

第三代 SiC 纤维及其在核能领域的应用现状

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摘要: 第三代 SiC 纤维具有近化学计量比的元素组成和高结晶致密的特性, 与第一、第二代 SiC 纤维相比, 在耐高温、抗氧化、抗蠕变及抗辐射等性能上均有明显的提升, 因此在工程应用上尤其在核能领域拥有更明显的优势和更广阔的前景。本文对第三代 SiC 纤维的制备工艺、性能特点进行了介绍和比较, 综述了第三代 SiC 纤维在核能领域的应用, 并对其发展前景进行了展望。

关键词: SiC 纤维; 第三代; 核能应用; 综述

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Third Generation SiC Fibers for Nuclear Applications

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Abstract: The third generation SiC fibers have near-stoichiometric composition and polycrystallinity with high density. Compared with the first and second generations, they have obvious improvements in heat-resistance, creep-resistance and radiation-resistance. Accordingly, they have more advantages and broader prospects in engineering applications, especially in the nuclear field. In this paper, the fabrication and performance characteristics of the third generation SiC fibers are introduced and compared. The applications of the third generation SiC fibers in the field of nuclear energy are reviewed, and the development prospects are prospected.

Key words: SiC fibers; third generation; nuclear application; review

连续 SiC 纤维具有高强度、高模量、耐高温抗氧化、辐照条件下的活性低等优异性能^[1-3], 自从 1975 年 Yajima 等^[4]以聚碳硅烷(Polycarbosilane, PCS)为先驱体制备细直径连续 SiC 纤维以来, SiC 纤维得到了迅猛发展, 到目前为止已经从富碳富氧且处于无定形态的第一代 SiC 纤维发展到具有近化学计量比和高结晶特性的第三代 SiC 纤维^[5]。目前市售第一代 SiC 纤维以日本碳公司(Nippon Carbon Co. Ltd.)生产的 Nicalon 200、日本宇部兴产公司(Ube Industries)生产的 Tyranno Lox-M 和国防科技大学生产的 KD-I 为代表,

由于氧含量偏高(~12wt%), SiC 纤维在 1200 °C 以上强度迅速下降^[6-9]。通过改进纤维交联工艺, 第二代 SiC 纤维实现了无氧不熔化, 显著降低了氧含量(可达 1wt% 以下), 在惰性气氛下可以在 1500 °C 以下保持较高的拉伸强度和模量, 典型代表有 Hi-Nicalon、Tyranno ZE、KD-II 等, 但由于碳含量偏高(C/Si≈1.4), 其抗氧化性能仍然不够理想^[5,10-12]。因此, 日本碳公司、宇部兴产公司和美国 Dow Corning 公司分别采用不同的技术路线研制出了近化学计量比的第三代 SiC 纤维, 商品号分别为 Hi-Nicalon S、Tyranno SA

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和 Sylramic(以及 Sylramic-iBN), 中国国防科技大学则研制出了 KD-S(与 Hi-Nicalon S 类似)和 KD-SA(与 Tyranno SA 类似)两种第三代 SiC 纤维。

从图 1 中典型 SiC 纤维的 TEM 照片可以看出, Nicalon、Hi-Nicalon 和 Hi-Nicalon S 纤维的晶粒依次增大, 分别约为 5、10 和 100 nm^[1]。

表 1 列出了所有三代 SiC 纤维的组成和力学特性, 总体来看, 三代 SiC 纤维在拉伸强度上并没有明显区别, 均在 3.0 GPa 左右, 而在杨氏模量上则是依次升高。第一代 SiC 纤维中包含大量富余碳和氧, 其结构由 β -SiC 微晶(<5 nm)、自由碳和无定型相 SiC_xO_y 组成^[19-20]; 第二代 SiC 纤维富碳, 主要由 β -SiC 微晶(5~10 nm)和自由碳组成^[5]; 第三代 SiC 纤维在组成上是近化学计量比, 由大尺寸的 β -SiC 晶粒(达到 100 nm 以上)形成致密结构^[1]。研究表明, 由于存在 SiC_xO_y 晶间相和自由碳限制了 SiC 结晶, 高温下 SiC_xO_y 晶间相容易分解、滑移, 自由碳易氧化, 因此第三代 SiC 纤维与前两代相比, 耐温性、抗氧

