

# US Patent & Trademark Office

## Patent Public Search | Text View

---

United States Patent	12391622
Kind Code	B2
Date of Patent	August 19, 2025
Inventor(s)	Jackson; Richard Wesley et al.

---

### Microstructured fiber interface coatings for composites

---

#### Abstract

Disclosed is a coated ceramic fiber including a zirconium interface coating layer deposited on the ceramic fiber, a zirconium dioxide interface coating layer adjacent to the zirconium interface coating layer, and an additional interface coating layer adjacent to the zirconium dioxide interface coating layer, wherein zirconium dioxide interface coating layer forms micro cracks after a crystal structure transformation. The coated ceramic fiber may be included in a composite material having a ceramic matrix.

---

**Inventors:** Jackson; Richard Wesley (Mystic, CT), She; Ying (East Hartford, CT), Srinivasan; Gajawalli V. (South Windsor, CT)

**Applicant:** RTX Corporation (Farmington, CT)

**Family ID:** 1000008764016

**Assignee:** RTX CORPORATION (Farmington, CT)

**Appl. No.:** 16/678833

**Filed:** November 08, 2019

#### Prior Publication Data

Document Identifier	Publication Date
US 20210139384 A1	May. 13, 2021

---

#### Publication Classification

**Int. Cl.:** C04B35/80 (20060101); C04B35/01 (20060101); C04B35/622 (20060101); C04B41/50 (20060101)

**U.S. Cl.:**

**Field of Classification Search**

**CPC:** C04B (2235/5224); C04B (2235/5228); C04B (2235/524); C04B (2235/5244); C04B (2235/5248); C04B (2235/6567); C04B (2235/76); C04B (2235/765); C04B (35/117); C04B (35/185); C04B (35/195); C04B (35/565); C04B (35/62236); C04B (35/62281); C04B (35/62855); C04B (35/62857); C04B (35/62863); C04B (35/62876); C04B (35/62884); C04B (35/62894); C04B (35/62897); C04B (35/80); C04B (41/5042)

---

**References Cited****U.S. PATENT DOCUMENTS**

<b>Patent No.</b>	<b>Issued Date</b>	<b>Patentee Name</b>	<b>U.S. Cl.</b>	<b>CPC</b>
2674445	12/1953	Diehl	N/A	N/A
3326704	12/1966	Scott et al.	N/A	N/A
4623228	12/1985	Galasso et al.	N/A	N/A
4657877	12/1986	Becher	264/333	C04B 35/185
4668579	12/1986	Strangman et al.	N/A	N/A
5110771	12/1991	Carpenter et al.	N/A	N/A
5275984	12/1993	Carpenter	428/378	C04B 41/52
5284685	12/1993	Rousseau	N/A	N/A
5455106	12/1994	Steffier	442/127	C04B 35/62897
5580643	12/1995	Kennedy et al.	N/A	N/A
5723213	12/1997	Carpenter et al.	N/A	N/A
6194083	12/2000	Yasuda et al.	N/A	N/A
6322889	12/2000	Lara-Curzio et al.	N/A	N/A
6361888	12/2001	Kriven et al.	N/A	N/A
6441341	12/2001	Steibel et al.	N/A	N/A
9133541	12/2014	Lee	N/A	N/A
12060304	12/2023	Jackson et al.	N/A	N/A
2006/0163773	12/2005	Gray	N/A	N/A
2015/0274979	12/2014	Lazur	N/A	N/A
2016/0376691	12/2015	Wadley et al.	N/A	N/A
2017/0218779	12/2016	Luthra et al.	N/A	N/A
2021/0139381	12/2020	Jackson et al.	N/A	N/A
2023/0167030	12/2022	Jackson et al.	N/A	N/A

**OTHER PUBLICATIONS**

European Search Report for European Application No. 20206017.4; Application Filing Date: Nov. 5, 2020; Date of Action: Apr. 6, 2021; 7 pages. cited by applicant

European Search Report for European Application No. 20206025.7; Application Filing Date: Nov. 5, 2020; Date of Action: Apr. 8, 2021, 8 pages. cited by applicant

Piquero et al., "Influence of Carbide Coatings on the Oxidation Behavior of Carbon Fibers", Carbon, vol. 33, No. 4, pp. 455-467, 1995. cited by applicant

Final Office Action issued in U.S. Appl. No. 16/678,852 on Jun. 10, 2022, 8 pages. cited by applicant

