Enhanced thermo - mechanical performance of TiB_2-reinforced CF/PEEK composites processed by high-temperature compression moulding

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Abstract. Carbon fiber reinforced poly-ether-ether-ketone (CF/PEEK) composites were fabricated using a high-temperature compression moulding process, and the influence of titanium diboride (TiB_2) nanoparticles (0–4 wt%) on their thermo-mechanical performance was evaluated. The process enabled effective filler dispersion and consolidation at elevated moulding temperatures (350–370 °C). Compressive strength tests performed on CF/PEEK specimens with 2 wt% TiB_2 reinforcement revealed maximum values of 201.5 MPa at 370 °C. The addition of TiB_2 primarily enhanced hardness and thermal behaviour. Rockwell hardness improved slightly with temperature and the values at different combinations (0-4wt%) TiB_2 reinforcement were discussed. SEM analysis confirmed uniform TiB_2 distribution and stronger fibre–matrix–filler bonding, while microstructural voids remained a limiting factor. Thermal studies showed excellent stability, with TGA indicating degradation onset above 550 °C and 65% char residue at 800 °C. DSC confirmed the semi-crystalline nature as both Tg (288 °C) and Tm (345 °C) were recorded, thus confirming the fact that stable crystallization of the hybrid composite has occurred. Overall, the findings prove that compression moulding at high temperature enhances CF/PEEK densification and bonding, besides promoting improvement in hardness and thermal resistance due to the presence of TiB_2. In case there is some further process optimization to reduce porosity, then TiB_2-reinforced CF/PEEK composites show strong prospects as lightweight, high-performance material for aerospace, automotive, and high-temperature structural applications.

1 Introduction

Being a tough, high-performance thermoplastic itself, PEEK exhibits enormous enhancement in its properties when carbon fibres are added in large quantities pertaining to Tensile Strength, Modulus (Stiffness), Thermal Conductivity, Dimensional Stability, Wear Resistance, Creep Resistance, Electrical Conductivity. CF/PEEK is a high-performance composite material whereby carbon fibers are embedded in the PEEK matrix. It is a semi-

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crystalline, thermoplastic polymer that demonstrates exceptional mechanical strength combined with chemical tolerance and thermal resistance. When reinforced with carbon fibers, its structural and functional properties are significantly enhanced, making CF/PEEK one of the most advanced engineering materials available. In the present study the composition consists of matrix material as 450CA30 carbon fiber reinforced. The yield of carbon fiber in the composite is 30% by weight. Short-fiber type carbon fibers are used. The general characteristics of titanium diboride (TiB_2) make it one of the more intriguing materials, due to which its value in industrial application is high. Whenever nano powder of titanium diboride (TiB_2) is incorporated into CF/PEEK composites, it imparts considerable improvements in both mechanical and functional properties. With the help of processes like Hot Compression moulding, there is the possibility of uniform dispersion in the polymer matrix.

1.1 Research gap and novelty

While many works have focused on the study of CF/PEEK composites with SiC, Al_2O_3, or CNT fillers, very few works have been reported regarding the understanding of the incorporation of TiB_2 nanoparticles within the CF/PEEK matrix under high-temperature compression moulding. This work will fulfill that lacuna by exploring systematically the TiB_2 reinforcement on the thermo-mechanical and thermal stability aspects of CF/PEEK composites.

High-performance polymer composites have emerged as critical materials in aerospace, biomedical, and automotive sectors owing to their excellent mechanical properties combined with their chemical resistance and light weight [1-2]. Among these, CF/PEEK, especially the 450CA30 grade, has shown excellent structural integrity, stiffness, and thermal stability [3-4]. Therefore, CF/PEEK is suitable for structural components, fasteners, and tribological applications in hostile environments [5,1]. Addition of nano-fillers has been found to enhance mechanical and thermal performance of PEEK-based composites, increasing their wear resistance, hardness, toughness, and thermal stability [6-8].

Among several fillers, TiB_2 is a hard material with superb thermal and electrical conductivity, along with excellent tribological properties. Although graphene oxide-, ZrO_2-, MoS_2-, and nano-silica-reinforced CF/PEEK composites have gained significant attention in previous works [6-7, 9], the synergistic role of CF and TiB_2 nano-fillers in a PEEK matrix remains unexplored. This indicates a research gap regarding the design of multi-functional polymer composites to achieve both mechanical reinforcement and improved thermal/tribological properties.

