Solid Oxide Fuel Cell Seal Glass-BN Nanotubes Composites

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Solid oxide fuel cell seal glass G18 composites reinforced with ~4 weight percent of BN nanotubes were fabricated via hot pressing. Room temperature strength and fracture toughness of the composite were determined by four-point flexure and single edge V-notch beam methods, respectively. The strength and fracture toughness of the composite were higher by as much as 90% and 35%, respectively, than those of the glass G18. Microscopic examination of the composite fracture surfaces using SEM and TEM showed pullout of the BN nanotubes, similar in feature to fiber-reinforced ceramic matrix composites with weak interfaces. Other mechanical and physical properties of the composite will also be presented.
SOFC Seal Glass - Boron Nitride Nanotubes Composites

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Planar Solid Oxide Fuel Cell (SOFC) Seals

**Seal Requirements**
- Hermetic to separate and contain fuel and oxidant within the cell and to bond cell components together
- Chemically and mechanically compatible with various SOFC oxide and metallic components and electrical insulator

**SOFC Seal Glass**
- Composition (mol %): 35BaO-15CaO-5Al₂O₃-10B₂O₃-35SiO₂ (BCAS)
- Low mechanical reliability due to low strength (50 MPa) and low fracture toughness ($K_{IC}<1 \text{ MPa} \cdot \sqrt{\text{m}}$) -- resulting in limited seal life

S1: PEN to Frame
Ceramic to Metal Seal
S2: Interconnect to Frame
Metal to Metal Seal
S3: Frame to Spacer
Metal to Ceramic Seal
S4: Frame to Endplate
Metal to Metal Seal
Objective

• Improve strength and fracture toughness of SOFC seal glass (BCAS)

Approach

• Reinforce BCAS glass with BN nanotubes (BNNTs)
• Characterize glass-BNNT composite by SEM/TEM
• Measure mechanical properties of composite
• Fractography of composite fracture surfaces
Fabrication of BCAS glass-BNNT Composite

Synthesis of BNNTs

BCAS glass powder

Glass powder + 4 wt. % BNNT

Milling (acetone, 24 h)

Drying

Hot Pressing (10 MPa, 15 min, vacuum, 630 °C)

Billets

Machining Test Specimens
Mechanical Properties Evaluation

Strength

- Test bars: 2 mm x 3 mm x 25 mm
- Test rate: 50 MPa/s
- Test temperature/environment: RT/air
- No. of test specimens: 10
- Test method: ASTM C 1611 (4-point Bend)

Fracture toughness

- Test bars: 2 mm x 3 mm x 25 mm
- Test rate: 0.5 mm/min
- Test temp/environment: RT/air
- No. of test specimens: 3
- Test method: single edge V-notched beam (SEVNB)

- Elastic modulus (impulse excitation, ASTM C1259)
- Vickers hardness (ASTM C1327)
- Density (mass/volume)
FESEM Microstructures of BNNTs

- Diameter: tens to hundreds of nanometers
- Length: tens of micrometers
SEM/EDS of Glass-BNNT Composite

Black regions: BNNT clusters
TEM of Glass-BNNT Composite

Lattice structure and clusters of multiwalled BNNTs in composite

BNNT/glass interface in the composite
• **Significant strength increase (90%)**: from $48 \pm 7$ MPa for glass to $92 \pm 17$ MPa for composite
• **Flaws**: mostly volume (pores) associated
• **Fracture toughness increased (35%)**: from $0.51 \pm 0.037$ MPa$\sqrt{\text{m}}$ for glass to $0.70 \pm 0.09$ MPa$\sqrt{\text{m}}$ for composite
SEM of Fracture Surfaces

Glass-BNNT Composite

- Fracture surfaces showing pullout of BNNTs and resulting troughs
- Bridging may be a major contributing factor for reinforcement
Elastic Modulus, Density, & Hardness

Rule of mixture not-applicable to elastic modulus and density (Density: glass = 3.814; BN = 2.2; Composite = 3.582 g/cm³)
• BCAS glass composites reinforced with 4 wt % BNNTs fabricated (1st time ever)
• Reinforcement of BCAS glass with 4 wt % BNNTs results in:
  – Significant increase (90 %) in flexure strength
  – Moderate increase (35 %) in fracture toughness
  – Lower elastic modulus, density and hardness
• Rule of mixture not-applicable to modulus and density
• BNNT pullout is the toughening mechanism
• Addition of 4 wt% BNNTs will have little effect on viscosity of BCAS glass and its sealing behavior at SOFC operating temperatures
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