Microstructure and Nanoindentation Study of Gradient Multilayer SiC/TaC Ceramic Composite Interface Modified C/C Composites (Postprint)

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Abstract

Using the chemical vapor infiltration (CVI) process and controlling the flow direction of the reactive gases, C/C composites modified with a multilayer SiC/TaC ceramic composite interface featuring gradient distribution of the ceramic phase were prepared. The results indicate that along the thickness direction of the C/C composites, the content of the SiC/TaC ceramic phase decreases rapidly, the interface thickness diminishes, and the interfacial structure transitions from a multilayer SiC/TaC laminated interface (Zone I) to a cluster-like SiC/TaC ceramic composite phase interface (Zone II) and a monolayer TaC ceramic interface (Zone III). In Zone I, the multilayer SiC/TaC ceramic composite interface consists of five sub-interfacial layers: a SiC layer (layer i), a TaC layer (layer ii), a TaC composite layer embedded with SiC particles (layer iii), a SiC composite layer embedded with TaC phase (layer iv), and a TaC layer (layer v). In Zone II, the ceramic phase no longer coats in a lamellar form but instead grows cluster-like on the carbon fiber surface. This paper also investigates the distribution of nanoindentation hardness and Young' s modulus across the multilayer SiC/TaC ceramic composite interface.

Full Text

Microstructure and Nano-indentation of C/C Composites Modified with Multi-interlayers of SiC/TaC Ceramics Gradient Distribution

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Abstract

Multi-interlayers of SiC/TaC ceramics with gradient distribution were inserted into C/C composites by chemical vapor infiltration (CVI) while controlling the flow direction of reaction gas in the porous C/C preforms. The results show that along the thickness direction of C/C composites, the content of SiC/TaC ceramic phase and the thickness of the multi-interlayer decrease, while the structure of the ceramic phase changes from multi-interlayer (Region I) to composite interlayer (Region II) and then single interlayer (Region III). In Region I, the SiC/TaC multi-interlayer is composed of five interlayers: the first SiC layer, the second TaC layer, the third TaC composite interlayer embedded with SiC particles, the fourth SiC composite interlayer embedded with fine mosaic-like TaC phase, and the fifth white TaC layer. In Region II, the ceramic phase no longer coats the surface of carbon fiber in the form of an interlayer, but instead grows as cauliflower-like clusters. Finally, the nano-hardness and elastic modulus of the SiC/TaC ceramic multi-interlayer in Region I were also discussed.

KEY WORDS: composites, multi-interlayer, nano-indentation, gradient, microstructure

Introduction

The high-temperature oxidation and ablation protection of C/C composites has been a research focus for materials scientists worldwide [1-4]. Studies have shown that oxidation of C/C composites initiates at the carbon fiber/matrix interface [5]. Therefore, besides the properties of carbon fibers and matrix carbon, the interface between fiber and matrix carbon is also a critical factor affecting the oxidation and ablation performance of C/C composites. Based on this understanding, researchers have combined coating and matrix modification approaches by introducing high-strength, high-hardness ceramic phases with excellent ablation resistance as coatings at the carbon fiber/matrix interface to enhance toughening and improve oxidation/ablation resistance. Labruquère et al. [6] introduced Si-B-C composite coatings at the carbon fiber/matrix interface using chemical vapor infiltration (CVI), which improved the mechanical properties and oxidation resistance of C/C composites. C. Verdon et al. [7] introduced HfC-SiC multi-interlayers in C/C composites. A. Sayir [8] prepared carbon fiber reinforced HfC and TaC/HfC matrix composites. Chen et al. [9] and Zhu et al. [10] also conducted related research. Central South University has performed extensive research on improving the oxidation and ablation resistance of C/C composites through ceramic composite interface modification, finding that introducing high-strength, high-hardness ceramic composite interfaces with high melting points and ablation resistance, such as PyC/TaC/PyC or PyC/SiC/TaC/PyC, between carbon fiber and matrix carbon can significantly enhance the mechanical properties and oxidation/ablation resistance of C/C composites [11-14]. However, extensive ablation tests have shown that oxidation and ablation of C/C composites with ceramic protection mainly occur within 1-2 mm or even thinner regions from the ablation surface. Consequently, most ceramic phases introduced uniformly throughout the C/C composite, particularly those far from the ablation surface, cannot fulfill their oxidation/ablation protection role. Moreover, introducing this excess ceramic phase not only substantially increases the density of C/C composites but also leads to high preparation costs, which does not align with the development trend of low-cost, high-performance C/C composites.

