

Effect of temperature and atmosphere on the fracture toughness and failure mechanisms of two-dimensional plain-woven SiC_f/SiC composites: Experiments and modeling

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Received October 11, 2024; accepted November 25, 2024; published online February 14, 2025

Ceramic matrix composites have broad application prospects in the aerospace field due to their high temperature resistance and oxidation resistance. The effect of temperature and environment atmosphere on the fracture toughness and failure mechanisms of two-dimensional plain-woven SiC_f/SiC composites was investigated. The results show that they exhibit pseudo-plastic deformation behavior at different temperatures. The fracture toughness is as high as 48 MPa m^{1/2} at room temperature, and gradually decreases with rising temperature. The difference in fracture toughness between argon and air initially increases and then decreases with rising temperature. Furthermore, the high-temperature failure mechanisms of these composites were analyzed through macro and micro analysis. Based on this, a physic-based temperature-dependent fracture toughness model considering matrix toughness, plastic power, fiber pull-out, and residual thermal stress was developed for fiber-reinforced ceramic matrix composites. The model has been well validated by experimental results. An analysis of influencing factors regarding the evolution of fracture toughness was conducted by the proposed model. This work contributes to a better understanding of the mechanical performance evolution and failure mechanisms of ceramic matrix composites under multi-field coupling conditions, thereby promoting their applications.

SiC_f/SiC composites, Fracture toughness, Analytical modelling, Failure mechanisms, High-temperature

Citation: Y. Deng, Y. Hao, H. Wang, W. Li, Q. Qin, B. Pan, and C. Zhang, Effect of temperature and atmosphere on the fracture toughness and failure mechanisms of two-dimensional plain-woven SiC_f/SiC composites: Experiments and modeling, Acta Mech. Sin. 41, 124333 (2025), <https://doi.org/10.1007/s10409-024-24333-x>

1. Introduction

The service temperature of thermal-structure material in hypersonic vehicles and aero-engines has been steadily increasing in recent years. As the primary structural materials for the hot-end of the engine, nickel-based superalloys are

confronted with significant challenges. Due to their low density, excellent high temperature resistance and oxidation resistance, as well as impressive high-temperature mechanical properties, ceramic matrix composites have been receiving increasing research attention in the aerospace field [1-3]. In particular, SiC_f/SiC composites, well-known for their high specific strength and non-brittle fracture characteristics, have become a popular choice for replacing nickel-based superalloys as hot-end structural materials in

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Executive Editor: Yuli Chen

engines [4].

The service environment within aircraft engines often exposes SiC_f/SiC composites to high-temperature conditions. Therefore, it is essential to investigate the impact of temperature and environmental atmosphere on the mechanical properties and failure mechanisms of SiC_f/SiC composites [5]. This research will contribute to a more comprehensive understanding of the properties of these composites at elevated temperatures, as well as facilitate their life prediction and service safety assessment. Because of the intrinsic brittleness of ceramic matrix, SiC_f/SiC composites show relatively low fracture toughness compared with alloys, thereby somewhat limiting their engineering applications [4]. To this end, fracture toughness is considered to be one of the most critical indices for high-temperature mechanical performance [6,7].

Currently, there are many studies devoted to the effects of environment conditions and preparation technologies on the mechanical properties and damage evolution of SiC_f/SiC composites [8-19]. The impact of heat-treatment temperature on the microstructure and interface properties of SiC_f/SiC composites was studied by Ma et al. [14]. The findings indicated a gradual decrease in interfacial bond strength as the heat treatment temperature increased. Ai et al. [15] conducted systematic mechanical performance tests at 1300 and 1500 °C in an inert atmosphere. The results indicated that the compressive and interlaminar shear strengths increased with temperature increasing, while the flexural strength decreased. Xie and Chen [16] studied the evolution of mechanical properties of SiC_f/SiC composites under vacuum. It was observed that as the temperature increases to 1350 °C, the bending strength remains basically unchanged, while the fracture toughness slightly decreases at 1350 °C owing to the damage of the interface. Wu et al. [17] conducted systematic thermophysical and mechanical performance testing experiments of 3D Hi-Nicalon™/SiC composite from 25 to 1400 °C in vacuum. The research results indicate that the flexural modulus and strength decreased, while the fracture toughness increased with rising temperature. Interphase layer plays a crucial role in toughening fiber-reinforced ceramic matrix composites (FRCMCs) [20,21]. Zhang et al. [21] explored the impact of the interphase thickness on the mechanical properties of SiC_f/SiC composites, and found that the decrease of PyC thickness led to the increase in the interfacial shear stress and strength of the composites. According to the modified interfacial debonding criterion, a novel matrix cracking model for FRCMCs considering the influence of interphase bonding strength was developed by Niu et al. [20], and the mechanism of the non-monotonic effect of interphase thickness was proposed.

Moreover, Hong et al. [18] conducted research on the evolution of microstructure and mechanical properties for

SiC_f/SiC composites in a gas environment. The study revealed that the fracture toughness decreases owing to the interfacial transition. Several studies [22,23] have demonstrated that high-temperature oxidation significantly influences the fracture toughness and interfacial properties of SiC_f/SiC composites. The long-term oxidation behaviors and their impact on the strength of self-healing SiC_f/SiC composites were investigated by Ji et al. [23], the composites exhibited a high retention rate in strength, attributed to their good self-healing capacity. Previous studies have contributed to a more comprehensive understanding of the evolution of damage mechanisms and mechanical properties of ceramic matrix composites at elevated temperatures. However, there is a lack of systematic research on the impact of temperature and environment atmosphere on the fracture toughness and corresponding damage mechanisms of two-dimensional plain-woven SiC_f/SiC composites. This deficiency hinders the effective support the application assessment and performance evaluation of these composites.

Establishing theoretical models to investigate the high-temperature properties of thermal protection materials is also an important area of interest for researchers. In the aspect of theory, several models of fracture toughness for ceramic matrix composites have been developed at ambient temperature [24-27]. Song et al. [24] proposed a theoretical toughening model that takes into account fiber bridging and fiber pullout, which can be utilized for the calculation of the R-curve of a propagating crack. Li and Zhou [25] developed a semi-empirical model of fracture toughness for brittle two-phase ceramic composites, taking into account microstructure, interfacial bonding characteristics, and constituent properties. In addition, Luh and Evans [26] proposed a model for describing the contribution of fiber pull-out to fracture toughness. The influence of thermal residual stress on fracture toughness of FRCMCs has been assessed by Skripnyak and Skripnyak [27]. The above-mentioned models focus on the theoretical study for the fracture toughness of FRCMCs and major influencing factors at ambient temperature. In the case of high temperatures, several studies have also been carried out. For particle-reinforced ceramic matrix composites, Wang et al. [28] proposed a theoretical prediction model considering the influence of plastic power on fracture toughness, and good consistency was achieved with the experimental results. Moreover, for whisker-reinforced ceramic matrix composites, a prediction model of fracture toughness suitable for high temperature was proposed by Shao et al. [29], the contributions of fracture toughness of matrix, residual thermal stress, crack deflection and bridging were all included. However, there has been limited research dedicated to the theoretical study of high-temperature fracture toughness of FRCMCs.

This study aims to investigate the high-temperature fracture toughness and failure mechanisms of SiC_f/SiC com-

posite experimentally and theoretically. The present work is arranged as follows. In Sect. 2, we briefly describe the experimental method including materials preparation, mechanical testing, and macro and micro view. Section 3 introduces the temperature-dependent fracture toughness prediction model of FRCMCs, mainly taking into account of the contribution of ceramic matrix, fiber pull-out toughening, and residual thermal stress. Section 4 shows the results and the discussion of the single edge notched-three-point bending (SENB) tests, high-temperature failure mechanisms, and the model validation and comparison. Section 5 concludes the paper. The results of this work are of great significance for the preparation, service performance evaluation, and high-temperature application of SiC_f/SiC composites.

2. Experiment

2.1 Materials preparation

The two-dimensional plain-woven SiC_f/SiC composites are prepared by chemical vapor infiltration process with Cansas-3 fibers (Fujian Leadasia New Material Co., Ltd.) with their parameter displayed in Table 1. The detailed properties of the composites are displayed in Table 2. The detail preparation process can be referenced in the Ref. [30]. The prepared composite sheet is cut into 3 mm × 6 mm × 30 mm, each of which is cut parallel or perpendicular to the fiber axis. The notch of the specimen was made with a 0.2 mm thick diamond grinding wheel. The notch has a total length of 3 mm and a width of 0.3 mm.