A big challenge in the fabrication of CF/PEEK composites is the uniform densification and dispersion of fillers with retention of microstructural integrity. Injection moulding, though suitable for mass production, often leads to fiber breakage, anisotropic fiber orientation, and high residual stresses, compromising mechanical performance [10]. Alternative processing techniques, such as hot pressing or compression moulding, offer better retention of fibers, lower internal stresses, and higher filler loading capability, though at the cost of reduced production rates [11-12]. Multi-scale reinforcement studies and processing under high-shear melt demonstrate that controlled compounding followed by hot pressing can achieve maximum synergy between fiber-nano filler, thereby improving tensile strength, hardness, and thermal stability [13-15].

In the present study, TiB_2 nano-filler reinforced CF/PEEK composites are fabricated by a [melt compounding + hot pressing/injection moulding]. This was characterized for mechanical properties by tensile and flexural tests, tribological properties by a wear test, thermal properties by TGA and DSC analyses, and microstructure by SEM analysis. The present work discusses the effect of incorporation of TiB_2 on the performance of CF/PEEK

composites, provides an insight into the development of lightweight, improved performance polymer composites for aerospace, automotive, biomedical, energy, industrial and electronic applications that meet the demand for metal-like performance at reduced weight [3,6].

Previous studies have focused on SiC, Al_2O_3, or CNT fillers, but TiB_2 reinforcement remains underexplored. This work addresses that gap by analyzing its effect on CF/PEEK's thermo-mechanical response. Also, addresses that gap by systematically examining the influence of TiB_2 nanoparticles (0–4 wt%) on the mechanical and thermal behavior of CF/PEEK composites processed at elevated temperatures (350–370 °C). The work aims to correlate process temperature with property enhancement and to demonstrate the feasibility of TiB_2 as an effective nano-reinforcement for next-generation high-performance polymer composites.

2 Materials and methodology

The base matrix material employed in this study is 450CA30 CF/PEEK. It is a high-performance thermoplastic composite consisting of polyether ether ketone (PEEK) reinforced with 30 wt% short carbon fibers. PEEK is an aromatic, semi-crystalline polymer well known for its excellent mechanical strength, thermal resistance (glass transition temperature ~143 °C, melting temperature ~343 °C), chemical resistance, and wear resistance. The combination of 30% carbon fiber improves its stiffness, tensile strength, and dimensional stability while maintaining relatively low density, makes it extremely suitable for aerospace, automotive, and biomedical applications where lightweight and durability are critical.

In addition, titanium diboride (TiB_2) nanoparticles were added as secondary fillers in varying concentrations (0–4 wt%) to improve the performance of the CF/PEEK composite further. TiB_2 is a ceramic material with a high melting point (~ 3225 °C), high hardness, and elastic modulus, with good electrical conductivity and excellent chemical resistance. In the case of reinforcement by dispersion within the polymer matrix, nanoparticles of TiB_2 act to improve load transfer and inhibit polymer chain mobility, with enhanced interfacial bonding to carbon fibers. These characteristics contribute to improvements in mechanical strength, thermal stability, and tribological performance of the hybrid nanocomposite. Thus, the 450CA30 CF/PEEK + TiB_2 (0–4 wt%) hybrid nanocomposite combines the toughness and thermal resistance of PEEK, the stiffness of carbon fibers, and the hardness and stability of TiB_2 nanoparticles, offering a synergistic enhancement of structural and functional properties suitable for advanced engineering applications. The proper mixing of CF/PEEK with TiB_2 in 4 different proportions is done by using (Plastograph) bra blender at a constant speed and mixing time at a temperature of 340 °C. The four different combinations prepared at different parameters are tabulated below in **Table 1.** mixing ratios of the TiB_2 particles and CF-PEEK.

Table 1. Mixing ratios of the TiB_2 particles and CF-PEEK.

Sample No	CF-PEEK Grade	Wt. of CF/PEEK	%Wt of TiB_2	Wt. of TiB_2	Temp	Speed	Time
1	30% Carbon reinforced PEEK	90 gms	0 %	0.0 gms	340°C	46 rpm	20 mins
2		90 gms	1 %	0.5 gms	340°C	46 rpm	20 mins
3		90 gms	2 %	1.0 gms	340°C	46 rpm	20 mins
4		90 gms	3 %	1.5 gms	340°C	46 rpm	20 mins
5		90 gms	4 %	1.6 gms	340°C	46rpm	20 mins

2.1 Methodology

The following Figure 1 Procedure steps of sample preparation show the workflow or methodology of the process. First, we select the matrix material as Victrex_TDS_450CA30 CF/PEEK, which is a high-performance thermoplastic material, 30% carbon fibre reinforced Poly-Ether-Ether-Ketone, semi crystalline, granules for injection moulding and extrusion, standard flow, FDA food contact compliant, colour black. These granules are then mixed with TiB_2 in different proportions (0 - 4%wt) as shown in the Table 1. mixing ratios of the TiB_2 particles and CF-PEEK by using a bra-blender (plastograph).