Non-Final Office Action issued in U.S. Appl. No. 16/678,852 on Mar. 2, 2022, 8 pages. cited by applicant

US Notice of Allowance issued in U.S. Appl. No. 16/678,852, dated Sep. 30, 2022, pp. 1-12. cited by applicant  
US Non-Final Office Action for U.S. Appl. No. 18/161,343, dated May 26, 2023, pp. 1-24. cited by applicant  
U.S. Non-Final Office Action corresponding to U.S. Appl. No. 18/595,101; Issue date, Sep. 11, 2024. cited by applicant

---

*Primary Examiner:* Rickman; Holly

*Assistant Examiner:* Chau; Linda N

*Attorney, Agent or Firm:* CANTOR COLBURN LLP

---

## **Background/Summary**

### **BACKGROUND**

- (1) Exemplary embodiments pertain to the art of composites, including ceramic matrix composites.
- (2) Composite components are finding increased use into aerospace applications due to their unique, tailorable properties which can result in significant weight savings, increased performance and durability, as well as reductions in specific fuel consumption. In particular, gas turbine engines, such as aircraft engines, operate in severe environments and show significant benefit from incorporation of composite materials. Additionally, other aerospace components, such as brakes, can benefit from incorporating composite materials.
- (3) As an example, ceramic matrix composite (CMC) components have desirable high temperature mechanical, physical, and chemical properties which allow gas turbine engines to operate at much higher temperatures with significant weight savings as compared to current engines with superalloy components. As opposed to traditional, monolithic ceramics, CMCs exhibit a significant amount of damage tolerance when under an applied load. This damage tolerance is due in part to the formation of multiple matrix cracks that aid in the redistribution of stresses to the high strength fibers.
- (4) The formation of matrix cracks can extend to the fiber and damage to the fiber may negatively impact the strength of the composite. Interface coatings have been used to address this issue. As CMCs, along with many other composites, are used under harsh environmental conditions improved interface coatings are desired.

### **BRIEF DESCRIPTION**

- (5) Disclosed is a coated ceramic fiber including a zirconium interface coating layer deposited on the ceramic fiber, a zirconium dioxide interface coating layer adjacent to the zirconium interface coating layer and an additional interface coating layer adjacent to the zirconium dioxide interface coating layer, wherein zirconium dioxide interface coating layer forms micro cracks after a crystal structure transformation.
- (6) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness of 50 nanometers (nm) to 10,000 nm, or, 100 to 1,000 nm. Additionally, in some embodiments, the zirconium interface coating layer has a thickness of 1000 nm to 20,000 nm, or, 2,000 to 10,000 nm.
- (7) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness greater than 200 nm and includes monoclinic zirconium dioxide which forms micro cracks after transforming to tetragonal zirconium dioxide and back to monoclinic zirconium dioxide.

- (8) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness less than 200 nm and includes tetragonal zirconium dioxide which forms micro cracks after transforming to monoclinic zirconium dioxide.
- (9) Also disclosed is a composite material including a ceramic matrix material and a plurality of ceramic fibers embedded within the matrix material. The plurality of fibers includes a multi-layer interface coating. The multi-layer interface coating includes a zirconium interface coating layer deposited on the plurality of fibers, and a zirconium dioxide interface coating layer adjacent to the zirconium interface coating layer and an additional interface coating layer adjacent to the zirconium dioxide interface coating layer, wherein the zirconium dioxide interface coating layer forms micro cracks after a crystal structure transformation.
- (10) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness greater than 200 nm and includes monoclinic zirconium dioxide which forms micro cracks after transforming to tetragonal zirconium dioxide and back to monoclinic zirconium dioxide.
- (11) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness less than 200 nm and includes tetragonal zirconium dioxide which forms micro cracks after transforming to monoclinic zirconium dioxide.
- (12) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness of 50 nanometers (nm) to 10,000 nm, or, 100 to 1,000 nm. Additionally, in some embodiments, the zirconium interface coating layer has a thickness of 1000 nm to 20,000 nm, or, 2,000 to 10,000 nm.
- (13) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the additional interface coating layer can have the same composition as the zirconium interface coating layer. Alternatively, the additional interface coating layer can have a composition different from the zirconium interface coating layer.
- (14) Also disclosed is a method for forming a multi-layer interface coating onto a ceramic fiber including depositing a zirconium interface coating layer onto the ceramic fiber, oxidizing a portion of the zirconium interface coating layer to form a zirconium dioxide interface coating layer, depositing an additional interface coating layer on the zirconium dioxide interface coating layer and heat treating the multilayer coating such that the zirconium dioxide interface coating layer forms micro cracks after a crystal structure transformation.
- (15) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness greater than 200 nm and includes monoclinic zirconium dioxide which forms micro cracks after transforming to tetragonal zirconium dioxide and back to monoclinic zirconium dioxide.
- (16) In addition to one or more of the features described above, or as an alternative to any of the foregoing embodiments, the zirconium dioxide interface coating layer has a thickness less than 200 nm and includes tetragonal zirconium dioxide which forms micro cracks after transforming to monoclinic zirconium dioxide.