2.2 Experiment details

The fracture toughness of 2D SiC_f/SiC composites sample was measured by the SENB test. The reference test standard is the enterprise standard of Q/AVIC 06185.6 from Aviation

Industry Corporation of China (Test Method for Mechanical Properties of Continuous Fiber Reinforced Ceramic Matrix Composites at Elevated Temperatures-Part 6: Fracture Toughness (K_{IC})). High-temperature tests were performed at 25, 800, 1000, 1200, 1350 and 1500 °C in an inert atmosphere and air environment. As depicted in Fig. 1, the SENB test was carried out using a universal testing machine (force transducer: 0-10 kN) equipped with a high-temperature environment chamber. The maximum temperature heated by the heating furnace is 1650 °C. The heating furnace is equipped with a temperature controller to control the heating temperature, heating rate, and holding time. Internal temperature is measured by platinum rhodium thermocouples. During the tests, the load data are acquired using an analog-to-digital converter. There is a vent on one side of the heating furnace, which is capable of introducing argon gas after being connected to the gas source. When conducting tests in an inert atmosphere, argon gas is pre-introduced into the heating furnace until the experiment is accomplished. It is worth noting that nitrogen is continuously introduced throughout the entire testing process to prevent the sample from coming into contact with the surrounding air. The diagram drawings and in physical

Table 1 General properties of Cansas-III fibers

Fibers	Cansas 3303
Strength (GPa)	3.537
Young's modulus (GPa)	366.6
Density (g cm ⁻³)	2.983
Diameter (μm)	14

Table 2 General properties of the SiC_f/SiC composites

Material parameter	Data
Interface thickness (nm)	300
Fiber content (vol%)	35
Matrix modulus (GPa)	320
Density (g cm ⁻³)	2.6
Porosity	9%-13%

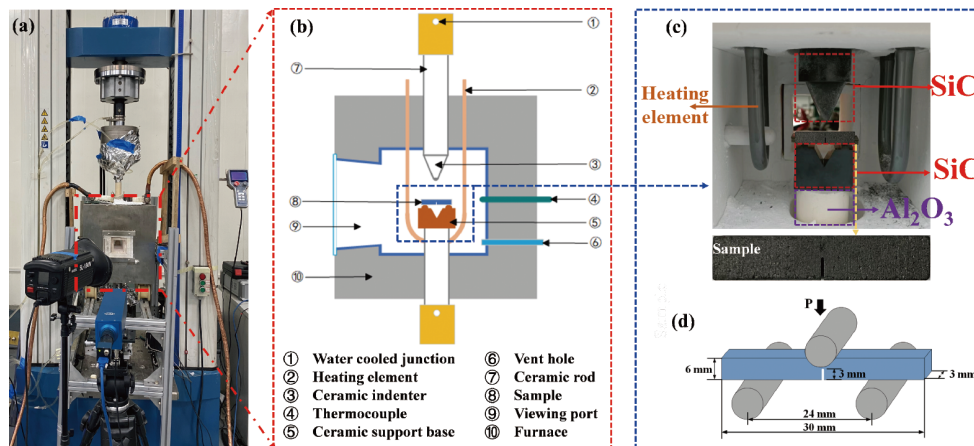


Figure 1 Test equipment. (a) Experimental equipment; (b) schematic diagram of device; (c) physical space of thermal loading system; (d) schematic drawing of the SENB.

space of SENB are shown in Fig. 1(d) and (c). The high-temperature bending fixture was made of high-hardness SiC ceramics. All experiments were conducted at a loading rate of 0.05 mm min^{-1} and a heating rate of $20 \text{ }^{\circ}\text{C min}^{-1}$, with insulation for 30 min. Five samples were tested in each testing condition. The value of fracture toughness K_{IC} is obtained based on the following formula [31,32].

$$K_{IC} = \frac{3PL}{2BH^2} a^{1/2} f(a/H), \quad (1)$$

$$f(a/H) = 1.93 - 3.07(a/H) + 14.53(a/H)^2 - 25.11(a/H)^3 + 25.8(a/H)^4, \quad (2)$$

where H and B denote the thickness and width of the test sample. P denotes the bending load, a is the total length of the flaw, and L denotes the span of the support roller (Fig. 1(d)).

2.3 Characterization

The fracture morphology and microstructure of the specimen were analyzed through a digital microscope (KEYENCE VHX-7000, Japan) and a scanning electron microscope (SEM, SUPRA 55, ZEISS, Germany). X-ray diffractometer (XRD, Brook D8 DISCOVER A25, Germany) was used to study the crystal structure of the SiC_f/SiC sample. The detailed experimental results will be presented in Sect. 4.

3. Theoretical modelling

In this part, the temperature-dependent fracture toughness of FRCMCs is modeled. The model takes into account the contribution of ceramic matrix, fiber pull-out, residual thermal stress, matrix plastic power owing to brittle-ductile transition, as well as their evolution with temperature. The detailed modeling process is given as follows.

3.1 High-temperature prediction model of fracture toughness of ceramic matrix

To accurately predict the fracture toughness of FRCMCs at elevated temperatures, it is essential to include the contribution of the ceramic matrix and its quantitative impact at different temperatures. In our previous study [33,34], we developed a physic-based temperature-dependent prediction model of fracture toughness for single-phase ceramics

$$K_m(T) = K_m(T_0) \left\{ \frac{1 - \nu^2(T_0)}{1 - \nu^2(T)} \times \frac{E_m(T)}{E_m(T_0)} \left[1 - \frac{1}{\int_{T_0}^{T_m} C_p(T) dT} \int_{T_0}^T C_p(T) dT \right] \right\}^{1/2}, \quad (3)$$

where T_0 and T denote the reference temperature and ambient temperature, respectively. $K_m(T)$ denotes the fracture toughness of ceramic matrix, and $K_m(T_0)$ is the fracture toughness at T_0 . E_m and ν denote the Young's modulus and poisson's ratio, T_m denotes the melting point, and $C_p(T)$ denotes the heat capacity. Moreover, the above model can be simplified [35]

$$K_m(T) = K_m(T_0) \left[\frac{1 - \nu^2(T_0)}{1 - \nu^2(T)} \times \frac{E_m(T)(T_m - T)}{E_m(T_0)(T_m - T_0)} \right]^{1/2}. \quad (4)$$

This model offers a practical and effective approach to predict the fracture toughness of ceramics at elevated temperatures, and the applicability of this model to SiC ceramics has been previously verified by our work [36]. The transition from brittle to ductile behavior in ceramics occurs as the temperature increases. The plastic power starts to impact the fracture toughness of FRCMCs above the brittle-ductile transition temperature. Based on the work of Wang et al. [28], the increase in fracture toughness caused by plastic power above the brittle-ductile transition temperature can be expressed as

$$K_{m2}(T) = K_{m2}(T_1) \left[\frac{1 + \nu(T_1)}{1 + \nu(T)} \times \frac{E_m(T)(T_m - T)}{E_m(T_1)(T_m - T_1)} \right]^{-0.5}, \quad (5)$$

where T_1 denotes the brittle-ductile transition temperature; and $K_{m2}(T_1)$ is the increment of fracture toughness due to the plasticity at temperature T_1 . Therefore, when the temperature is higher than the brittle-ductile transition temperature, the contribution of matrix on fracture toughness of FRCMCs can be calculated by summing Eqs. (4) and (5).

3.2 The contribution of fiber pull-out on fracture toughness

The toughening mechanism of FRCMCs with weak inter-phase is primarily attributed to fiber pull-out and crack deflection. The contribution of reinforced fibers to the fracture toughness of FRCMCs can be calculated by [26]

$$\Delta K_3(T) = L_p(T) [\tau(T) E_f(T) V_f / r]^{1/2}, \quad (6)$$

where $\tau(T)$ is interfacial shear strength, $L_p(T)$ is the median pull-out length, V_f is fiber content, r is fiber radius. The pull-out length of fiber $L_p(T)$ can be measured using microscopic observations of fracture surface of the specimen directly. The temperature-dependent interfacial shear strength $\tau(T)$ is expressed as [37]

$$\tau(T) = \tau(T_0) \left[\frac{E_l(T)}{E_l(T_0)} \times \left(\frac{T_{m1} - T}{T_{m1} - T_0} \right) \right]^{1/2}, \quad (7)$$

where E_l represents the Young's modulus of the interface phase, and T_{m1} represents the lower melting point between

matrix and fiber. The applicability of this model to SiC fiber reinforced ceramic composites was verified in our previous work [37].

