run 1	HDPE	Liquid Nitrogen	7.68	0.08	17.7072

Run 2	HDPE	Heat treatment followed by liquid nitrogen	5.52	0.43	14.8388
Run 3	HDPE	γ-aminopropyl triethoxysilane	6.25	0.78	15.9176
Run 4	PET	Liquid Nitrogen	5.73	0.83	15.1631
Run 5	PET	Heat treatment followed by liquid nitrogen	6.33	0.93	16.0281
Run 6	PET	γ-aminopropyl triethoxysilane	6.65	1.05	16.4564
Run 7	PC	Liquid Nitrogen	6.63	0.49	16.4303
Run 8	PC	Heat treatment followed by liquid nitrogen	5.9	0.54	15.417
Run 9	PC	γ-aminopropyl triethoxysilane	9.34	0.81	19.4069

Table 4 presents the IFSS obtained from the pull-out test, including the standard deviation and Signal-to-Noise Ratio (SNR) calculations.. The highest IFSS value was obtained for the PC material and APTS-treated fiber variation, which was 9.34 MPa. The lowest value obtained for the HDPE material and liquid nitrogen-treated fiber variation that had previously been subjected to heat treatment was 5.52 MPa.

The IFSS in the data can be analyzed that the PC material and APTS treatment have high IFSS because the composite with PC material and fiber treatment using silane coupling agents can increase the interaction on the interface bond so that the IFSS can increase. PC thermoplastic matrix and silane coupling agent treatment can increase interfacial shear strength because the polarity strength of the interface increases due to silane interaction on the fiber surface pushing chemical bonds at the interface to get effective increases (Shao et al., 2017). The combination of polycarbonate matrix and silane coupling agent fiber treatment can increase IFSS due to chemical reactions at the interface because of silane coupling agent treatment (Wang et al., 2021).

Signal to Noise Ratio (SNR)

The response parameter of the tested specimens was analyzed using the Larger is Better method or focused on a specific value. Then, the data was transformed into S/N form to find the influential factor values on quality characteristic variations, when S/N for quality characteristics increases, the obtained results will be better. At this stage, the selection is needed to minimize the disturbance by choosing the highest SNR value (Muñoz, 2013). The calculation of the Main effects of Signal-to-Noise Ratio (SNR) concerning IFSS was conducted for each observed factor, namely the variation of thermoplastic types and the variation of treatments on fibers, as can be seen in Table 5.

Table 5. Main effects of SNR response on IFSS value

Level	Average SNR based on thermoplastic type	Average SNR based on fiber treatment
1	16,154	16,433
2	15,882	15,427
3	17,084	17,259
Delta	1,20	1,83
Rank	2	1

Based on the main effect of thermoplastic type, level 3 has the highest value of 17.084, which indicates that it has the greatest influence on the IFSS value. At level 3, the type of thermoplastic used was polycarbonate (PC). The use of a thermoplastic matrix with PC increases the electrostatic attracting force, increasing the compatibility between the carbon fiber interface and polycarbonate matrix, thus increasing the IFSS value (Yao et al., 2017).

From the fiber treatment point of view, level 3 has the highest value of 17.259, which indicates that it has the greatest influence on the IFSS. At level 3, the type of fiber treatment used was γ-aminopropyl triethoxysilane (APTS). Surface treatment of fiber using APTS can strengthen the chemical activity on the surface of the fiber, thereby increasing the roughness of the fiber surface and enlarging the fiber surface area (Zhang et al., 2004).

The main effect of both factors potentially influences the result, thereby producing the best combination. Table 6 shows the best combination of factors that potentially result in higher impregnation quality.

Table 6. The best factor combination

Material	*Polycarbonate* (PC)
Fiber treatment	*γ-aminopropyl triethoxysilane* (APTS)

Based on the research findings, it is known that the combination of factors resulting in the highest IFSS is a composite with a PC matrix reinforced by carbon fibers treated with a silane coupling agent..

Analysis of Variance (ANOVA)

ANOVA was used to determine the significance of the overall effect of the independent variables (factors) on the dependent variable (impregnation quality) (Pareek & Bhamniya, 2013; Zheng et al., 2017). It assesses whether there are significant differences in means among the different groups or levels of the independent variables. The calculation results of ANOVA can be seen in Table 7. The calculation included degrees of freedom (Df), a sequential sum of squares (Seq SS), an adjusted sum of squares (Adj SS), an adjusted mean square (Adj MS), an F-statistic from the adjusted mean square, and percentage contribution (p%).

Table 7. ANOVA calculation results

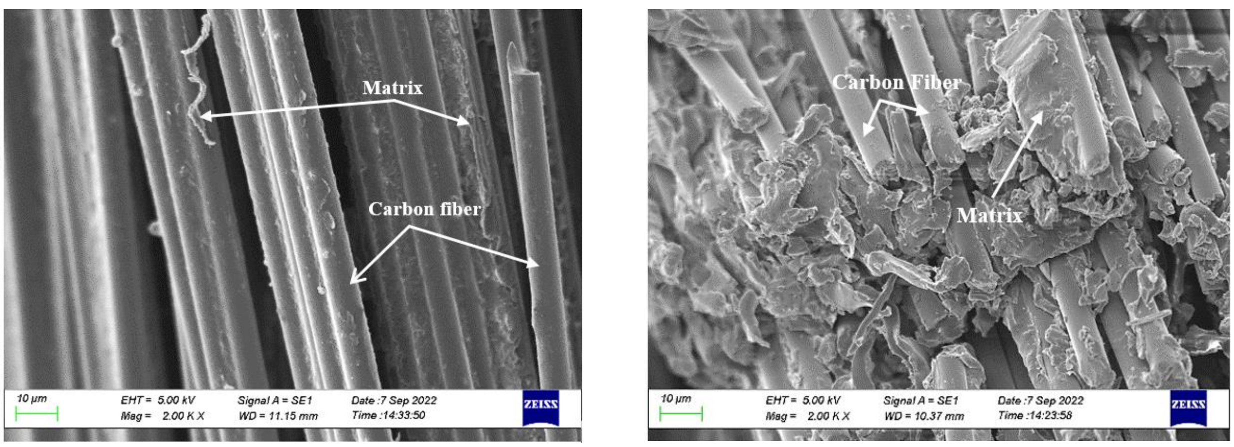
Factors	Df	Sq	Adj SS	Adj MS	F	p%
Thermoplastic type	2	2,3845	2,3845	1.1922	0.552	61,3
Fiber treatment	2	5,0521	5,0521	2.526	1.150	39,7
Error	4	8,6264	8,6264	2.1566	-	-