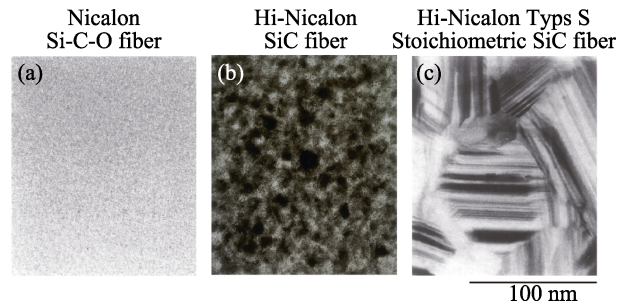


图 1 三代 SiC 纤维的 TEM 照片^[1]

Fig. 1 TEM images of three generation SiC fibers^[1]
(a) Nicalon fiber; (b) Hi-Nicalon fiber; (c) Hi-Nicalon S fiber

化性和抗蠕变性能都显著升高^[21-25]。

图 2 是用 BSR(Bend Stress Relaxation)法测得不同纤维的抗蠕变性能, 其中 m 值越大表明纤维的高温抗蠕变性能越好。从图 2 中可以看出, 在相同温度下, 第三代 SiC 纤维的 m 值明显大于其它两代纤维, 其高温抗蠕变性能大幅增强。图 3 表明当温度高于 1400 °C 时, 前两代纤维的拉伸强度迅速下降, 而 Tyranno SA 纤维的强度可以保持到 1900 °C 以上。

表 1 三代 SiC 纤维的组成和力学特性^[5,13-18]

Table 1 Compositions and mechanical properties of three generations SiC fibers^[5,13-18]

	Trade mark	Tensile strength/GPa	Young's modulus/GPa	Diameter/ μm	C/Si
First generation	Nicalon 200	3.0	200	14	1.33
	Tyranno Lox-M	3.3	185	11	1.38
	KD-I	>2.5	>170	11.5	1.29
Second generation	Hi-Nicalon	2.8	270	12	1.39
	Tyranno ZE	3.5	233	11	1.34
	KD-II	>2.7	>250	11.5	1.35-1.40
Third generation	Hi-Nicalon S	2.6	340	12.0	1.05
	KD-S	2.7	310	11.0	1.08
	Tyranno SA	2.8	375	8.0&10.0	1.08
	KD-SA	2.5	350	10.5	1.05
	Sylramic	3.2	400	10.0	1.01

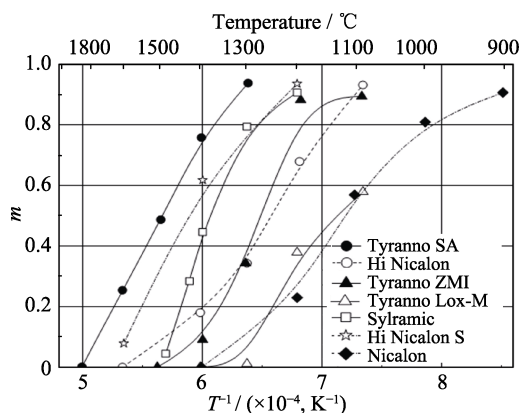


图 2 三代 SiC 纤维的高温抗蠕变性能^[21,26-27]

Fig. 2 High-temperature creep-resistance of three generation SiC fibers^[21,26-27]

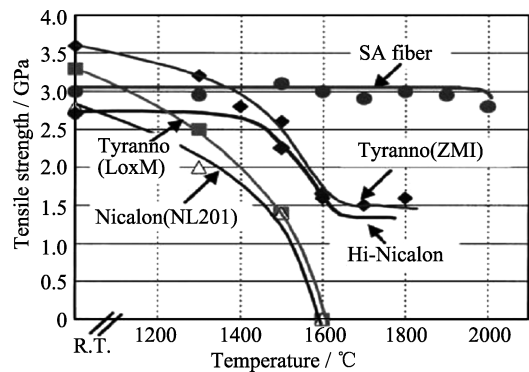


图 3 高温处理 SiC 纤维后的拉伸强度^[25]

Fig. 3 Heat-resistance of the three generations SiC fibers^[25]

Sylramic-iBN 纤维晶粒更大, 晶界更干净, 抗蠕变性能和电导率进一步提高, 抗氧化性也得到增强^[33]。

总之, 第三代 SiC 纤维不仅 C/Si 比接近化学计量比、氧含量更低, 而且 Tyranno SA、KD-SA 和 Sylramic 纤维等具有高结晶致密的特性, 使其具有更好的耐高温和高温抗蠕变性能。