3.3 The contribution of residual thermal stress on fracture toughness

As we all know, the thermal residual stress has a remarkable impact on the fracture toughness of FRCMCs. The existence of the local stress (q) owing to thermal expansion mismatch decreases the stress intensity factor ΔK . Taya et al. [38] proposed an analytical model to consider the influence of thermal residual stress on fracture toughness for particulate-reinforced ceramic-matrix composite.

$$\Delta K = 2q\sqrt{\frac{2(\lambda-d)}{\pi}}, \quad (8)$$

where q denotes the local residual thermal stress, λ is interparticle distance, and d denotes the mean size. The above formula is extended and applied to predict the toughening increment owing to residual stress for whisker or chopped fiber reinforced ceramic composites by Silvestroni et al. [39].

Similarly, due to the same toughening mechanism and model applicability conditions, and it is assumed that the continuous fibers are evenly distributed, the contribution of residual thermal stress on fracture toughness of FRCMCs can also be calculated in Ref. [39].

$$\Delta K_4(T) = \frac{-2V_f\sigma_{\text{res}}(T)}{1-V_f}\sqrt{\frac{2(L-d)}{\pi}}, \quad (9)$$

where L is the average distance between fibers, which equals to $r/\sqrt{V_f}$, and d is the average size of reinforced fiber, here equals to $2r$. $\sigma_{\text{res}}(T)$ is the thermal residual stress of matrix, which can be expressed as [40]

$$\sigma_{\text{res}}(T) = A \frac{E_f(T)E_m(T)V_f\phi_t(T)}{E_c(T)(1-\nu_m)} \cdot [\alpha_f(T) - \alpha_m(T)](T - T_{\text{proc}}), \quad (10)$$

where $\phi_t(T) = \left[\frac{0.5[1 + E_c(T)/E_f(T)]}{1 - 0.5(1 - 2\nu)[1 - E_c(T)/E_f(T)]/(1 - \nu)} \right]$, α denotes the thermal expansion coefficient, T_{proc} is the preparation temperature.

3.4 Temperature-dependent fracture toughness model for FRCMCs

Finally, the overall fracture toughness of FRCMCs could be predicted by the sum of matrix toughness and these active toughening terms introduced by plastic power, fiber pull-out, and thermal residual stress. Combining Eqs. (4), (5), (6) and (9), the physic-based temperature-dependent model of fracture toughness for FRCMCs with weak interphase has been obtained

$$\begin{aligned} K(T) &= K_m(T) + K_{m2}(T) + \Delta K_3(T) + \Delta K_4(T) \\ &= K_m(T_0) \left[\frac{E_m(T)}{E_m(T_0)} \frac{(T_m - T)}{(T_m - T_0)} \right]^{1/2} \\ &\quad + K_{m2}(T_1) \left[\frac{E_m(T)}{E_m(T_1)} \frac{(T_m - T)}{(T_m - T_1)} \right]^{-1/2} \\ &\quad + L_p(T) [\tau(T) E_f(T) V_f / r]^{1/2} \\ &\quad - \frac{2V_f\sigma_{\text{res}}(T)}{1-V_f} \left[\frac{2(L-d)}{\pi} \right]^{1/2}, \end{aligned} \quad (11)$$

where $K(T)$ denotes the fracture toughness of FRCMCs at T temperature, $K_m(T_0)$ is the matrix fracture toughness at T_0 , and $K_{m2}(T_1)$ denotes the increment of fracture toughness due to plastic work. The interfacial shear strength $\tau(T)$ and residual thermal stress $\sigma_{\text{res}}(T)$ can be obtained by Eqs. (7) and (10). The Young's modulus of component is available in the literature. As a result, the developed model establishes a quantitative relationship among the fracture toughness of FRCMCs, the basic material parameters of their components, and temperature. It offers a convenient and effective means for analyzing and predicting the high-temperature fracture toughness of FRCMCs.

4. Results and discussion

4.1 High-temperature experimental results

Figure 2(a) and (b) depicts the force-displacement curves of the samples at temperatures ranging from room temperature (RT) to 1500 °C in argon and air, respectively. The corresponding fracture toughness was determined from the load-displacement curves and documented in Fig. 3, the corresponding maximum load from the load-displacement curves in Fig. 2 was used to calculate the fracture toughness. As illustrated, the load of the composite decreases in a stepped manner once reaching the maximum load both in argon and air at different temperatures, which shows typical features of pseudo-plastic fracture. This behavior significantly differs from the traditional brittle fracture mode of single-phase

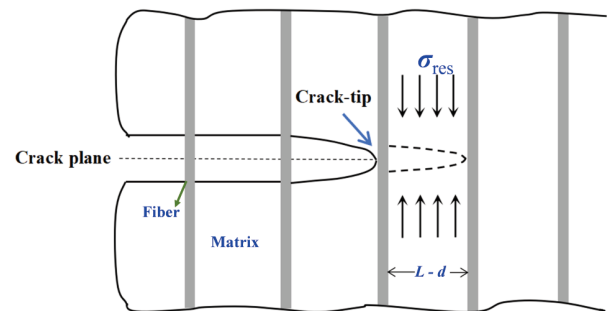


Figure 2 Semi-infinite crack advances, L , in compression region of matrix to tensile region of fiber.

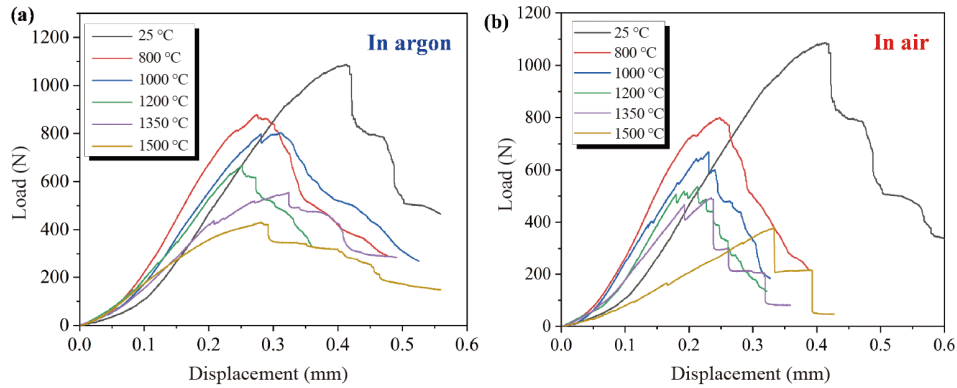


Figure 3 The load-deflection curves of the samples evaluated with the SENB test. (a) In argon; (b) in air.

ceramics, indicating that continuous SiC fibers have a significant toughening effect on ceramics. With the increase of load, the matrix cracks gradually expand, and the load-displacement curve transitions from the initial linear segment to the nonlinear segment in Fig. 2. Owing to the significant dissipation of energy caused by interface debonding and fiber pull-out, the load-displacement curve exhibits a step-wise downward trend. The maximum load declines with the increase of temperature both in argon and air.

Additionally, the failure displacement at high temperatures is much lower than that at RT. Unlike in an argon atmosphere, the load-displacement curve in an air atmosphere exhibits a more pronounced step-like decrease in load at temperatures exceeding 1200 °C, with the load decreasing more rapidly. This is due to the fact that the high-temperature oxidation increases the bond strength between the matrix and the fibers, leading to a tendency for SiC_f/SiC composites to exhibit brittle failure. Moreover, the maximum load in air is lower than that in argon at high temperatures, indicating that both the temperature and environment atmosphere have a large effect on the mechanical behaviors of the sample.

Figure 4 illustrates the fracture toughness at various temperatures and atmospheres. The fracture toughness at RT is as high as 47.7 MPa m^{1/2}, significantly greater than that reported in the literature for the SiC_f/SiC composites (12–35 MPa m^{1/2}) [8,10,13,17]. It is indicated that the composites prepared in this work exhibit excellent toughness. As shown in Fig. 4, the high-temperature fracture toughness of the samples in air is lower than that in argon. It decreases to 42.3 and 34.5 MPa m^{1/2} at 800 °C in argon and air atmosphere, respectively. When the test temperature reaches 1000 °C, it further declines to 38.5 and 28.9 MPa m^{1/2}, respectively, owing to the high-temperature oxidation and fiber properties degradation. Similarly, it decreases to 31.4 and 21.1 MPa m^{1/2} at 1200 °C, respectively.