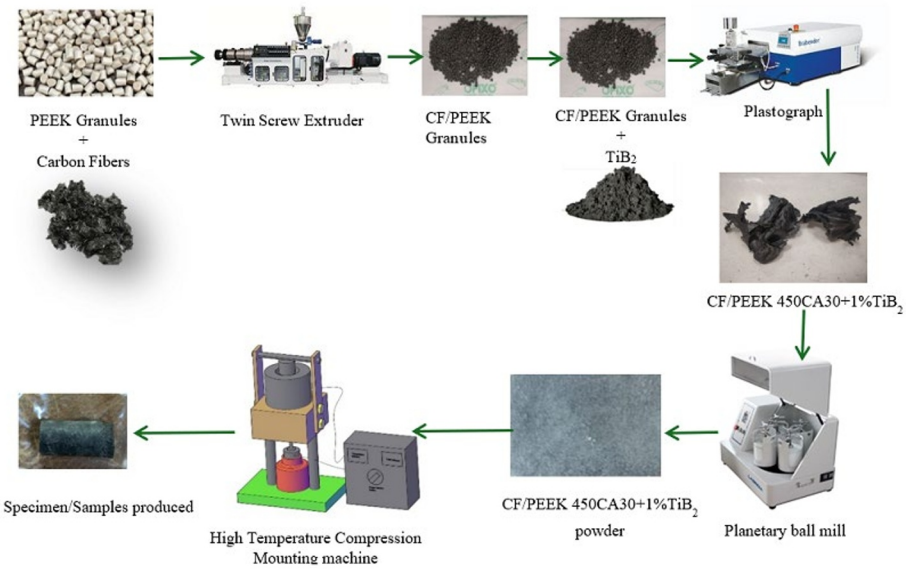

Fig. 1. Procedure steps of sample preparation.

The mixture is blended for a specific amount of time under predetermined conditions, such as temperature, speed, and pressure, with in the Plastograph. After the blending is complete, the Plastograph stops automatically, and the blended material is removed manually from the mixing screws. The blended material is shredded into smaller pieces or powder (0.5-1mm grain size) by using planetary ball mills. Shredding helps in preparing the material for molding or any other subsequent process that requires uniform particle size. The shredded composite is processed in a customised compression moulding machine shown in Figure 2. Schematic Diagram of Compression moulding machine & Table 2. list of parts in compression moulding machine to produce testing samples at different conditions which are listed below in Table 3. This process involves subjecting the material to heat and pressure to shape it into thin, standardized specimens for tensile & compression testing.

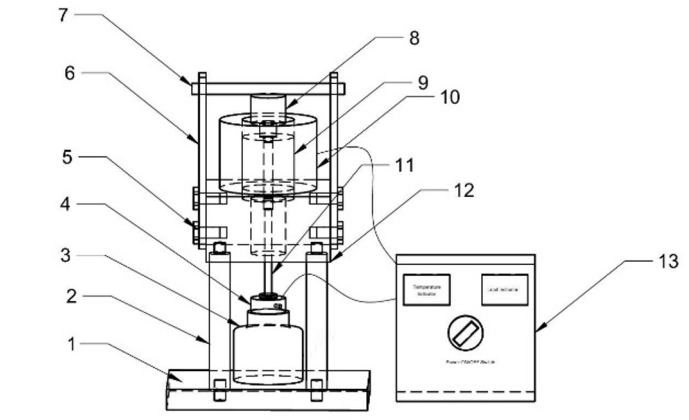

Fig. 2. Schematic Diagram of Compression moulding machine.

Table 2. List of Parts in compression moulding machines.

