学校编码：10384 密级_____

学号：20720071150003

厦门大学

硕 士 学 位 论 文

显微 CT 在 C/SiC 微结构表征中的初步应用

The Preliminary Application of MicroCT on Microstructure Characterization of C/SiC

冯 炎 建

指导教师姓名： 冯 祖 德 教授

专 业 名 称： 材 料 学

论文提交日期： 2010 年 11 月

论文答辩日期： 2010 年 11 月

学位授予日期： 2010 年 12 月

2010 年 11 月

The Preliminary Application of MicroCT on Microstructure Characterization of C/SiC

Thesis Submitted to

Xiamen University

In partial fulfillment of the requirement

for the degree of

Master of Engineering

By

Yanjian Feng

(Materials Science)

Supervisor: Prof. **Zude Feng**

November 2010

厦门大学学位论文原创性声明

本人呈交的学位论文是本人在导师指导下，独立完成的研究成果。本人在论文写作中参考其他个人或集体已经发表的研究成果，均在文中以适当方式明确标明，并符合法律规范和《厦门大学研究生学术活动规范(试行)》。

另外，该学位论文为(　　)课题(组)的研究成果，获得(　　)课题(组)经费或实验室的资助，在(　　)实验室完成。(请在以上括号内填写课题或课题组负责人或实验室名称，未有此项声明内容的，可以不作特别声明。)

声明人(签名)：

年　　月　　日

厦门大学学位论文著作权使用声明

本人同意厦门大学根据《中华人民共和国学位条例暂行实施办法》等规定保留和使用此学位论文，并向主管部门或其指定机构送交学位论文(包括纸质版和电子版)，允许学位论文进入厦门大学图书馆及其数据库被查阅、借阅。本人同意厦门大学将学位论文加入全国博士、硕士学位论文共建单位数据库进行检索，将学位论文的标题和摘要汇编出版，采用影印、缩印或者其它方式合理复制学位论文。

本学位论文属于：

(　　)1.经厦门大学保密委员会审查核定的保密学位论文，

于　　年　　月　　日解密，解密后适用上述授权。

(　　)2.不保密，适用上述授权。

(请在以上相应括号内打“√”或填上相应内容。保密学位论文应是已经厦门大学保密委员会审定过的学位论文，未经厦门大学保密委员会审定的学位论文均为公开学位论文。此声明栏不填写的，默认为公开学位论文，均适用上述授权。)

声明人(签名)：

年　　月　　日

摘 要

连续纤维增韧碳化硅陶瓷基复合材料(CMC-SiCs)具有耐高温、低密度、高比模、抗氧化和抗烧蚀等优异性能，使其成为新一代高温结构材料。化学气相渗透法(CVI)是目前唯一商业化制造 CMC-SiCs 的方法，但 SiC 基体的致密化周期长，非均匀沉积产生密度梯度，尤其是材料内部的残余孔隙，显著影响材料机械性能。对 CMC-SiCs 开展微观结构研究有助于改进 CVI 工艺，提升材料机械性能。而传统平面分析技术如扫描电子显微镜(SEM)和透射电子显微镜(TEM)的试样制备过程往往易破坏材料的本征结构，且信息局限于二维尺度，阻碍了结构研究的力度。因此，利用无损技术对 CMC-SiCs 进行三维尺度的结构观察具有重要的意义。

本文以 CVI 法制备的碳纤维增韧碳化硅陶瓷基复合材料(C/SiC)为研究对象，采用显微 CT 为主要研究手段，系统观察了 C/SiC 在不同尺度的三维结构特征，分析了 C/SiC 的孔隙形成机制和 SiC 沉积形貌的影响因素；探索了 C/SiC 高温蠕变前后孔隙率及微裂纹的变化；研究了干氧和湿氧环境下 C/SiC 材料表面 SiC 涂层的氧化行为，为探索 C/SiC 服役性能变化及其制备改性提供了实证数据。主要内容与结果如下：

1、观察了 2D C/SiC、3D C/SiC、碳布沉积体、Mini-C/SiC 的重构形貌。结果表明：显微 CT 能有效地分辨碳纤维预制体的沉积形貌以及在不同热处理条件下的氧化形貌，能有效检测 C/SiC 内部微米级尺度的缺陷。

2、重构了 3D C/SiC 的孔隙形貌，探讨了 CVI 过程的气体传输特征。结果表明：节点树枝状孔隙结构是由于 CVI 过程中预制体对沉积气体的传输存在滞留作用造成的，预制体内外压力差对 SiC 沉积物的表面颗粒尺寸有显著影响。