At 1350 and 1500 °C, the difference in fracture toughness between air and argon decreases, which is likely due to crack healing by oxidation. Section 4.2.3 (Micromorphology

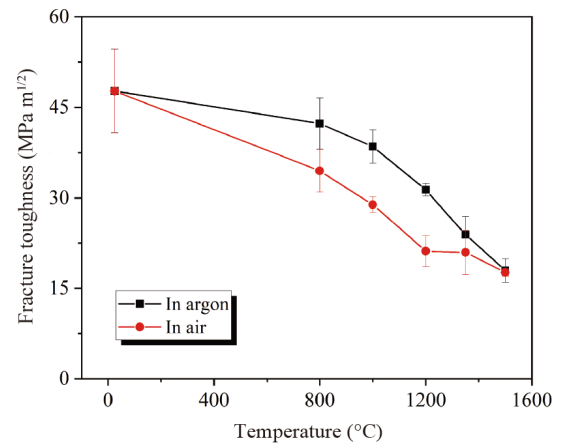


Figure 4 High-temperature experimental results of fracture toughness in argon and air.

and microstructure changes) will elaborate and demonstrate this in detail. Its value is 23.9 and 20.9 MPa m^{1/2} at 1350 °C in argon and air, respectively. Compared with RT, the fracture toughness decreased by 62.9% at 1500 °C. The difference in fracture toughness of SiC_f/SiC between argon and air initially increases (800–1200 °C) and then decreases (1200–1500 °C) with increasing temperature. This phenomenon is likely attributed to the combined effect of oxidation products of components as well as their impact on interface performance and oxygen entry channel. The above experimental results indicate that the service temperature and environment atmosphere have a prominent impact on the fracture toughness of the samples.

4.2 High-temperature failure mechanisms

4.2.1 Macroscopic failure morphology

Figures 5 and 6 show the macroscopic failure morphology from RT to 1500 °C in argon and air by digital microscope. It is evident that there are significant changes in the surface color of the specimen and fiber pull-out length with increasing temperature. Many fibers are pulled out from 25 to 1000 °C in both argon and air, which contributes to en-

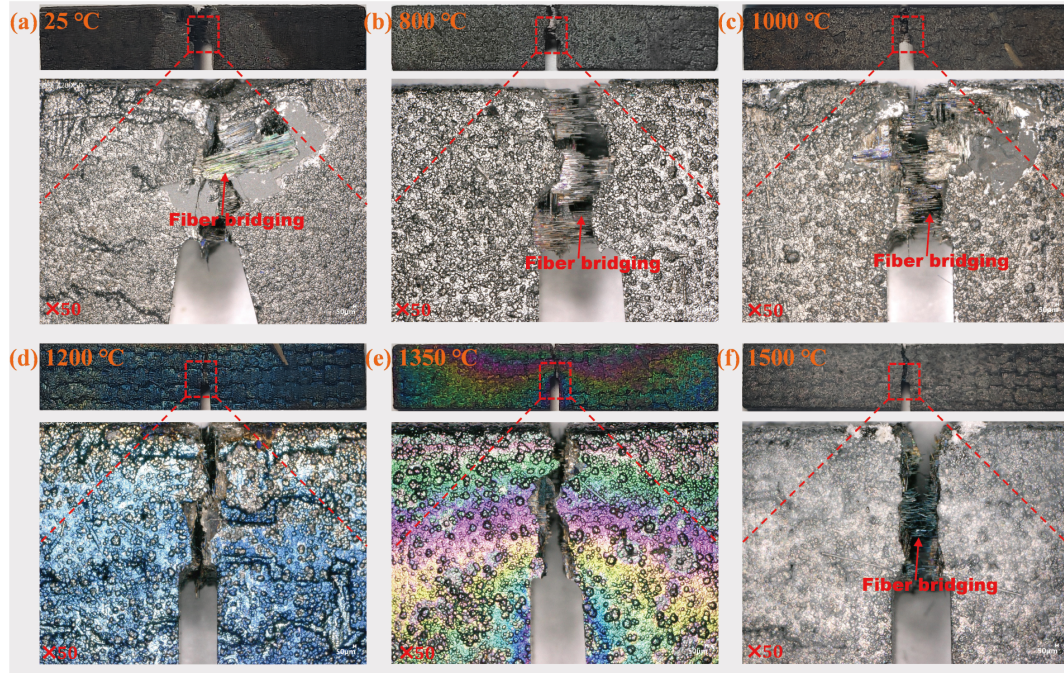


Figure 5 Macroscopic failure morphology of the samples in argon.

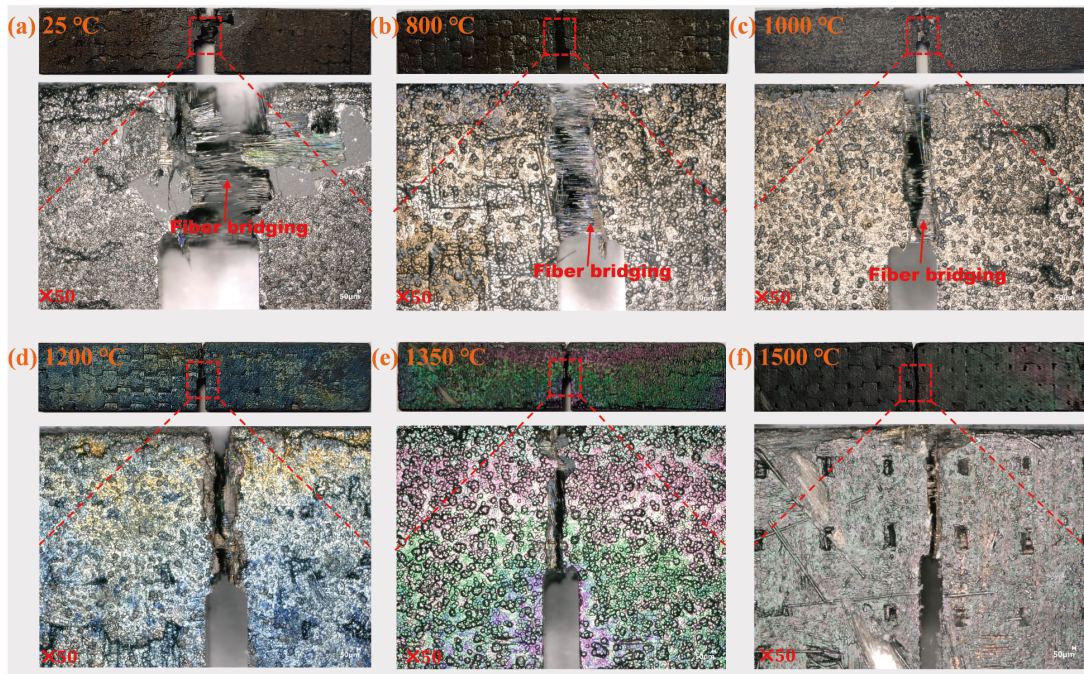


Figure 6 Macroscopic failure morphology of the samples in air.

hancing the fracture toughness of the samples. However, when the temperature exceeds 1000 °C as shown in Figs. 5 and 6, the pull-out length of SiC fiber is significantly reduced, particularly in air. At this point, the toughening effect of the fiber weakens, leading to a gradual decline in fracture toughness. Meanwhile, there is an increase in the silver oxide on the surface of the sample. It should be noted that

the surface of the specimen becomes colored at 1200 and 1350 °C in Figs. 5 and 6(d)-(e), which is likely associate with light refraction caused by glass oxides. Moreover, at 1500 °C in argon, there are still slight fiber pull-out and bridging phenomena of the SiC_f/SiC sample, which maintain good fracture toughness. Therefore, high-temperature properties of the interphase have significant influence on the

fracture toughness of the SiC_f/SiC composites.