Part No	Name of the Part	Quantity	Part Material
1	Base Plate	1	MS
2	Supporting Rods	2	Steel
3	Hydraulic Jack	1	Iron
4	Load Cell	1	Steel
5	Hexagonal Headed Bolts (M16)	4	MS
6	Frame	2	MS
7	Top Holding Rod	1	Steel
8	Top Die cavity Holder	1	EN 30
9	Die	1	EN 30
10	Heater	1	Ceramic
11	Plunger	1	EN 30
12	Heater Supporting Block	1	Cast Iron
13	Power Unit	1	Iron

The test sample specimens are prepared using lab developed high temperature compression moulding machine. The procedure begins with the powdered nano composite (CF/PEEK +TiB_2) material is weight measured to the required quantity for example to prepare a compression test sample of ASTM D 695 standards we require a quantity of 5gms nano composite powder as shown in Figure 3. cylindrical specimen dimensions of ASTM D 695. The measured powdered is then poured into the die cavity from the top opening. Close the cavity by a top die cavity holder and then pack the holder with a holding rod to keep it tight when the load is applied. The load is applied on to the plunger by a 2-ton capacity hydraulic jack. Which is fixed on the bottom frame. Once the material is packed in the die cavity the power supply is given to the heater and set the temperature to 350 °C. To reach a temperature of 350 °C from room temperature it takes 3-4 minutes time. Once the temperature reached to 350 °C hold the temperature for 15 minutes and latter apply load on the material by hydraulic jack. The amount of load applied is displayed in the indicator. Load is determined by a load cell (two-ton capacity) which is placed between the plunger and

hydraulic jack. After load is applied leave it for 10 minutes setting time. Next take the sample out from the cavity by pushing the plunges upwards using hydraulic jack. The specimens were fabricated at three moulding temperatures as described in Table 3, Working Parameters.

Table 3. Working parameters.

S. No	Material	Temperature (°C)	Holding Time (Minutes)	Load (Kilograms)	Pressure (kg/mm^2)
1	CF/PEEK + 0%wt TiB_2	350	15 min	50	3.819
2		360			
3		370			
4	CF/PEEK + 1%wt TiB_2	350			
5		360			
6		370			
7	CF/PEEK + 2%wt TiB_2	350			
8		360			
9		370			
10	CF/PEEK + 3%wt TiB_2	350			
11		360			
12		370			
13	CF/PEEK + 4%wt TiB_2	350			
14		360			
15		370			

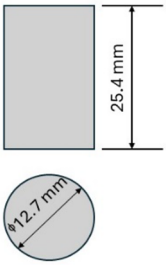

Fig. 3. Cylindrical specimen dimensions of ASTM D 695 standards.

2.2 Testings

The test specimens which are fabricated at different parameters exhibits different properties. Here are few samples of CF/PEEK+ 2%wt TiB_2 fabricated at temperatures of 350°C, 360°C & 370°C shown in Figure 4. (a, b &c) Hardness test specimens of CF/PEEK+2%wt TiB_2 at a temperature of 350°C, 360°C & 370°C.

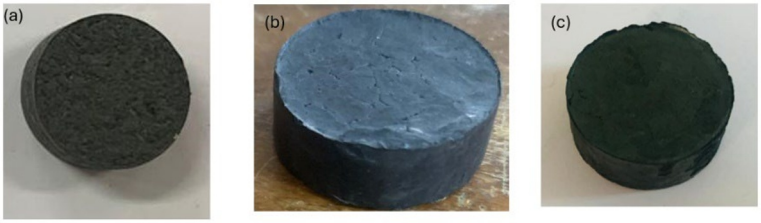

Fig. 4. Hardness test specimens of CF/PEEK+2%wt TiB_2 at a temperature of (a) 350°C, (b) 360°C, and (c) 370°C.

Hardness

Rockwell M hardness is commonly employed to assess the hardness of materials, like CF/PEEK. The addition of carbon fibers and TiB_2 nano powder to PEEK enhances its mechanical strength and stiffness, impacting its overall hardness. Typically, exhibits a higher Rockwell M hardness compared to unfilled PEEK due to the strengthing effect of carbon fibers and TiB_2 as shown in Table 4. Rockwell Hardness test results.

Table 4. Rockwell Hardness test results.