---

## Description

### BRIEF DESCRIPTION OF THE DRAWINGS

(1) The following descriptions should not be considered limiting in any way. With reference to the accompanying drawing, like elements are numbered alike:

(2) The FIGURE shows a representative interface coating and method of making.

### DETAILED DESCRIPTION

- (3) A detailed description of one or more embodiments of the disclosed coated fiber and method are presented herein by way of exemplification and not limitation with reference to the FIGURE.
- (4) Composites exhibit a significant amount of damage tolerance when under an applied load. In ceramic matrix composites (CMC) in particular, this damage tolerance is due to the formation of multiple matrix cracks that aid in the redistribution of stresses. However, the formation of matrix cracks can result in environmental exposure to the fiber and fiber interface coating. The fiber interface coating protects the fiber from the environmental exposure and provides a weak interface to allow for composite behavior.
- (5) The multi-layer interface coating described herein provides a weak interface with the fiber to allow for composite behavior and also provides environmental protection. The multiple layers can prevent crack propagation from reaching the fibers and protect the fiber from environmental degradation. In particular, the zirconium dioxide interface coating layer is a crystalline material which goes through a crystal structure transformation under conditions of normal use or heat treatment. The crystal structure transformation results in the presence of micro cracks which can deflect any matrix cracks from reaching the fiber.
- (6) Referring now to the FIGURE, a coated ceramic fiber for use in a composite is shown. The coated ceramic fiber includes ceramic fiber **12** and a multi-layer interface coating **22**. The multi-layer interface coating **22** as shown includes three coating layers **24**, **32** and **28**. Exemplary ceramic fiber materials include silicon carbide, carbon, hafnium carbide, zirconium carbide, tantalum carbide, niobium carbide, boron carbide, titanium carbide, hafnium boride, zirconium boride, tantalum boride, niobium boride, titanium boride, aluminum oxide, mullite, and combinations thereof.
- (7) The first interface layer **24** includes zirconium. A portion of the first interface layer is oxidized to form an initially formed zirconium dioxide layer **30**. The third interface layer **28** (the additional interface layer) may include silicon carbide, zirconium, zirconium boride or a combination thereof.
- (8) The first interface layer may have a thickness of 1000 nm to 20,000 nm, or, 2,000 nm to 10,000 nm. The second interface layer may have a thickness of 50 nm to 10,000 nm, or, 100 nm to 1,000 nm. The third interface layer may have a thickness of 1000 nm to 20,000 nm, or, 2000 nm to 10,000 nm.
- (9) The first interface layer is deposited on the ceramic fiber by chemical vapor deposition (shown as step **110**). The combination of the first interface layer and the ceramic fiber are then heat treated in the presence of oxygen to oxidize a portion of the first interface layer to form an initially formed zirconium dioxide layer **30** (shown as step **120**). If the zirconium dioxide layer **30** has a thickness less than 200 nm then the zirconium dioxide crystal structure may be tetragonal and after heating to a temperature greater than or equal to 1000° C. and cooling will transform to a monoclinic crystal structure with micro cracks. If the zirconium dioxide layer **30** has a thickness greater than 200 nm then the crystal structure may be monoclinic which will convert to tetragonal at a temperature greater than or equal to 1000° C. and return to monoclinic with micro cracks upon cooling.
- (10) In some embodiments an interdiffusion layer forms between the zirconium interface coating layer and the ceramic fiber.
- (11) An additional interface coating layer **28** is deposited on the initially formed zirconium dioxide layer **30** (shown as step **130**) after which the fiber with the multilayer coating is subjected to a heat treatment (shown as step **140**) to change the crystal structure of the initially formed zirconium dioxide layer. Exemplary additional interface coating layer materials include zirconium, silicon carbide, zirconium boride or combinations thereof. The additional interface coating layer **28** may be deposited by chemical vapor deposition for example although other methods could be employed. Exemplary matrix materials include silicon carbide, aluminum oxide, mullite, cordierite, and combinations thereof.
- (12) Exemplary heat treatment temperatures (step **130**) are 1250 to 1800° C., or, 1250 to 1400° C. Exemplary times for the heat treatment include 1 minute to 100 hours, or, 10 minutes to 5 hours.