Gradient materials are a novel class of materials in which the constituent elements (composition, structure) vary continuously along the thickness direction from one side to the other, resulting in continuous gradient changes in material properties. Researchers have investigated the preparation and properties of modified C/C composites with ceramic gradient structures. For example, Y. C. Zhu et al. [15] prepared gradient SiC-modified C/C composites by infiltrating molten Si into the C/C matrix surface using melt infiltration to form a functionally gradient (Si_3N_4+SiC)/C layer. Li et al. [16] prepared SiC-$HfSi_2$-$TaSi_2$ coatings on C/C composites using pack cementation, where partial Si element diffused into the C/C matrix to form SiC with gradient distribution. However, these studies utilized diffusion of molten Si into surface pores of C/C composites during coating preparation to form ceramic phase gradient transitions, without conducting design and control studies on the gradient distribution of ceramic phases in porous C/C composites.

Building on previous research, Central South University designed forced convection tooling to control the flow direction and deposition location of reaction gas in the preform, achieving gradient distribution of TaC along the Z-axis direction of C/C composites and preparing gradient-distributed TaC interface-modified C/C composites, with preliminary investigation of microstructure and macroscopic flexural strength [17]. Compared with simple ceramic interfaces, multi-layer ceramic composite interfaces composed of alternating ceramic and carbon phases or multiple ceramic phases offer greater advantages for enhancing toughening and improving oxidation/ablation resistance of C/C composites. Therefore, this study employed chemical vapor infiltration (CVI) to introduce multi-layer SiC/TaC ceramic composite interfaces at the carbon fiber/matrix interface. By controlling the flow of precursor gas from the ablation surface to the non-ablation surface of low-density C/C preforms using forced convection, gradient distribution of multi-layer SiC/TaC ceramic composite interfaces was achieved in C/C preforms. The resulting gradient-distributed multi-layer SiC/TaC ceramic composite interface-modified C/C composites were prepared, and the distribution of composition and structure of the multi-layer SiC/TaC ceramic composite interface in C/C composites, as well as its nano-hardness and Young' s modulus, were thoroughly investigated.

Experimental

The preform used in this study was a quasi-three-dimensional needled carbon felt with a density of (0.56$±0.05)g/cm^{3}$, reinforced with T300-12K carbon fibers at a volume fraction of 32.5%.

CVI was employed to deposit a transitional PyC layer, multi-layer SiC/TaC ceramic composite interlayer, and dense PyC layer on the carbon fiber surfaces in the preform. The deposition temperature for the transitional PyC layer and dense PyC layer was 1120°C at a pressure of approximately 10 kPa, using C_3H_6 as the precursor. For SiC deposition, the temperature was 950°C at 200-600 Pa, with MTS (CH_3SiCl_3) as the precursor and high-purity H_2 as the carrier gas. Gaseous MTS evaporated from a water bath was introduced into the reactor via bubbling, with an H_2:MTS molar ratio of 10 and high-purity Ar as the dilution gas. TaC deposition was performed at 800°C and 200-600 Pa, using $TaCl_5$ and C_3H_6 as precursors with Ar serving as both the $TaCl_5$ carrier gas and protective gas.

A custom-designed chemical vapor deposition furnace with forced convection tooling was used to control the flow direction and deposition location of reaction gas in the preform, enabling gradient distribution of SiC/TaC ceramic composite interfaces along the Z-axis direction of C/C composites. After depositing the gradient-distributed multi-layer SiC/TaC ceramic composite interfaces and dense PyC layer, the samples were further densified using impregnation-carbonization cycles with resin carbon.