S. No.	Sample Description	Temperature	Rockwell Hardness		
			Trial	Value	Average
1	98% CF/PEEK (450CA30) + 2% TiB2	350 °C	Trial 1	90.5	90.68
			Trial 2	91.6	
			Trial 3	90.3	
			Trial 4	91.6	
			Trial 5	90.6	
2	98% CF/PEEK (450CA30) + 2% TiB2	360 °C	Trial 1	90.5	91.24
			Trial 2	91.6	
			Trial 3	90.8	
			Trial 4	92	
			Trial 5	91.3	
3	98% CF/PEEK (450CA30) + 2% TiB2	370 °C	Trial 1	91.8	92.06
			Trial 2	92.6	
			Trial 3	91.7	
			Trial 4	92.6	
			Trial 5	91.7	

Compressive strength

The compressive strength of pure CF/PEEK is very important because it directly reflects the material's capacity to withstand high compressive stresses without failure, deformation, or micro-buckling. A strong compressive property ensures no relaxation or creep, which is common in pure polymers. Screws in aerospace/automotive structures face vibrations, cyclic loads, and shocks. High compressive strength helps resist fatigue-induced thread failure. Since PEEK is already known for its high strength, toughness, and chemical resistance, and carbon fiber reinforcement (CF) significantly improves stiffness, while TiB_2 nanoparticles enhance hardness, thermal stability, and load transfer, their combination leads to a high-performance composite with superior compressive behaviour. The test samples are fabricated

to cylindrical specimens of ASTM D 695 standards as shown in Figure 5. (a, b & c) Test specimens of CF/PEEK at 3 different moulding temperatures of 350°C, 360°C & 370°C.

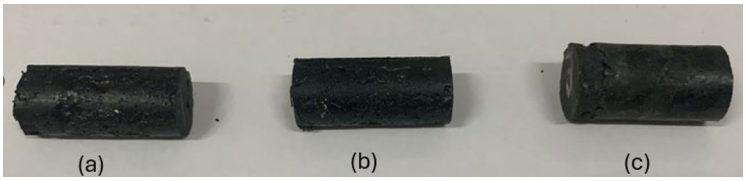

Fig. 5. Test specimens of CF/PEEK at 3 different moulding temperatures of (a) 350°C, (b) 360°C, and (c) 370°C.

Microstructure observation

The microstructure of the CF/PEEK with 2%wt TiB_2 sample is observed using field emission scanning electron microscope (Zeiss Gemini 500 SEM, Germany). The samples which were tested in Rockwell Hardness machine were selected. The SEM samples were sputtered with gold for 300 s to enhance the conductivity of the fracture surface before the test. The SEM image of hybrid composite of CF/PEEK with 2%wt TiB_2 fabricated at 370 °C is shown in **Figure 7.** SEM image of CF/PEEK with 2 %wt TiB_2 fabricated at 370 °C.

3 Results & discussions

3.1 Hardness

The Rockwell M hardness values shown in Figure 6. Hardness values of compression-moulded CF/PEEK composites with 2 wt% TiB_2 fabricated at 350 °C, 360 °C, and 370 °C. The results indicate a progressive increase from 90.68 HRM at 350 °C to 92.06 HRM at 370 °C. According to the explained improvement, at higher processing temperatures, enhanced polymer chain mobility, better impregnation of carbon fibers, and more uniform distribution of TiB_2 particles are realized. This combined effect strengthened interfacing bonding, restrained localized deformation, and resulted in improved hardness. These results provided clues that optimizing moulding temperature within the thermal stability range of PEEK will be most beneficial for maximizing its mechanical performance in the composite.

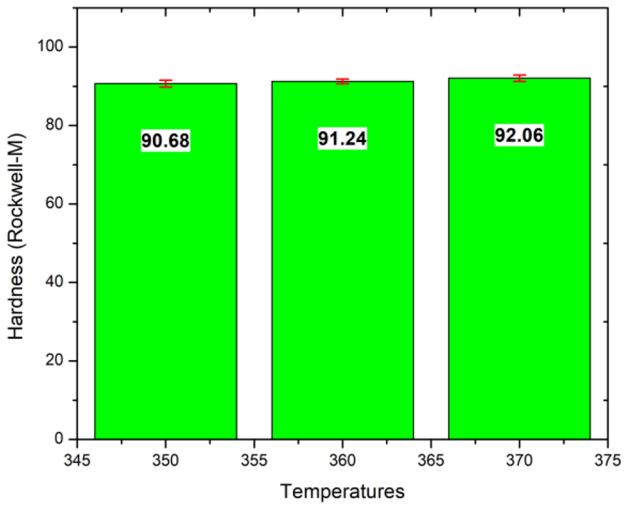

Fig. 6. Hardness values of CF/PEEK composites with 2 wt% TiB_2 fabricated at 350 °C, 360 °C, and 370 °C.