2 第三代 SiC 纤维在复合材料制备工艺上的优势

连续 SiC 纤维增强 SiC 复合材料(SiC_f/SiC)主要应用于高温、强氧化、强腐蚀及辐照条件下的结构部件, 如航空涡轮发动机叶片、喷气式发动机燃烧机的内衬、核聚变反应堆包层结构、包层流道内衬和转化器等^[34-37]。随着应用领域技术的发展, 对 SiC_f/SiC 复合材料的耐温性、抗氧化性、热导率和气密性等性能的要求越来越高。陶瓷基复合材料的制备工艺经过几十年的发展已经趋于成熟, 目前制备 SiC_f/SiC 复合材料的主要工艺有化学气相渗透(Chemical Vapor Infiltration, CVI)、先驱体浸渍裂解(Polymer Infiltration and Pyrolysis, PIP)、熔渗(Melt Infiltration, MI)和纳米浸渍瞬态共晶相(Nano-powder Infiltration and Transient Eutectoid, NITE)等。

从制备工艺的角度来看, 第三代 SiC 纤维同样具有更大的优势。以第一、二代 SiC 纤维为增强体时, 一般采用制备温度较低(900~1200 °C)的 CVI 和 PIP 工艺, 这两种工艺在制备过程中, 纤维不需要经历 1400 °C 以上高温, 所得 SiC_f/SiC 也就不具备高结晶、高致密化的特征, 因此无法满足高温应用领域的要求。Riccardi 等^[38]采用 CVI 工艺制备了 2D 和 3D Tyranno SA/SiC 复合材料, 其杨氏模量分别为 293 和 198 GPa, 剪切强度分别为 54 和 45 MPa。MI 工艺制备温度高于 Si 的熔点(1410 °C), 一般在 1400~1600 °C 范围内^[39-40], 对纤维损伤较大, 目前只有 Tyranno SA、KD-SA 和 Sylramic-iBN 第三代纤维的制备温度

高于 MI 工艺温度, 因此纤维在制备复合材料的过程中损伤较小。Morscher 等^[41]对 MI 工艺制备的 Sylramic/SiC 复合材料进行了抗蠕变和疲劳测试, 结果表明, 该材料在 220 MPa 的应力水平下可以保持 500 h 不失效。类似 MI 的方法也被用于 SiC_f/SiC 部件的反应连接(Reaction bonding), 要求所连接的部件中 SiC 纤维能够承受 1500 °C 高温^[42]。同样地, NITE 工艺要在 1700~1800 °C 的高温 and 15~20 MPa 的高压条件下进行, 现有连续 SiC 纤维中只有 TyrannoSA、KD-SA 和 Sylramic 满足 NITE 工艺过程中的高温高压条件。在用 NITE 方法制备复合材料的研究方面, 已有采用 TyrannoSA 纤维作为增强体的研究报道^[43-44]。Kishimoto 等^[45]研究了 NITE-Tyranno SA/SiC 复合材料的辐照性能(辐照粒子为 Si 离子和 He 离子, 温度为 1200 °C, 剂量为 60 dpa), 除了产生少量的微腔外, 其他无明显变化, 表现出良好的稳定性。

表 2 统计了不同型号 SiC/SiC 复合材料在室温和高温的性能, 可以看出第三代 Sylramic、Sylramic-iBN 和 Hi-Nicalon S 纤维制备的复合材料可以在更高的温度下保持稳定。

3 第三代 SiC 纤维在核能领域的应用

核聚变反应堆的结构材料长期处于高温、高辐照和高应力的苛刻条件下, 以含有大量 SiC_xO_y 无定型相或 β-SiC 微晶(<10 nm)为特点的第一和第二代 SiC 纤维制备的 SiC_f/SiC 复合材料, 经辐照后会引发 SiC_xO_y 相的分解、无定型态的结晶和 β-SiC 晶粒的长大(图 6), Nicalon 纤维和 Hi-Nicalon 纤维经辐照后, 纤维的密度均出现了增大, 意味着纤维体积收缩严重, 易导致纤维和基体剥离, 最终使 SiC_f/SiC 失效^[47-48]。第三代 SiC 纤维具有近化学计量比的组成, 几乎不含 SiC_xO_y 相, 并且结晶度高(图 6), Tyranno SA 纤维与 CVD 法制备的 SiC 经辐照后密度基本不变, 说明由二者制备 SiC_f/SiC 复合材料在辐照条件下结构稳定,

表 2 不同型号 SiC_f/SiC 复合材料及性能^[46]

Table 2 Different SiC_f/SiC composites and their properties^[46]