(13) The micro cracks in the zirconium dioxide interface coating layer provide protection to the fiber from matrix cracking by deflecting the cracks into the second interface layer. The first interlayer provides environmental protection to the fiber so the combination of layers results in a composite with more robust properties. The additional interface coating layer protects the micro cracked zirconium oxide interface layer.

(14) The terminology used herein is for the purpose of describing particular embodiments only and is not intended to be limiting of the present disclosure. As used herein, the singular forms “a”, “an” and “the” are intended to include the plural forms as well, unless the context clearly indicates otherwise. The term “adjacent to” is defined as being in contact with the underlying material. It will be further understood that the terms “comprises” and/or “comprising,” when used in this specification, specify the presence of stated features, integers, steps, operations, elements, and/or components, but do not preclude the presence or addition of one or more other features, integers, steps, operations, element components, and/or groups thereof.

(15) While the present disclosure has been described with reference to an exemplary embodiment or embodiments, it will be understood by those skilled in the art that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the present disclosure. In addition, many modifications may be made to adapt a particular situation or material to the teachings of the present disclosure without departing from the essential scope thereof. Therefore, it is intended that the present disclosure not be limited to the particular embodiment disclosed as the best mode contemplated for carrying out this present disclosure, but that the present disclosure will include all embodiments falling within the scope of the claims.

## Claims

1. A coated ceramic fiber comprising: a zirconium interface coating layer consisting of zirconium metal deposited on the ceramic fiber, a zirconium dioxide interface coating layer directly adjacent to the zirconium interface coating layer, and an additional interface coating layer directly adjacent to the zirconium dioxide interface coating layer, wherein the zirconium dioxide interface coating layer forms micro cracks after a crystal structure transformation; wherein the additional interface coating layer consists of zirconium and silicon carbide, zirconium boride, or a combination thereof; wherein the zirconium dioxide interface coating layer has a thickness greater than 200 nm and includes monoclinic zirconium dioxide which forms micro cracks after transforming to tetragonal zirconium dioxide and back to monoclinic zirconium dioxide; or wherein the zirconium dioxide interface coating layer has a thickness less than 200 nm and includes tetragonal zirconium dioxide which forms micro cracks after transforming to monoclinic zirconium dioxide.
2. The coated ceramic fiber of claim 1, wherein the zirconium interface coating layer has a thickness of 1000 nm to 20,000 nm.
3. The coated ceramic fiber of claim 1, wherein the additional interface coating layer has the same composition as the zirconium interface coating layer.
4. The coated ceramic fiber of claim 1, wherein the additional interface coating layer has a different composition from the zirconium interface coating layer.
5. A composite material comprising a ceramic matrix material and a plurality of ceramic fibers embedded within the ceramic matrix material wherein the plurality of ceramic fibers comprises a multi-layer interface coating and the multi-layer interface coating comprises a zirconium interface coating layer consisting of zirconium metal deposited on the plurality of ceramic fibers, a zirconium dioxide interface coating layer directly adjacent to the zirconium interface coating layer and an additional interface coating layer directly adjacent to the zirconium dioxide interface coating layer, wherein the zirconium dioxide interface coating layer forms micro cracks after a crystal structure transformation; wherein the additional interface coating layer includes silicon carbide, zirconium boride, or a combination thereof; wherein the ceramic matrix material includes

mullite, cordierite, or a combination thereof; wherein the zirconium dioxide interface coating layer has a thickness greater than 200 nm and includes monoclinic zirconium dioxide which forms micro cracks after transforming to tetragonal zirconium dioxide and back to monoclinic zirconium dioxide; or wherein the zirconium dioxide interface coating layer has a thickness less than 200 nm and includes tetragonal zirconium dioxide which forms micro cracks after transforming to monoclinic zirconium dioxide.

6. The composite material of claim 5, wherein the zirconium interface coating layer has a thickness of 1000 nm to 20,000 nm.

7. The coated ceramic fiber of claim 5, wherein the additional interface coating layer has the same composition as the zirconium interface coating layer.

8. The coated ceramic fiber of claim 5, wherein the additional interface coating layer has a different composition from the zirconium interface coating layer.

---