3.2 Compressive strength

Table 5. Compression strength test results of CF/PEEK with 2% TiB2 fabricated at different temperatures provides the compressive strength for the composites. It can be seen that the trend shows that with increasing temperature, the compressive strength increases from 180.8 MPa for 350 °C to 201.5 MPa at 370 °C. This is primarily due to better mobility of the polymer chains, improved impregnation of the carbon fibers, and a more homogeneous distribution of TiB_2 particles at increased moulding temperature. Such conditions offer a decrease in void formation along with improvement in interfacial bonding, which provides better load-carrying capability. Therefore, the highest compressive performance has been obtained by processing at 370 °C. Increased mechanical properties with respect to increased processing temperature can be explained by better matrix consolidation and interfacial bonding between carbon fibers and PEEK matrix. At 370 °C, the polymer viscosity decreases sufficiently to facilitate better wetting of fibers and dispersion of fillers, leading to a denser and more homogeneous microstructure. This optimal processing temperature ensures complete melting and interdiffusion of polymer chains without inducing thermal degradation and therefore provides higher hardness and compressive strength compared to the samples moulded at lower temperatures.

Table 5. Compression strength test results of CF/PEEK with 2% TiB_2 fabricated at different temperatures.

S. No.	Sample Description	Temperature	Compressive Strength	
			Failure load KN	Value MPa
1	98% CF/PEEK (450CA30) + 2% TiB2	350 °C	21.5	180.8
		360 °C	23.3	193.3
		370 °C	25.6	201.5

3.3. Microstructure study (SEM)

Scanning electron microscope (SEM) analysis Figure 6 SEM image of CF/PEEK with 2 %wt TiB2 fabricated at 370 °C shows the SEM micrograph of compression-moulded CF/PEEK (450CA30) composites reinforced with 2 wt% TiB_2. The image clearly illustrates the distribution of carbon fibers within the PEEK matrix and the presence of TiB_2 particles embedded in the structure. The carbon fibers are well dispersed, forming a reinforcing network that provides strength and stiffness to the composite. TiB_2 particles, highlighted in the circled regions, are uniformly distributed and strongly bonded with the surrounding polymer matrix. Their presence plays a crucial role in restricting polymer chain mobility, improving load transfer, and enhancing resistance to localized deformation. The absence of significant voids or pull-out regions indicates good inter facial adhesion between the carbon fibers, TiB_2 particles, and the PEEK matrix, which is consistent with the observed improvements in hardness and compressive strength. This micro-structural evidence confirms the beneficial role of TiB_2 reinforcement and optimized processing temperature in improving composite performance. The Thermogravimetric Analysis (TGA) of CF/PEEK Figure 8. (a) TGA Curve shows the thermal stability of the CF/PEEK composite as a function of temperature. The material exhibits and decomposition behavior of the composite. The sample remains stable up to approximately 500 °C with negligible weight loss, indicating excellent thermal resistance. A major weight loss occurs between 500–600 °C, corresponding to the decomposition of the PEEK matrix. At around 600 °C, about 74% weight is retained, which is mainly due to the presence of carbon fibers and thermally stable residues. The residual weight beyond 800 °C (~66%) confirms the high char yield and superior thermal stability of carbon fiber–reinforced PEEK. The degradation onset (≈550 °C) exceeds that of SiC-filled CF/PEEK (≈530 °C) and matches ZrO_2-reinforced systems (≈545 °C), confirming improved thermal stability due to TiB_2 addition.The Differential Scanning Calorimetry Figure 8 (b) DSC curve shows the melting and crystallization behavior of the composite. The glass transition temperature (Tg) is observed around 287 °C, while the melting peak (Tm) appears at approximately 341–355 °C, consistent with typical PEEK behavior. These values confirm that the processing temperatures used, from 350 to 370 °C, fall within the suitable window for effective consolidation without inducing degradation.