3、探索了含 B_xC 基体改性 2D C/SiC 试样高温蠕变前后的孔隙率及微裂纹变化。结果表明：自愈合组元 B_xC 对孔隙有愈合作用，蠕变过程中材料表面的裂纹仅在垂直于蠕变应力方向的扩展，而在数量上没有增加。

4、研究了 1300ºC 干氧和湿氧环境中 3D C/SiC 表面 SiC 涂层的氧化特征。结果表明：氧化产物 SiO_2 沿 SiC 涂层表面和厚度方向皆呈非均匀分布，干氧条件下非均匀氧化的深度可达约 19μm，而湿氧中的非均匀氧化深度约为干氧条件的 1.5 倍。

关键词：显微 CT　C/SiC 复合材料　微观结构

Abstract

Continuous fiber reinforced silicon carbide matrix composites (CMC-SiCs), are considered as the most promising thermo-structural materials due to their long durability, high specific strength, high specific modulus, enhanced oxidation resistance, good ablation resistance. Chemical vapor infiltration (CVI) has been demonstrated to be a very effective and matured enough preparation method to fabricate CMC-SiCs. However, the main problems associated with CVI are the long time periods that are usually required to reach the desired densification level, the nonuniformity of the densification profile in the final product, and particularly the densification process is incomplete, leaving a residual porosity in the composite that has a strong effect on its mechanical behavior. Thus, research on the microstructure of CMC-SiCs helps improve the CVI process and enhance the mechanical properties. Traditional imaging techniques namely scanning electron microscopy (SEM) and transmission electron microscopy (TEM) which usually destroy intrinsic structure of the sample during sample preparation, can only reveal the surface or two-dimensional morphology. Furthermore, the two-dimensional sections provide limited information for structure research. Consequently, it is very significant for do research on nondestructive characterization three-dimensional structure CMC-SiCs.

In this thesis, targeting carbon fiber reinforced SiC-matrix composites (C/SiC) fabricated by CVI, the microstructure of C/SiC was mainly characterized using commercial X-ray microcomputed tomography (MicroCT). Three dimensional structure features of C/SiC in different scales were observed systematically. The formation mechanism of porosity and the influence factors on the deposition morphology of SiC was investigated. The variation of porosity and microcrack in C/SiC composite under high temperature creep environment were explored. The oxidation behavior of C/SiC composite under dry and wet oxidation environment was investigated. It provides empirical data for exploring the service performance and improving preparation process. The major results are listed below:

1、The deposition morphology of 2D C/SiC and 3D C/SiC and Mini-C/SiC and carbon fabric composite were observed. Results show that MicroCT can facilitate characterization of the fabric and the oxidation morphology of C/SiC in different heat treatment conditions. And the internal geometry of flaws in micrometer scale in C/SiC were examined effectively by MicroCT.

2、Three-dimensional structure of 3D C/SiC composites was reconstructed, and the gas transport property during CVI was discussed. Results show that the“node-bond” porosity structure was revealed for a gas retention and viscous flow formation at the preform in CVI. Pressure difference in the preform has significant influence on the particle size of surface morphology of SiC deposition.

3、The variation of porosity and the microcracking in 2D C/SiC composites with boron carbide self-sealing matrix composites under high temperature creep environment was investigated. Results show that the boron carbide self-sealing matrix has influence the porosity, and surface microcrack only propagate longitudinally.

4、The oxidation morpology of 3D C/SiC composites annealed in 1300ºC dry and wet oxidizing environment were investigated. Results show that inhomogeneous oxidation in the SiC coating is identified by reconstructed images of the surface coating at different depth. There exists oxidant products of SiO_2 at the 19μm depth of SiC coating were confirmed in the dry oxygen. And the oxidation depth in wet oxygen was 1.5 times deeper than in dry oxygen.

