SiC fiber-reinforced SiC composites with a BN interphase are proposed for use as leading edge structures of hypersonic vehicles. The durability of these materials under hypersonic flight conditions is therefore of interest. Thermogravimetric analysis was used to characterize the oxidation kinetics of both the constituent fibers and composite coupons at four temperatures: 816, 1149, 1343, and 1538°C (1500, 2100, 2450, and 2800°F) and in oxygen partial pressures between 5% and 0.1% (balance argon) at 1 atm total pressure. One edge of the coupons was ground off so the effects of oxygen ingress into the composite could be monitored by post-test SEM and EDS. Additional characterization of the oxidation products was conducted by XPS and TOF-SIMS. Under most conditions, the BN oxidized rapidly, leading to the formation of borosilicate glass. Rapid initial oxidation followed by volatilization of boria lead to protective oxide formation and further oxidation was slow. At 1538°C in 5% oxygen, both the fibers and coupons exhibited borosilicate glass formation and bubbling. At 1538°C in 0.1% oxygen, active oxidation of both the fibers and the composites was observed leading to rapid SiC degradation. BN oxidation at 1538°C in 0.1% oxygen was not significant.
Oxidation of SiC/BN/SiC Composites in Reduced Oxygen Partial Pressures

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Motivation
Technical challenge for hypersonic vehicles: Develop lightweight, durable, reusable, 3000°F (1650°C) structurally-integrated Thermal Protection Systems (TPS) to carry both thermal and mechanical loads using ceramic matrix composite materials.

Objectives
- Characterize the oxidation resistance of BN-coated SiC fiber-reinforced SiC composites at temperatures and oxygen partial pressures relevant for hypersonic environments
- Develop understanding of oxidation degradation kinetics and mechanisms
- Provide data to Materials Research and Design, Inc. for incorporation in FEM for SiC/SiC degradation
- SEM, EDS, TOF-SIMS, XPS to characterize oxidation products

Materials and Procedure
- Sylicom BN fibers
  - Stoichiometric polycrystalline 5-SiC
  - 3 wt% Ti, 1 at 1% N2, 0.7 wt% BN
  - Heat treated in N2 to form a BN surface layer (BN) 1000 nm
  - Size: 0.5 x 3 x 6 mm
- SiC/BN/SiC composites
  - Sylicom BN fibers
  - CVI SiC-coated fabric
  - CVI SiC matrix
- Thermogravimetric Analysis (TGA)
  - 5% O2 at 0.1% O2, 24h
  - 816, 1149, 1538°C (1500, 2100, 2450, 3000°F)
- 100h maximum time, shorter times to investigate kinetics
- SEM, EDS, TOF-SIMS, XPS to characterize oxidation products

Possible reactions
- Oxide formation
  \[ 2 \text{SiC} + 2\text{O}_2(g) \rightarrow \text{SiO}_2(g) + 2\text{C}(g) \]
- Oxide volatilization
  \[ \text{B}_2\text{O}_3 + 2\text{O}_2(g) \rightarrow 2\text{BO}_2(g) \]
- Active Oxidation
  \[ \text{SiC} + \text{O}_2(g) \rightarrow \text{SiO}_2(g) + \text{CO}(g) \]

Summary and Conclusions
- Minimal oxidation of Sylicom IBN fibers or SiC/BN/SiC composites occurs at 816°C
- Transient borosilicate glass formation occurs at 1149 and 1343°C followed by boro volatility, leaving a protective silica scale on both fibers and composites
- Destructive oxidation of fibers and composites occurs at 1538°C
  - 5% O2, excessive borosilicate glass formation, SiC volatilizing, and glass bubble formation
  - 0.1% O2, active oxidation of SiC to form SiO(g) observed

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Fiber Oxidation
- Oxidation weight change for Sylicom IBN fibers
- Microstructure of oxidized fibers
- TOF-SIMS and XPS analysis of oxide scale

Coupon Oxidation
- Oxidation of SiC/BN/SiC coupons
- Microstructure of Oxidized Coupons
- Summary of SiC/BN/SiC composite oxidation

Oxidation at 1538°C
- Oxidation of Sylicom IBN Fibers, SiC/BN/SiC coupons
- Active oxidation of Sylicom IBN Fibers
- Active oxidation of SiC/BN/SiC coupons

Oxide volatilization
\[ \text{B}_2\text{O}_3 + \frac{1}{2} \text{O}_2(g) \rightarrow 2\text{BO}_2(g) \]
\[ \text{B}_2\text{O}_3 + \text{H}_2\text{O}(g) \rightarrow 2\text{HBO}_2(g) \]