Total	8	16,213	-	-	-

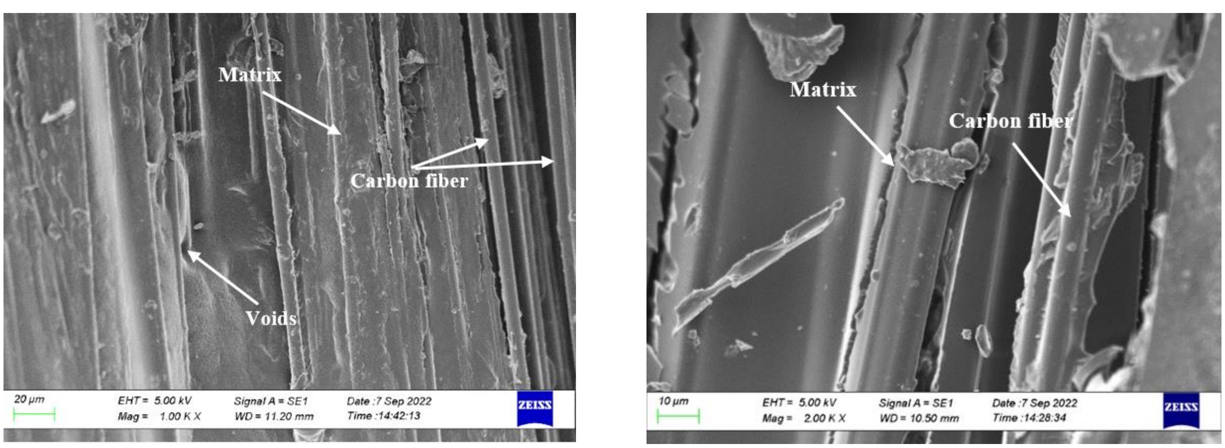
The IFSS is greatly influenced by the type of thermoplastic material, while the type of fiber treatment shows a lower contribution towards IFSS. The type of thermoplastic material contributes 61.3% and the type of treatment contributes 39.7% to the value of IFSS.

Morphological observations

Morphological changes in impregnation quality due to process variables can be observed using a Scanning Electron Microscope (SEM). SEM images in Figure 2 show specimens that have undergone tensile testing where the fibers detached from the matrix. Figure 2.A is a micrograph of a specimen resulting from the combination of HDPE thermoplastic and fiber treatment using heat and cryogenic treatment, resulting in the lowest IFSS value. The micrograph shows that the fiber has a smooth surface, and there is minimal matrix attached to the fiber surface, indicating poor interface strength between the fiber and matrix, resulting in an IFSS of only 5.02 MPa. Figure 2.B shows a combination of specimens with PC thermoplastic matrix and fiber treatment using heat treatment and cryogenic treatment. The micrograph shows that many matrices adhere to the fiber. However, many fibers broke, resulting in an IFSS value of only 5.56 MPa. Figure 2.C shows a micrograph of a specimen resulting from the combination of HDPE thermoplastic and APTS fiber treatment. Impregnation quality improves, as shown by increased adhesion between the fiber and matrix, with more matrix attached to the fiber. However, this combination only resulted in an IFSS value of 8.18 MPa. Figure 2.D is a combination of a PC matrix specimen and fiber treatment using APTS. The micrograph shows that many matrices adhere to the fiber, resulting in a drastic increase in adhesion between the fiber and matrix, leading to a high IFSS value of 10.24 MPa.



(A) (B)



(C) (D)

Figure 2. The SEM results: (A) HDPE matrix and fiber treatment with heat followed by cryogenic treatment, (B) PC matrix and fiber treatment with heat followed by cryogenic treatment, (C) HDPE matrix and APTS fiber treatment, (D) PC matrix, and APTS fiber treatment.

CONCLUSIONS

Based on the results of this study, it can be concluded that the type of thermoplastic material used, and the treatment of the fiber have a significant impact on the interfacial shear strength of the composite. The SEM images obtained from the tested specimens further confirm this conclusion, showing that the quality of impregnation and adhesion between the fiber and matrix plays a critical role in determining the IFSS values.

These findings have important implications for the development of high-performance thermoplastic composites for various applications, such as aerospace, automotive, and sporting goods. By carefully selecting the appropriate thermoplastic matrix and fiber treatment methods, manufacturers can achieve significantly improved mechanical properties and greater durability of the composite materials.

Furthermore, the results of this study highlight the value of using advanced analytical techniques such as SEM to gain insights into the microstructure and performance of composite materials. By combining SEM analysis with mechanical testing, researchers and manufacturers can develop a more comprehensive understanding of the factors that contribute to the strength and durability of thermoplastic composites, leading to the development of more reliable and high-performance materials.

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