4.2.2 Compositional analysis by XRD

The XRD patterns of the samples after loading at different atmospheres and temperatures are displayed in Fig. 7. The characteristic peaks of SiO₂ crystal were detected above 1200 °C in air in Fig. 7(b). As temperatures rise, they become more pronounced. The results indicate that the high-temperature oxidation is very significant. Thus, the fracture toughness at 1200 °C in air is much lower than that in argon. Moreover, the characteristic peak of SiO₂ crystal at 1500 °C is also observed in argon, which is due to the permeation of oxygen during the cooling process. However, due to the low content and amorphous structure of B₂O₃ and BN, their peaks are not observed [41,42]. On the one hand, high-temperature oxidation causes certain damage to SiC fibers, leading to a reduction in their strength. On the other hand, as the temperature increases, the surface of the SiC_f/SiC composites is covered with a relatively dense glass phase of SiO₂. This results in a reduced number of channels for external oxygen to enter the content [43]. Thus, the fracture toughness does not significantly decrease above 1350 °C.

4.2.3 Micromorphology and microstructure changes

Figures 8 and 9 show the cross-sectional morphology of the SiC_f/SiC composites in argon and air. It is evident that numerous SiC fibers are pulled out at 25 °C, and the interface debonding and fiber fracture consuming energy play an important role in increasing the fracture toughness. The fibers pull-out length reduced as the temperature increased, resulting in a decrease in fracture toughness. Additionally, the interface debonding is observed due to the evolution of interface properties and thermal residual stress (Fig. 8(e)). When the temperature reaches 1000 °C, the pull-out length of SiC fiber is equivalent to 800 °C in argon, but is significantly decreased in air (Fig. 8(f)). As a result, the value of fracture toughness in air is 25% lower than that in argon. Compared with 25 °C, the fracture toughness at 1500 °C reduces by 34% and 56% in argon and air, respectively.

As depicted in Fig. 9(a) and (d), the pulled-out length of fibers in the fracture morphology of the sample is significantly shorter in air compared to that in argon at 1200 °C. This difference can be attributed to a change in interface bonding performance caused by high-temperature oxidation, which further results in a decrease in fracture

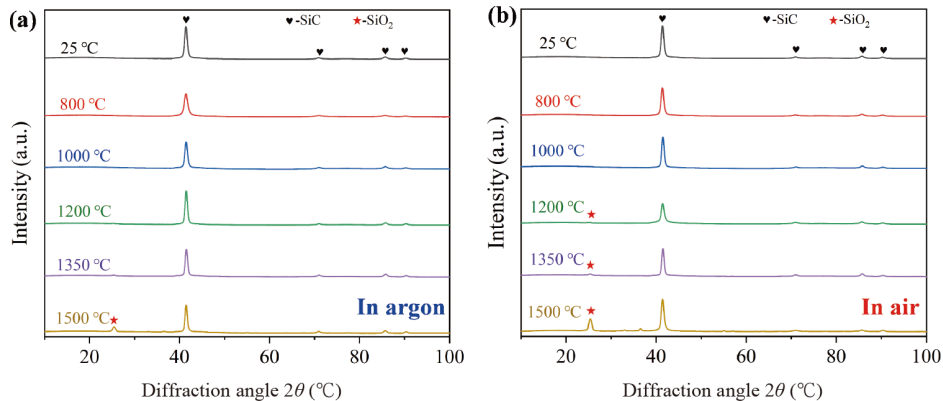


Figure 7 XRD patterns for SiC_f/SiC. (a) In argon; (b) in air.

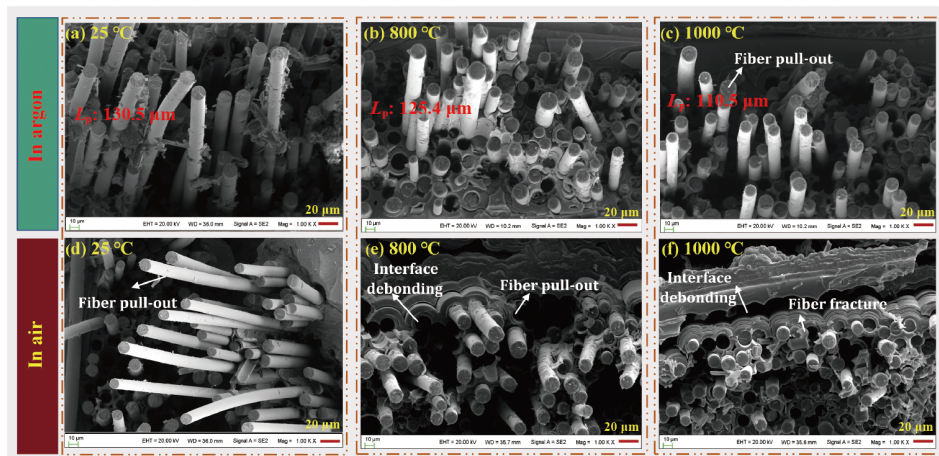


Figure 8 Microscopic morphology of cross-section of samples from 25 to 1000 °C.

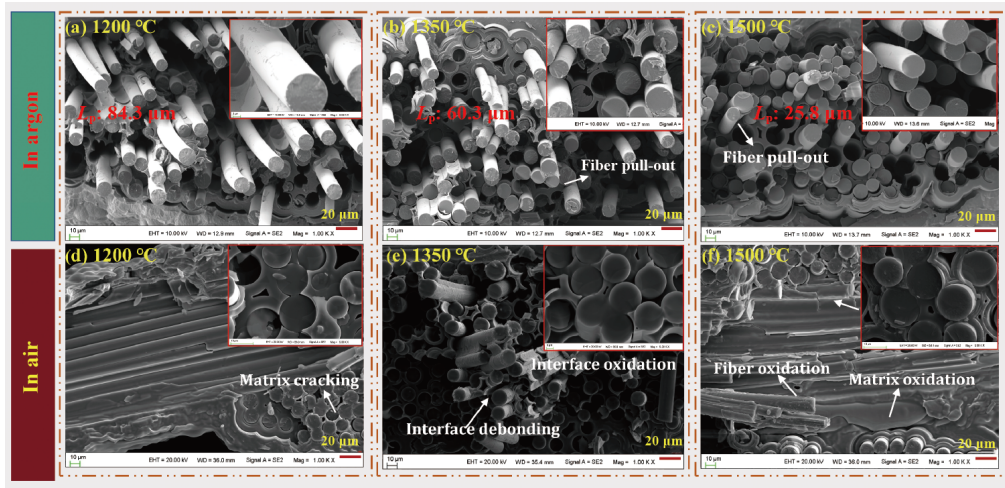


Figure 9 Microscopic morphology of cross-section of samples from 1200 to 1500 °C.

toughness. According to Fig. 9(e) and (f), the SiC fibers surface is covered with a thick layer of SiO₂ formed by oxidation of SiC matrix at 1350 and 1500 °C in air, confirmed by XRD analysis of its oxygen elements. Meanwhile, fiber strength deteriorates in air owing to high-temperature oxidation, which results in a further decrease in the fracture toughness. Due to the obstructive effect of oxides, the difference of fracture toughness in air and argon decreases above 1350 °C.

Based on the above macro and micro analysis, it can be concluded that the crack deflection, interface debonding, and fiber pull-out are the primary mechanisms that cause the composites to maintain high fracture toughness. The impact of oxidation on the composite fracture toughness is complex involving changes in interface bonding properties, thermal residual stress, and oxidation channels.

4.3 Model validation and comparison

To validate the proposed temperature-dependent fracture toughness model in Sect. 3 (Eq. (11)), the fracture toughness of FRCMCs was predicted by the developed model over a wide range of temperatures. The model predictions were compared with our experimental results and the available experimental data from the published literature, including the high-temperature fracture toughness of Cansas_f/SiC, Hi-Nicalon_f/Al₂O₃, YAG_f/Al₂O₃, SiC_f/SiC, Hi-Nicalon_f/SiC, Nicalon_f/Duran composites. During the prediction process, set RT as reference temperature (T_0). The material parameters shown in Tables 3 and 4, such as matrix fracture toughness and interfacial shear strength at RT, melting point, brittle-ductile transition temperature, modulus and thermal expansion coefficient of components, were obtained from