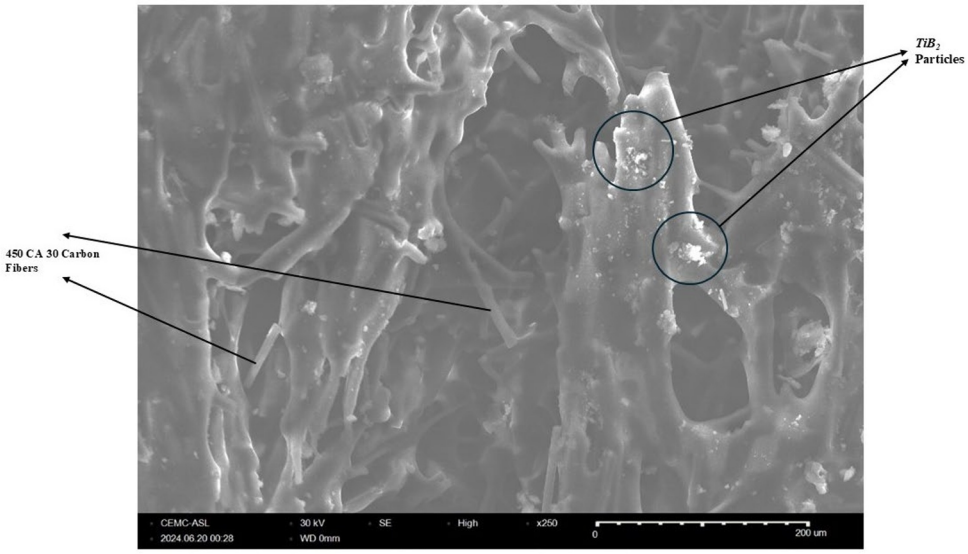

Fig. 7. SEM image of CF/PEEK with 2 %wt TiB_2 fabricated at 370 °C.

3.4 Thermal analysis

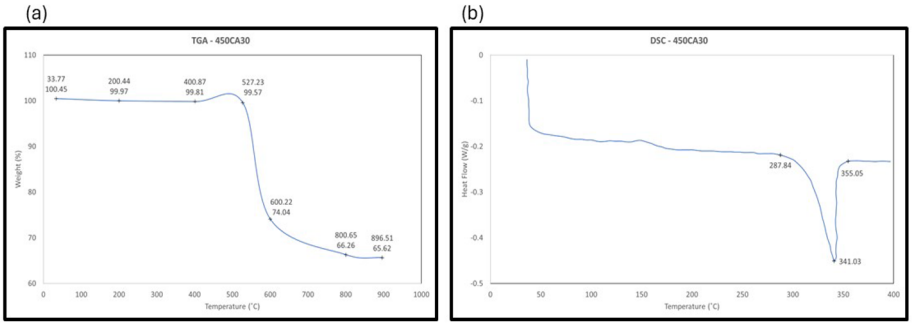

Fig. 8. (a) TGA Curve, and (b) DSC Curve.

4 Conclusions

This study investigated the effect of TiB_2 nano particles on the thermo-mechanical performance of CF/PEEK composites processed by high-temperature compression moulding. Based on the results, the following conclusions are drawn:

1. CF/PEEK (450CA30) composites reinforced with TiB_2 nano particles were successfully fabricated using a high-temperature compression moulding process at 370 °C.
2. Rockwell M hardness increased slightly with processing temperature, achieving the highest hardness of 92.06 HRM at 370 °C for 2 wt% TiB_2 reinforcement CF/PEEK, demonstrating improved inter facial bonding and restricted localized deformation.
3. Compressive strength for 2 wt% TiB_2 reinforcement CF/PEEK improved significantly with temperature, increasing from 180.8 MPa at 350 °C to 201.5 MPa at 370 °C,

indicating better densification, fiber wetting, and load transfer at higher moulding temperatures.

4. SEM analysis confirmed uniform dispersion of TiB_2 particles and strong bonding between fibers, matrix, and nanoparticles, while minor porosity was observed as a limiting factor.
5. Thermal analysis demonstrated excellent stability, with TGA showing degradation onset above 550 °C and ~66% char yield at 800 °C, while DSC confirmed semi-crystalline behaviour with Tg ≈ 287 °C and Tm ≈ 345 °C, validating safe processing within the studied temperature window.
6. Overall, the incorporation of TiB_2 nano particles improved hardness and thermal resistance, while compression moulding at elevated temperature enhanced densification and compressive performance. With further process optimization to minimize porosity, TiB_2-reinforced CF/PEEK composites show strong potential for lightweight, high-performance applications in aerospace, automotive, and high-temperature structural components.

Limitations and Future Work : Although the present results demonstrate significant improvements in hardness and compressive strength, minor porosity still limits performance. Future work will focus on optimizing moulding pressure and employing vacuum-assisted consolidation to further reduce voids and enhance densification.

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