The final material density was approximately 2.2-2.4 g/cm^3 with an open porosity of 5%. Microstructural characterization of the gradient C/C composites and multi-layer SiC/TaC ceramic composite interfaces was performed using a Nova Nano SEM230 field emission scanning electron microscope (FESEM). Elemental composition in different regions and within the multi-layer ceramic composite interfaces was qualitatively and quantitatively analyzed using a JEOL JXA-8230 electron probe microanalyzer (EPMA). Nanoindentation hardness and Young' s modulus of each sub-interlayer in the multi-layer SiC/TaC ceramic composite interface were measured using a CSM nanoindenter (model OPX). The Berkovich indenter was calibrated on standard silicon samples using the continuous stiffness method. During testing of the SiC/TaC ceramic composite interface, the Berkovich indenter was continuously loaded onto the interlayer, held at a maximum load of 5 mN for 10 s, then unloaded to obtain the complete load-depth curve. Hardness and Young' s modulus values were calculated using Oliver' s formula combined with the load-depth curves [18]. Prior to testing, samples were sequentially ground with water sandpaper and polished to achieve a surface finish of 5 m, meeting the requirements for nanoindentation testing.

Results and Discussion

2.1 Gradient Distribution of SiC/TaC Ceramic Composite Interface Along C/C Composite Thickness Direction

Figure 1a [Figure 1: see original paper] shows the microstructure of the C/C preform after SiC/TaC ceramic composite interface deposition. The white regions represent the SiC/TaC ceramic phase. During CVI preparation of the SiC/TaC ceramic composite interface, forced convection was employed to drive reaction gas through the porous C/C preform, creating gradient distribution of SiC/TaC ceramic phase along the thickness direction (Z-axis): from left to right in the C/C preform, the SiC/TaC ceramic phase content gradually decreases. Based on the varying ceramic phase content, the C/C preform can be divided into three regions (I, II, III) along the Z-axis. Region I, located 0-1.3 mm from the surface, contains the highest ceramic phase content with a thick SiC/TaC ceramic composite interface between carbon fibers and matrix of approximately 12000.0 nm (Fig. 1b). Region II, spanning 1.3-2.8 mm from the surface, shows decreased ceramic content but still exhibits a distinct SiC/TaC ceramic composite interface with a thickness of about 637.6 nm (Fig. 1c). Region III, at 2.8-5.2 mm from the surface, displays further reduced ceramic content with an interface thickness of only 187.5 nm (Fig. 1d). However, due to diffusion of reaction gas, small amounts of SiC/TaC ceramic phase inevitably formed on the right side of the preform.

Figure 2a [Figure 2: see original paper] presents the microstructure of the SiC/TaC ceramic composite interface-modified C/C composites after pyrocarbon and resin carbon densification. The ceramic phase distribution retained the characteristics observed before pyrocarbon densification. Region I contains the most ceramic phase with relatively less matrix carbon, Region II shows reduced ceramic phase with increased matrix carbon content, and Region III has even less ceramic phase with more matrix carbon. Higher magnification images of Regions I, II, and III are shown in Figs. 2b, 2c, and 2d, respectively. The microstructures of the ceramic composite interfaces differ significantly among the three regions due to varying ceramic phase content. In Region I, the thick ceramic composite interface exhibits both white and gray ceramic phases distributed in layered forms on the carbon fiber surface, forming a multi-layer SiC/TaC ceramic composite interface. In Region II, the interface thickness decreases and the ceramic phase no longer coats the carbon fiber surface as multiple layers but instead shows a structure of gray SiC particles dispersed in a white TaC ceramic phase matrix, growing sporadically on the carbon fiber surface as SiC/TaC composite ceramic interfaces. In Region III, the ceramic interface thickness is extremely low, making it impossible to distinguish the gray SiC ceramic phase, leaving only a thin layer of TaC ceramic interface visible.