Table 3 General properties for model validation for the FRCMCs

Material parameters	Nicalon _f /Al ₂ O ₃	SiC _f /SiC	YAG _f /Al ₂ O ₃	Nicalon _f /Duran	Hi-Nicalon _f /SiC	Cansas _f /SiC
T_0 (°C)	25	25	25	25	25	25
$K_m(T_0)$ (MPa m ^{1/2})	3 [46]	3.51 [48]	3 [47]	0.84 [49]	3.51 [48]	3.51 [43]
T_m (°C)	2054 [50]	2700 [51]	2054 [50]	800 [18]	2700 [51]	2700 [56]
T_{mf} (°C)	2700 [51]	2700 [51]	1940 [59]	2700 [51]	2700 [51]	2700 [56]
$K_{m2}(T_1)$ (MPa m ^{1/2})	7.269	2.67	0.39	—	4.85	4.85
$E_m(T_0)$ (GPa)	383.2 [52]	410 [51]	383.2 [52]	63 [53]	410 [51]	410 [56]
$E_f(T_0)$ (GPa)	185 [54]	187 [55]	299 [60]	185 [54]	283.6 [58]	270 [72]
T_l (°C)	1200 [56]	1000 [57]	1200 [56]	—	1000 [57]	1000 [61]
V_f (%)	35 [44]	40 [16]	8.7 [45]	40 [46]	40 [17]	35
V_m (%)	65 [44]	60 [16]	92.3 [45]	60 [46]	60 [17]	65
r (μm)	7 [68]	6.5 [16]	11.5 [59]	7 [68]	7	7
$\sigma_{mu}(T_0)$ (MPa)	544 [61]	200 [51]	544 [61]	60 [46]	200 [51]	200 [51]
T_{proc} (°C)	1000 [62]	1000 [16]	1300 [45]	1000 [59]	1100 [17]	1150 [2]
$\tau(T_0)$ (MPa)	24.4 [58]	26.6 [63]	40 [58]	5.2 [69]	26.6 [63]	18.2 [70]
$\alpha_f \times 10^{-6}$	6 [64]	4.4 [67]	8 [66]	3.25 [46]	3.1 [65]	3.5 [71]
$\alpha_m \times 10^{-6}$	8 [64]	4.6 [64]	8 [64]	3 [46]	4.6 [64]	4.6 [64]

existing literature. The value of pull-out length was derived from the high-temperature experimental data of fracture toughness (in Table 5), except for Cansas_f/SiC composites (obtained from SEM). The Young's modulus of component for the FRCMCs we validated are given in Table 4. The high-temperature pull-out length of fibers are displayed in Table 5.

(1) Singel phase SiC ceramic

Firstly, taking single phase SiC ceramic as an example, the formula (Eqs. (4) and (5)) for the fracture toughness of ceramic matrix was verified at elevated temperatures. The fracture toughness of SiC from 25 to 1500 °C was predicted, as displayed in Fig. 10. The experimental results are sourced from the Ref. [74], and $E(T) = 460 - 0.04T \times \exp(-962/T)$ [74], $K_{m}(T_0)$ equals to 2.68, $K_{m2}(T_1)$ is 2.6. It is stated in the literature that the fracture toughness increased with temperature for SiC near 1073 K due to plastic deformation at the crack tip [74]. Thus, in the process of model verification, the influence of plastic deformation of SiC is included through Eq. (5), when temperature above 1073 K. Figure 10 indicates that the prediction accuracy of the model is acceptable from RT to 1723 K, especially at high tempera-

tures. The difference between the predicted and experimental values at 973 K is likely due to plastic deformation that occurs earlier than the reported temperature (1073 K).

(2) Cansas_f/SiC composites

The fracture toughness of Cansas_f/SiC composites tested (Fig. 4) in argon at elevated temperatures has been predicted by the developed model (Eq. (11)). During calculation, the pull-out length of fiber ($L_p(T)$) in Eq. (6) at different temperatures was obtained from the SEM results, it equals 130.5, 125.4, 110.5, 84.3, 60.3 and 25.8 μm at 25, 800, 1000, 1200, 1350 and 1500 °C, respectively (displayed in Figs. 8 and 9). As depicted in Fig. 11, the results predicted by the model are highly consistent with our experimental results. However, the influence of component oxidation on the fracture toughness has not yet been included in the proposed model, so the fracture toughness in air is not predicted, which will be considered in our future work.

(3) SiC_f/SiC composites

The fracture toughness of SiC_f/SiC composite fabricated by PIP in the Ref. [16] from RT to 1350 °C in vacuum was predicted. The necessary material parameters can be found in Tables 3 and 4. The brittle-ductile transition temperature

Table 4 The Young's modulus of component (in GPa)

Temperature (°C)	SiC _f /SiC [51,58]		Hi-Nicalon _f /SiC [51,58]		Cansas _f /SiC [58,73]		Nicalon _f /Al ₂ O ₃ [52,55]		Nicalon _f /Duran [53,54]		YAG _f /Al ₂ O ₃ [52,60]	
	$E_f(T)$	$E_m(T)$	$E_f(T)$	$E_m(T)$	$E_f(T)$	$E_m(T)$	$E_f(T)$	$E_m(T)$	$E_f(T)$	$E_m(T)$	$E_f(T)$	$E_m(T)$
25	270	410	284	410	270	410	185	383	190	63	299	383
500	—	—	—	—	—	—	—	—	184	63	—	—
600	—	—	—	—	—	—	—	—	183	55	—	—
700	—	—	—	—	—	—	—	—	181	47	—	—
800	255	393	—	—	230	393	—	—	—	—	—	—
1000	—	—	—	—	220	386	—	—	—	—	281	332
1200	250	379	281	379	209	379	127	259	—	—	277	259
1300	—	—	236	376	—	—	125	207	—	—	—	—
1350	245	374	—	—	157	374	—	—	—	—	—	—
1400	—	—	194	372	—	—	110	143.6	—	—	274	144
1500	—	—	—	—	102	369	—	—	—	—	—	—

Table 5 The temperature-dependent pull-out length of fiber (in μm) in different composites

Temperature (°C)	SiC _f /SiC	Hi-Nicalon _f /SiC	Nicalon _f /Al ₂ O ₃	Nicalon _f /Duran	YAG _f /Al ₂ O ₃	Cansas _f /SiC
25	39	78	60	97	8	130.5
300	—	—	—	116	—	—
400	—	—	—	128	—	—
500	35	70	56	141	—	—
600	33	66	59	129	—	—
700	32	60	57	117	—	—
800	32	59	57	—	—	125.4
1000	32	60	54	—	9	110.5
1200	31	60	53	—	8	84.3
1300	—	58	48	—	—	—
1350	30	—	—	—	—	60.3
1400	—	42	47	—	9	—
1500	—	—	—	—	—	25.8

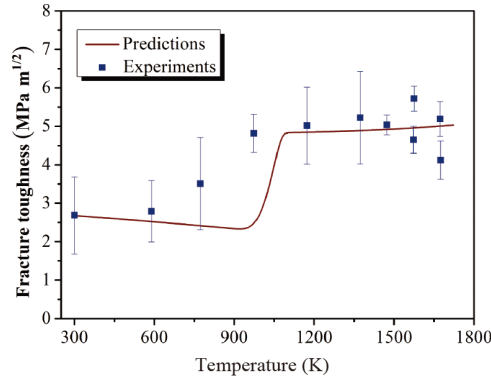


Figure 10 Comparison between experimental values [74] and model predictions of SiC ceramic.

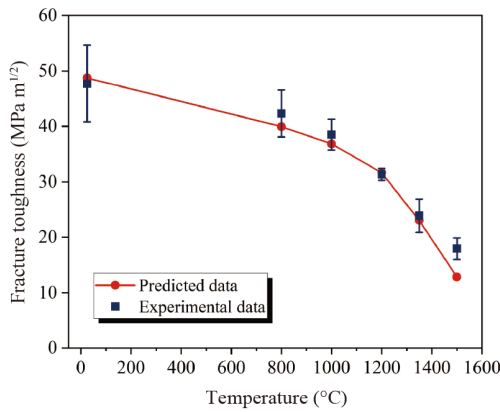


Figure 11 Comparison between experimental values and model predictions of Cansas/SiC composites.

of SiC is 1000 °C [57]. Figure 12(a) demonstrates that the proposed model exhibits strong predictive capability for the fracture toughness of SiC_f/SiC composites at elevated temperatures. The fracture toughness retention rate is 92.7% in vacuum at 1350 °C, but the value at low temperatures is significantly smaller than that of the Cansas/SiC composites. Moreover, the turning points of fracture toughness predictions at 1000 °C can be found in Fig. 12(a), which is attributed to the effect of the increase in fracture toughness

resulting from the consideration of plastic power above brittle-ductile transition temperature.