Key Words: MicroCT; C/SiC composite; Microstructure

目 录

摘 要 I

Abstract II

目 录 IV

Table of Contents VII

第一章 绪论 1

1.1 研究背景 1

1.1.1 碳化硅陶瓷基复合材料(CMC-SiCs) 1

1.1.2 CMC-SiCs微结构研究现状与发展趋势 2

1.2 显微CT技术 3

1.2.1 显微CT原理 3

1.2.2 显微CT性能参数 8

1.2.3 显微CT应用领域 11

1.3 显微CT在复合材料微观结构表征中的应用进展 14

1.3.1 同步辐射显微CT在复合材料微结构表征中的应用进展 14

1.3.2 常规显微CT在复合材料微结构表征的应用进展 16

1.4 本文的选题依据和研究目标 18

1.4.1 选题依据 18

1.4.2 研究目标 18

1.5 本文研究思路 19

1.6 本文研究内容 19

参考文献 20

第二章 实验仪器与实验方法 27

2.1 主要实验仪器 27

2.2 实验样品 27

2.3 实验方法 29

2.3.1 显微CT分析 29
2.3.2 扫描电镜(SEM)和能谱(EDS)分析 30
2.3.3 X射线衍射(XRD)物相分析 30
参考文献 31
第三章 结果与讨论 32
3.1 显微CT对C/SiC织构观察分析 32
3.1.1 2D C/SiC织构重构观察 32
3.1.2 3D C/ SiC织构重构观察 36
3.1.3 碳布沉积体重构观察 39
3.1.4 Mini-C/SiC织构重构观察 41
3.1.5 本节小结 44
3.2 3D C/SiC孔隙结构重构分析 45
3.2.1 孔隙结构二维重构分析 45
3.2.3 孔隙结构三维重构分析 50
3.2.3 孔隙壁重构分析 52
3.2.4 本节小结 57
3.3 2D C/SiC高温蠕变后的孔隙和裂纹分析 59
3.3.1 孔隙率分析 59
3.3.2 微裂纹重构分析 62
3.3.3 本节小结 64
3.4 高温氧化环境下 3D C/SiC的显微CT分析 65
3.4.1 干氧环境下 3D C/SiC表面SiC涂层SEM分析 65
3.4.2 干氧环境下 3D C/SiC表面SiC涂层重构分析 66
3.4.3 干氧环境下 3D C/SiC基体重构分析 69
3.4.4 湿氧环境下 3D C/SiC表面SiC涂层SEM分析 70
3.4.5 湿氧环境下 3D C/SiC表面SiC涂层重构分析 71
3.4.6 本节小结 75
参考文献 76
第四章 结论与展望 81
4.1 主要结论 81

4.2 研究展望 82

攻读硕士学位期间发表的论文 83

致 谢 84

Table of Contents

Abstract in Chinese ..... **I**

Abstract in English ..... **II**

Table of Contents in Chinese ..... **IV**

Table of Contents in English ..... **VII**

Chapter 1 Introduction **1**

1.1 Research background **1**

1.1.1 Silicon carbide ceramic matrix composites (CMC-SiCs) 1

1.1.2 Research progress on microstructure of the CMC-SiCs 2

1.2 MicroCT introduction **3**

1.2.1 MicroCT principle 3

1.2.2 MicroCT performance parameter 8

1.2.3 The application field of MicroCT 11

1.3 Research progress of MicroCT in composites **14**

1.3.1 Synchrotron MicroCT 14

1.3.2 Commercial MicroCT 16

1.4 Research basis and objectives **18**

1.4.1 Research basis 18

1.4.2 Research objectives 18

1.5 Research methods **19**

1.6 Research contents **19**

Reference **20**

Chapter 2 Experimental instruments and methods **27**

2.1 Experimental instruments **27**

2.2 Experimental samples **27**

2.3 Experimental methods 29
2.3.1 MicroCT analysis 29
2.3.2 SEM & EDS analysis 30
2.3.3 XRD analysis 30
Reference 31

Chapter 3 Results and discussions 32

3.1 Characterization of C/SiC by MicroCT 32
3.1.1 2D C/SiC 32
3.1.2 3D C/SiC 36
3.1.3 Carbon fiber cloth 39
3.1.4 Mini-C/SiC 41
3.1.5 Conclusion 44
3.2 Characterization of porosity of 3D C/SiC by MicroCT 45
3.2.1 Two-dimensional porosity 45
3.2.3 Three-dimensional porosity 50
3.2.3 Porosity wall 52
3.2.4 Conclusion 57
3.3 Analysis of 2D C/SiC under high temperature creep by MicroCT 59
3.3.1 Porosity analysis 59
3.3.2 Microcracking analysis 62
3.3.3 Conclusion 64
3.4 Analysis of 3D C/SiC under high temperature oxygen by MicroCT 65
3.4.1 Analysis of the SiC coating under dry oxygen by SEM 65
3.4.2 Analysis of the SiC coating under wet oxygen by MicroCT 66
3.4.3 Analysis of the SiC matrix under dry oxygen by MicroCT 69
3.4.4 Analysis of the SiC coating under wet oxygen by SEM 70
3.4.5 Analysis of the SiC coating under wet oxygen by MicroCT 71
3.4.6 Conclusion 75
Reference 76

Chapter 4 Main conclusion and outlook 81

4.1 Main conlusions 81

4.2 Outlooks 82

Publications 83

Acknowledgements 84