2.2 Microstructure of SiC/TaC Ceramic Composite Interface

Figure 3a [Figure 3: see original paper] shows an enlarged micrograph of the multi-layer SiC/TaC ceramic composite interface in Region I. Based on morphological features, the SiC/TaC ceramic composite interface on the carbon fiber surface can be divided into five sub-layers from inner to outer: a gray SiC layer adjacent to the carbon fiber (layer i), a white TaC layer next to the SiC layer (layer ii), a TaC/SiC composite layer with gray particles in a white ceramic matrix (layer iii), a SiC/TaC composite layer with white fine mosaic patterns in a gray ceramic matrix (layer iv), and a white TaC outer layer (layer v), labeled as i, ii, iii, iv, and v in Fig. 3. Layer i consists of SiC, which has a relatively low thermal expansion coefficient (4.8$×10ˆ{-6}$ °C^{-1} [19]) and serves as a transition layer to relieve thermal stress between carbon fiber and TaC. In layer i, SiC exhibits a distinct layered structure coating the carbon fiber surface. Layer ii is a dense white TaC layer. Layer iii is primarily white TaC containing small amounts of gray SiC distributed as particles. This SiC particle + TaC matrix composite interface reduces residual thermal stress between the single ceramic coating and carbon phase. Layer iv is mainly gray SiC phase containing small amounts of white fine mosaic-like TaC. This structure likely forms because after TaC interface deposition, the sample must be cooled from 800°C to room temperature and reheated to 950°C for subsequent SiC deposition. During this thermal cycling, TaC in layer iii expands and contracts, forming blister-like protrusions that create mosaic patterns into which subsequently deposited SiC infiltrates, resulting in this composite interface structure with white patterns in a gray matrix. Layer v, the outermost composite interface layer, consists of TaC.

Figure 3b shows the microstructure of the SiC/TaC ceramic composite interface in Region II. The ceramic phase no longer coats the carbon fiber surface as layers but instead grows sporadically as cauliflower-like or plum blossom-like clusters composed of gray ceramic pistils and white ceramic petals. This morphology likely arises because, after depositing layer i SiC, the amount of SiC precursor gas reaching Region II is significantly reduced due to depletion effects, preventing the formation of a complete layered SiC coating. Additionally, the precursor gas is in the late stage of deposition with short residence time, resulting in only sporadic, island-like SiC clusters on the carbon fiber surface that create a very rough deposition substrate. Subsequent TaC deposition then uses these scattered island-like SiC clusters as nucleation sites, forming the observed cauliflower-like or plum blossom-like morphology.

2.3 Elemental Composition Analysis

Electron probe microanalysis was used to quantitatively characterize the content of C, Si, and Ta elements in Regions I, II, and III of the gradient-distributed multi-layer SiC/TaC ceramic composite interface-modified C/C composites, using a beam spot diameter of 100 m. The measurement zones are shown in Figs. 2b, 2c, and 2d, with the elemental content distribution presented in Fig.

4 [Figure 4: see original paper]. The results demonstrate distinct differences in C, Si, and Ta content among Regions I, II, and III. From Region I to Region III, C content gradually increases while both Ta and Si contents decrease. Notably, carbon is the dominant component in all three regions, with C content significantly higher than Si and Ta. Based on these results, it can be inferred that the white ceramic phase is primarily composed of Ta, while the gray ceramic phase is mainly Si.

A JEOL JXA-8230 electron probe was used to qualitatively describe the elemental distribution of C, Si, and Ta across the sub-layers in the multi-layer SiC/TaC ceramic composite interface in Region I. Figure 5 [Figure 5: see original paper] shows the elemental mapping results (qualitative analysis) for the three elements in the multi-layer SiC/TaC ceramic composite interface. Carbon is present throughout the composite interface but concentrated primarily at the carbon fiber. Silicon is mainly found in layer i (the gray region outside the carbon fiber) and in layers iii and iv, where it exists as localized aggregates. Tantalum is predominantly present in layers ii, iii, and v.

2.4 Nanoindentation Testing of SiC/TaC Ceramic Composite Interface

Nanoindentation was used to investigate the hardness and Young' s modulus values of each sub-interlayer in the multi-layer SiC-TaC ceramic composite interface in Region I, examining the distribution of these mechanical properties across the interface. The nanoindentation test points and corresponding hardness and Young's modulus values for the multi-layer SiC/TaC ceramic composite interface in Region I are shown in Fig. 6 [Figure 6: see original paper].

The results reveal significant variations in nanoindentation hardness and Young's modulus across different sub-interlayers. At points 1 and 2 in layer i, the highest hardness (27.7-28.2 GPa) and Young' s modulus (313.7-335.9 GPa) values were obtained. In layers ii and iii, these values rapidly decreased to 10.7-14.6 GPa and 98.4-262.8 GPa, respectively. In layer iv, the values increased sharply again to 16.6-27.8 GPa and 201.6-262.6 GPa. In layer v, the values decreased once more to 8.1-10.2 GPa and 83.7-137.3 GPa.