(4) Hi-Nicalon_f/SiC composites

The fracture toughness of Hi-Nicalon_f/SiC composites [17] was also predicted at different temperatures. The relative material properties of the Hi-Nicalon_f/SiC composite are given in Tables 3 and 4. Figure 12(b) shows the comparison between experimental data and our model predictions from RT to 1400 °C, and good consistency is achieved. Meanwhile, above the composite fabrication temperature (i. e., 1100 °C), it can be observed that the fracture toughness increased with temperature increase, which is likely due to a decrease in thermal stress and interface debonding [17].

(5) Hi-Nicalon_f/Al₂O₃ composites

We further predicted the fracture toughness of Hi-Nicalon_f/Al₂O₃ [44] from 25 to 1400 °C. The relative material parameters for Hi-Nicalon_f/Al₂O₃ composites are given in Tables 4 and 5. The brittle-ductile transition temperature of Al₂O₃ is 1200 °C [56]. As illustrated in Fig. 13(a), the fracture toughness decreased with increasing temperature, and good agreement is achieved between the experimental results and predicted results. At 1200 °C, the fracture toughness showed a slight increase due to the contribution of plastic power above brittle-ductile transition temperature.

(6) YAG_f/Al₂O₃ composites

In addition, the developed model was utilized to predict the fracture toughness of YAG_f/Al₂O₃ composite. For comparison, the fracture toughness of YAG_f/Al₂O₃ composite [45] and monolithic Al₂O₃ from RT to 1400 °C are both illustrated in Fig. 13. It can be seen that the YAG fiber has a good toughening effect at high temperatures, and the predicted data of the theoretical model achieved good consistency with the experimental values of YAG_f/Al₂O₃ composites.

(7) Nicalon_f/Duran composites

Finally, our proposed model was used to predict the fracture toughness of Nicalon_f/Duran composites, as reported in the Ref. [46]. The influence of thermal residual stress was ignored due to similar thermal expansion coef-

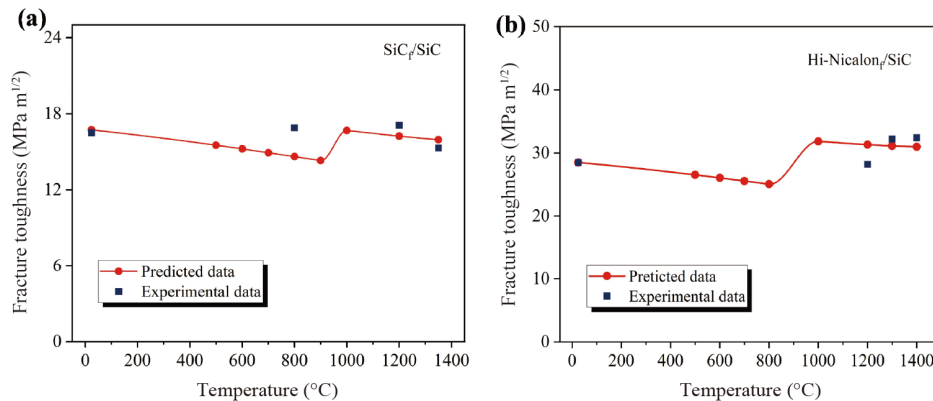


Figure 12 Comparison between experimental values and model predictions of: (a) SiC_f/SiC [16]; (b) Hi-Nicalon_f/SiC composites [17].

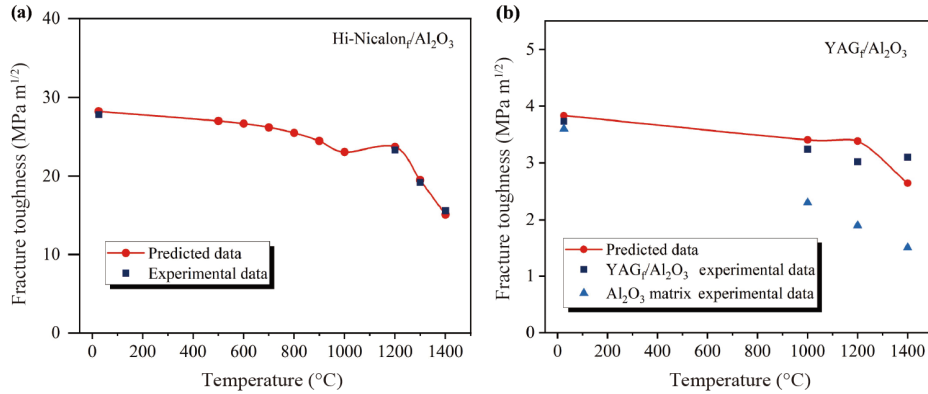


Figure 13 Comparison between experimental values and model predictions of: (a) Hi-Nicalon/Al₂O₃ composites [44]; (b) YAG/Al₂O₃ composites [45].

ficient. Moreover, because the test temperature does not reach the brittle-ductile transition temperature, there is no need to consider the effect of plastic power. In this example validation, we only take into account the contribution of the fiber pull-out and Duran matrix. Figure 14 indicates that the theoretical model has satisfied ability to predict high-temperature fracture toughness.

Based on the above comparison between the experimental data and model predictions, it has been confirmed that the model established in this study demonstrates strong predictive capability for fracture toughness of FRCMCs at high temperatures. The model develops the quantitative relationship among the fracture toughness of FRCMCs, temperature, fiber content, as well as the fundamental components parameters. It is worth mentioning that the physical parameters in the model can be easily obtained from the literature, so it offers a feasible and convenient prediction means of fracture toughness of FRCMCs at elevated temperatures.

4.4 Influencing factor analysis

To explore the quantitative impact of component properties on the fracture toughness of FRCMCs, this work conducted an analysis of influencing factors. In this section, the influence of Young's modulus of component, fiber strength and interfacial shear strength on the fracture toughness of composites has been discussed. Additionally, an investigation into the contribution of various toughening mechanisms to fracture toughness was also conducted.

4.4.1 The effect of Young's modulus of component

First, using the Hi-Nicalon/Al₂O₃ composites as an example, the proposed model was utilized to analyze the effect of Young's modulus of component on the fracture toughness of the composites. As shown in Fig. 15(a), improving the matrix Young's modulus helps to enhance the composites fracture toughness. Moreover, its sensitivity to matrix Young's

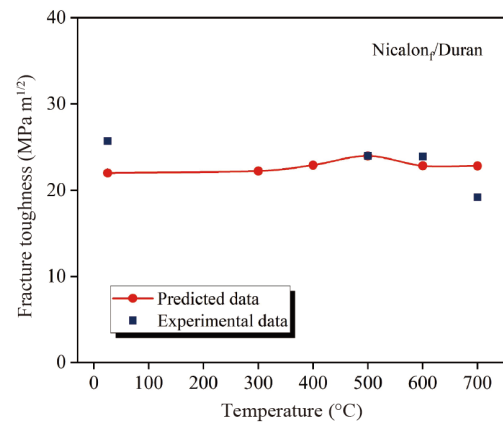


Figure 14 Comparison between experimental values [46] and model predictions of Nicalon/Duran composite.

modulus improves with the increase of temperature. On the contrary, Fig. 15(b) suggests that reducing fiber Young's modulus has a positive impact on enhancing the fracture toughness. Meanwhile, its sensitivity to fracture toughness decreases with rising temperature. Similar conclusions have also been reported in the Ref. [29]. Therefore, in the process of material preparation, increasing the Young's modulus of ceramic matrix or reducing the Young's modulus of reinforced fibers can effectively enhance the fracture toughness of FRCMCs.

4.4.2 The effect of fiber strength and interfacial shear strength

The quantitative effect of fiber strength and interface shear strength on fracture toughness of Hi-Nicalon/Al₂O₃ composites, as well as their evolution with temperature, has been studied in this study. As can be seen from Fig. 16(a), increasing fiber strength also helps to enhance the fracture toughness of Hi-Nicalon/Al₂O₃ composites, and its sensitivity to fiber strength declines with increasing temperature. As shown in Fig. 16(b), the reduction of interface shear strength has a significant impact on improving fracture

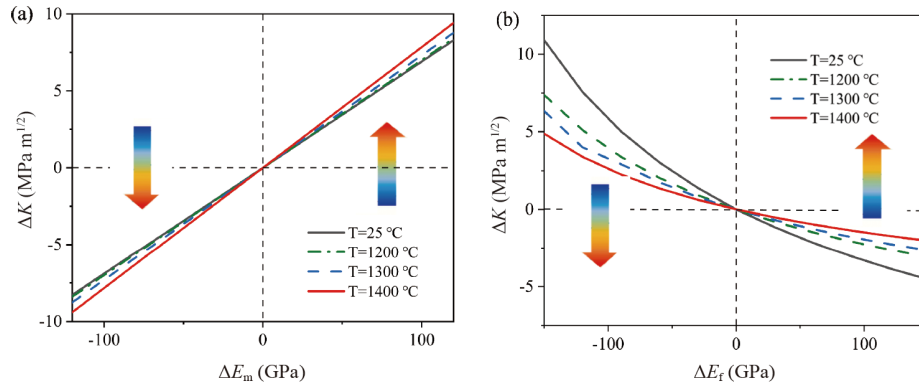


Figure 15 The sensitivity of fracture toughness to Young's modulus of (a) matrix and (b) fiber.