Brand name	Fiber type	Preparation technology	Tensile strength at room temperature / MPa	Failure duration
Hypercomp PP-HN	Hi-Nicalon	MI	321	>1000 h/1200 °C
Hypercomp SC-HN	Hi-Nicalon	MI	358	>1000 h/1200 °C
N22	Sylramic	CVI+MI	400	~500 h/1204 °C
N24-A	Sylramic-iBN	CVI+MI	450	~500 h/1315 °C
N24-B	Sylramic-iBN	CVI+MI	450	~500 h/1315 °C
N24-C	Sylramic-iBN	CVI+MI	310	>1000 h/1315 °C
N26	Sylramic-iBN	CVI+PIP	330	~300 h/1450 °C
A410	Hi-Nicalon	CVI	200-315	600 h/1200 °C
A416	Hi-Nicalon S	CVI	200-315	200 h/1400 °C

更有利于在核能领域的应用。但是 Sylramic-iBN 纤维中含的 B、N 元素在辐射环境中会发生嬗变并产生长寿命的同位素, 不适宜用于聚变反应堆结构材料^[49]。以 Tyranno SA 和 Hi-Nicalon S 纤维作为增强体, 在辐照条件下制备 SiC_f/SiC 复合材料的研究报道^[50], 主要是应用在包层的第一壁、流道插件、控制棒及偏滤器等部件^[35,51-53]。

同样地, Katoh 等^[54]用 Hi-Nicalon S 和 Tyranno SA 制备的 CVI SiC_f/SiC 试样与 CVD 工艺制备的纯 SiC 进行了不同辐照条件下的性能对比, 它们的体积膨胀规律基本相同, 并且膨胀率很低(图 7); JONES 等^[55]研究表明, 在 800 °C、10 dpa 的辐照条件下, 用 Hi-Nicalon S 和 Tyranno-SA 制备 SiC_f/SiC 试样的性能基本无变化。

反应堆中的结构材料承受高的热载荷, 具有高的热导率(λ)有利于减少热应力, 所以希望获得具有较高热导率的 SiC_f/SiC 复合材料^[56]。而使用 CVI 工艺制备的 Nicalon 纤维增强 SiC 基复合材料在室温下的热导率仅为 10 W/(m·K), PIP 工艺制备的 SiC_f/SiC 复合材料的热导率更低, 不能很好地满足核反应堆对结构材料的要求^[57-60]。第三代 SiC 纤维热导

率则较高, Hi-Nicalon S 为 18 W/(m·K), Tyranno SA 更是高达 65 W/(m·K)。Yamada 等^[61]分别以 Tyranno SA 和 Hi-Nicalon S 为增强体, 通过 CVI 方法制备了 3D SiC_f/SiC 复合材料, 并研究了热导率。前者在室温和 1000 °C 下的热导率分别为 40~50 和 24 W/(m·K), 后者则分别为 36 和 20 W/(m·K)。

西方发达国家十分重视 SiC_f/SiC 在核聚变领域的研究, 日本也凭借 SiC 纤维研制方面的优势, 积极与欧美合作。如日本的 DREAM 和 A-SSTR2 包层概念设计选用 SiC_f/SiC 复合材料作为第一壁/包层结构材料^[62-63]; 欧盟的 PPCS(the Power Plant Conceptual Study)包层概念设计采用 SiC_f/SiC 复合材料制造流道插件^[64]; 美国的 ARIES-AT 的偏滤器设计采取 SiC_f/SiC 复合材料作为结构材料^[65-66]。

日本使用 Tyranno SA 纤维通过 NITE 工艺制备了若干 SiC_f/SiC 复合材料构件。如 Satori 等^[67]制备了夹层结构的 SiC_f/SiC 复合材料隔热面板(图 8), 研究了孔的结构与热导率的关系, 对应用于核聚变反应的隔热系统有指导意义; Kishimoto 等^[68]制备了

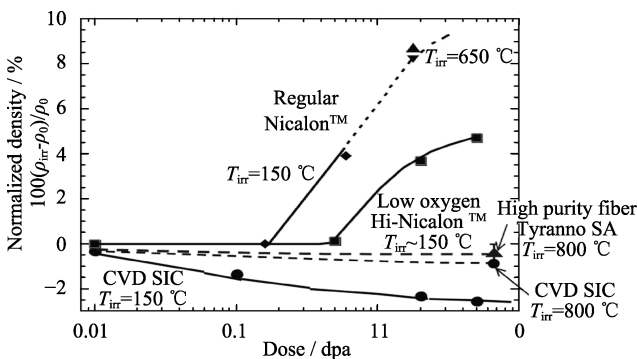


图 6 SiC 纤维和 CVD-SiC 经中子辐照后相对密度的变化^[47]
Fig. 6 Relative density change of SiC fibers and CVD-SiC by neutron irradiation^[47]