Based on the nanoindentation hardness and Young' s modulus values in Fig. 6 [Figure 6: see original paper] and the elemental composition characterization in Fig. 5 [Figure 5: see original paper], the distribution pattern across the multi-layer SiC/TaC ceramic composite interface shows that regions with higher SiC content exhibit higher hardness and Young' s modulus values. For example, layers i and iv, which are primarily SiC, demonstrate significantly higher hardness and modulus than layers ii, iii, and v, which are mainly TaC. The hardness and modulus of the composite layers iii and iv fall between those of the TaC-rich and SiC-rich layers.

Furthermore, the nanoindentation hardness and Young' s modulus of the SiC interlayer prepared under these conditions are lower than those of SiC coatings prepared at 1100-1200°C (hardness: 34-38 GPa; modulus: 380-490 GPa) [20], and the hardness of the TaC interlayer is also lower than that of pure TaC coatings (13.7-21.2 GPa) [21]. This is primarily attributed to: (1) the relatively high carbon content in the sub-interlayers, and (2) the lower crystallinity of SiC and TaC ceramic phases deposited at lower temperatures, which tend to form needle-like crystal structures with numerous nanopores [22].

Figure 7 [Figure 7: see original paper] shows the load-displacement curves for each indentation point in the multi-layer SiC/TaC ceramic composite interface. Figure 8 [Figure 8: see original paper] presents the maximum indentation depth and residual indentation depth for each point. In layer i (points 1, 2) where SiC is the main component, the maximum indentation depths under 5 mN load are 97.8 nm and 99.0 nm, respectively, with residual depths of 30.5 nm and 32.8 nm after unloading, indicating plastic deformation. In layer ii (points 3, 4, 5) where TaC dominates, the maximum indentation depth reaches 141.9 nm with a residual depth of 64.1 nm, showing that TaC-rich layers are softer, exhibit deeper penetration under the same load, and are more prone to plastic deformation.

In layer iii, the TaC/SiC composite layer, the load-displacement curves show three distinct cases depending on composition and structure. At points 7, 8, and 9 where TaC is dominant, the maximum indentation depth is 137.7 nm with a residual depth of 67.7 nm. At point 10 where SiC is enriched, the maximum depth is 115.4 nm with a residual depth of 42.2 nm—both values are higher than in layer i due to the influence of the TaC phase. Similarly, in layer iv, the SiC/TaC composite layer, two cases are observed. At point 13 where SiC is enriched, the maximum depth is 103.9 nm with a residual depth of 32.6 nm. At points 12, 14, and 15 where TaC is enriched, the maximum depth is 118.5 nm with a residual depth of 50.4 nm. In layer v (point 17) where TaC is the main component, the maximum indentation depth reaches 192.3 nm with a residual depth of 82.0 nm.

Additionally, Fig. 8 shows that at the boundaries between sub-interlayers, both maximum and residual indentation depths are higher than in adjacent sub-interlayers, as observed at point 6 (boundary between layers ii and iii) and point 11 (boundary between layers iii and iv).

Conclusions

1. Gradient-distributed SiC-TaC ceramic phases were formed along the Z-axis direction of C/C composites using isothermal CVI. From the ablation surface to the non-ablation surface of C/C composites, the ceramic phase content gradually decreases, interface thickness reduces, and the interface structure transitions from multi-layer SiC/TaC lamellar interface (Region I) to cluster-like SiC-TaC ceramic composite interface (Region II) and finally to single-layer TaC ceramic interface (Region III).

2. In Region I, the multi-layer SiC/TaC ceramic composite interface consists of five sub-layers: SiC layer (layer i), TaC layer (layer ii), TaC composite layer embedded with SiC particles (layer iii), SiC composite layer embedded with TaC phase (layer iv), and TaC layer (layer v).
3. Due to differences in elemental composition and microstructure, the nanoindentation hardness and Young' s modulus values vary significantly across sub-layers of the multi-layer SiC/TaC ceramic composite interface. Regions with higher SiC content exhibit higher hardness and modulus values, while the boundaries between sub-interlayers show relatively lower hardness and modulus.

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