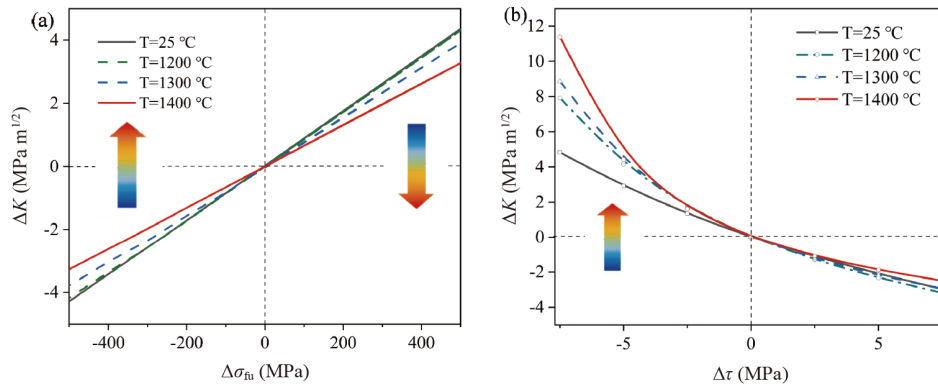


Figure 16 The sensitivity of fracture toughness to (a) fiber strength and (b) interface shear strength.

toughness, particularly at elevated temperatures. Furthermore, its sensitivity to interface shear strength increases with rising temperature. Therefore, in the process of material preparation, appropriately reducing the interfacial shear strength is more conducive to improving the fracture toughness of FRCMCs.

4.4.3 The contribution of various mechanisms to fracture toughness

As can be seen from Sect. 3, the high-temperature plastic

power, fiber pull-out, and thermal residual stress have a significant effect on the fracture toughness of FRCMCs. Experimental testing is unable to quantitatively characterize the individual contributions of different mechanisms to the fracture toughness. In order to address this issue, the proposed model was used to study the contribution of various mechanisms, including matrix effect, plastic power, fiber pull-out, and residual thermal stress to fracture toughness.

Figure 17(a) and (b) shows the contribution of the aforementioned mechanisms to the fracture toughness of Hi-

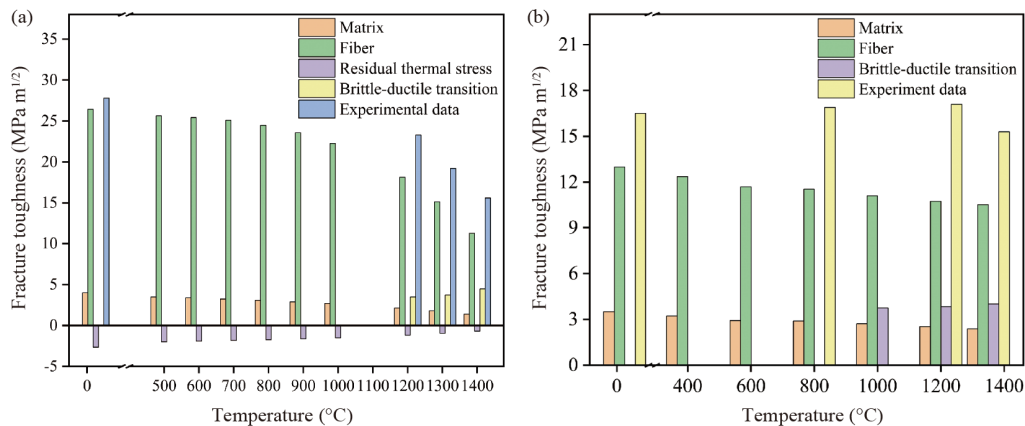


Figure 17 The contribution of various toughening mechanisms to the fracture toughness of (a) Hi-Nicalonf/Al₂O₃ and (b) SiC_f/SiC composites.

Nicalon_f/Al₂O₃ and SiC_f/SiC composites and their evolution with temperature. As displayed in Fig. 17, the contribution of the fiber pull-out mechanism to fracture toughness exceeds 80%, playing a dominant role overall and decreasing with increasing temperature. Above brittle-ductile transition temperature, the plastic power begins to play a positive role in improving the composites fracture toughness, and its contribution increases with increasing temperature (Fig. 17(a)). On the contrary, residual thermal stress has a negative impact on their fracture toughness, and its effect gradually decreases as the temperature approaches the preparation temperature. In conclusion, the proposed theoretical model not only provides a means to predict high-temperature fracture toughness, but also can accurately analyze the quantitative influence of different influencing mechanisms and material parameters on the fracture toughness of FRCMCs.

5. Conclusions

This study focuses on exploring the impact of temperature and atmosphere on the fracture toughness and failure mechanisms of two-dimensional plain-woven SiC_f/SiC composites experimentally and theoretically. The key findings can be summarized as follows:

(1) The fracture toughness is as high as 48 MPa m^{1/2} at RT and gradually decreases with increasing temperature. The value in air is smaller than that in argon, and the difference between the fracture toughness in argon and air initially increases and then reduces with increasing temperature.

(2) The interface debonding and fiber pull-out are the primary mechanisms contributing to the high fracture toughness. The performance degradation and oxidation of SiC fiber led to a decrease of fracture toughness at elevated temperatures, particularly in air. The impact of oxidation on the composite fracture toughness is multifaceted in air, such as changing interface bonding properties, residual thermal stress, and oxidation channels.

(3) A physic-based temperature-dependent model of fracture toughness considering matrix toughness, plastic power, fiber pull-out, and residual thermal stress for FRCMCs was established. It provides an effective theoretical approach for characterizing and predicting the high-temperature fracture toughness of FRCMCs.

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

Author contributions Yong Deng and Chao Zhang designed the research. Yong Deng, Yi Hao and Weiguo Li wrote the first draft of the manuscript. Yi Hao and Huanfang Wang set up the experiment set-up and processed the experiment data. Qiang Qin and Bing Pan helped organize the manuscript. Yong Deng and Chao Zhang revised and edited the final version.

Acknowledgements This work was supported by the National Natural Science Foundation of China (Grant Nos. 12102354, 12472214 and 12002288), the Young Elite Scientists Sponsorship Program by CAST (Grant No. 2022QNR001), the Guangdong Basic and Applied Basic Research Foundation (Grant Nos. 2023A1515012620 and 2024A1515012018), the independent research project of the National Key Laboratory of Strength and Structural Integrity (Grant No. LSSIZZYJ202305), and the Basic Research Program of Taicang (Grant No. TC2022JC09).

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温度和气氛对二维平纹编织SiC_f/SiC复合材料断裂韧性和失效机制的影响: 试验与建模

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摘要 陶瓷基复合材料因其耐高温和抗氧化特性在航空航天领域具有广泛应用前景。本文通过单边切口梁三点弯曲法系统研究了温度和环境气氛对二维平纹编织SiC_f/SiC复合材料断裂韧性和失效机制的影响。结果表明, SiC_f/SiC复合材料在不同温度下表现出伪塑性变形特征, SiC_f/SiC室温断裂韧性高达48 MPa m^{1/2}, 随温度升高断裂韧性逐渐降低, 并在氩气和空气中的差异先增大后减小。进一步, 通过宏观形貌和成分分析, 深入研究了SiC_f/SiC复合材料的高温失效机制和关键影响因素。在此基础上, 通过考虑基体韧性、高温塑性功、纤维拔出和残余热应力等因素的影响, 针对纤维增强陶瓷基复合材料建立了基于物理机理的温度相关性断裂韧性理论表征模型。模型与实验结果充分验证, 据此深入开展了断裂韧性的影响因素分析。该研究有助于理解SiC_f/SiC复合材料热力氧耦合失效机制和力学性能演变规律, 支撑其行业应用。