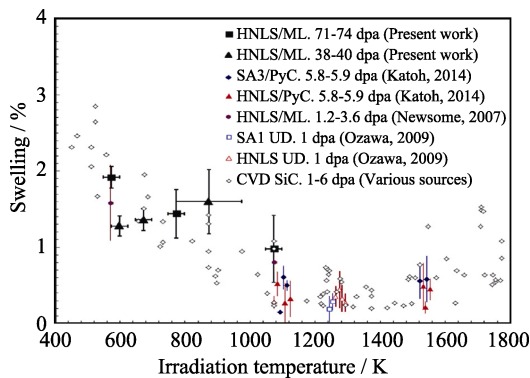


图 7 辐照后 CVI-Hi-Nicalon S /SiC 复合材料和 CVD-SiC 的体积膨胀率^[54]
Fig. 7 Swelling of Hi-Nicalon S, CVI SiC-matrix composites plotted against irradiation temperature^[54]

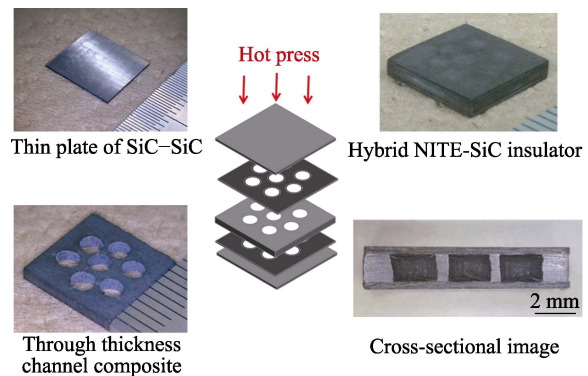


图 8 混合型 NITE-SiC_f/SiC 复合材料隔热面板的制备过程^[67]
Fig. 8 Process of hybrid NITE-SiC insulator^[67]

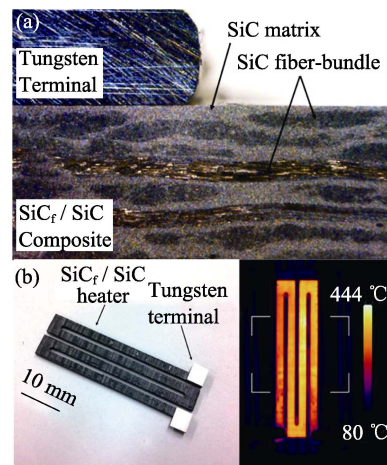


图 9 SiC_f/SiC 复合材料加热器的剖面照片(a)和实物照片及红外图像(b)^[68]
Fig. 9 (a) Sectional view of SiC_f/SiC heater with tungsten terminal, (b) SiC_f/SiC heater for BR2 with IR image^[68]

SiC_f/SiC 复合材料加热器(图 9), 材料的电导率在室温到 1000 °C 的范围内受温度变化影响较小, 且经过 1000 °C/1 h 空气中热处理后电导率变化不大, 为加热器的精确控温提供了良好的基础, 可应用于核裂变堆辐照部件。

4 结束语

第三代 SiC 纤维拥有比前两代更优异的耐温性能, 使其在制备 SiC_f/SiC 复合材料时可以经受更高的制备温度, 提高了复合材料的性能; 同时近化学计量比、高结晶的特性也使其在辐照条件下能够保持自身结构稳定, 在核能领域的应用具有明显优势。

目前第三代 SiC_f/SiC 复合材料在核能领域的应用方面需要关注以下几点:

1) 虽然第三代 SiC 纤维和 SiC 基体在辐照条件下比较稳定, 但其界面相(常见有 PyC 界面相、BN 界面相和复合界面相)却不稳定, 改进界面相的耐辐照性能十分关键;

2) 气密性是 SiC_f/SiC 复合材料在核能领域应用中需要考察的重要性能, 随着核技术的发展, 应用环境条件更加苛刻, 对气密性的要求也会越来越高;

3) SiC_f/SiC 复合材料与冷却剂和增殖剂的化学相容性对其服役寿命以及核反应堆的能量转换效率有重要影响, 需要进一步研究;

4) 在工业化生产中需进一步降低成本。

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