Silicon Carbide Tritium Permeation Barrier for Steel Structural Components

Phase II SBIR Program  Principle Investigator:  Matt Wright

Deuterium Permeation Studies:
Sandia National Laboratories – California
Dean Buchenauer, Robert Kolasinski, Rion Causey

Thermostructural Modeling and Testing:
Sandia National Laboratories – New Mexico
Dennis Youchison, Joe Garde, Thomas Holschuh

D/T Plasma Testing:
Idaho National Laboratory
Pattrick Calderoni

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Background
• Chemical vapor deposited (CVD) silicon carbide (SiC) has superior resistance to tritium permeation even after irradiation.
• Prior work has shown Ultramet foam to be forgiving when bonded to substrates with large CTE differences.

Technical Objectives
• Evaluate foams of vanadium, niobium and molybdenum metals and SiC for CTE mitigation between a dense SiC barrier and steel structure.
• Thermostructural modeling of SiC TPB/Ultramet foam/ ferritic steel architecture.
• Evaluate deuterium permeation of chemical vapor deposited (CVD) SiC.
• D testing involved construction of a new higher temperature (>1000°C) permeation testing system and development of improved sealing techniques.
• Fabricate prototype tube similar to that shown with dimensions of 7cm Ø and 35cm long
• Tritium and hermeticity testing of prototype tube.

Program Details
The goal is to measure permeation through SiC samples up to 1000°C with improved sensitivity compared with the existing system.

Furnace features a 12" long, 3" diameter uniform heating zone and uses a PID-based controller, which allows for ramp-to-setpoint operation. This is helpful to avoid thermal shock, but will also allow thermal desorption experiments to be performed in situ.

The clamping force to seal the sample in the tube assembly (light brown) is provided by an outer frame around the furnace (gray), and the entire assembly along with the pumps and vacuum hardware is supported by an 80/20 frame. The flow of deuterium gas is right to left through the furnace and sample, with all pumps located on the left side.

Second-generation system layout, designed to accommodate a 1200°C split tube furnace.
• The analysis volume (where the QMS measurements are made) was minimized for improved sensitivity to the permeating flux.
• Also visible are the calibrated leaks used for absolute sensitivity and mass tuning calibration of the QMS. The use of three pumping systems provides for isolation of the analysis volume section during system setup and bakeout, thus limiting the background of deuterium in this section.
• The system utilizes about a dozen mini-Conflat valves, one 2¾ Conflat valve, and one butterfly valve (to throttle the analysis volume pump).
• Sealing of the inner and outer tubes accomplished by a custom flange assembly using stock vacuum flanges and a specially designed large-diameter UltraTorr fitting. These fittings use a fluorocarbon FKM O-ring good to 200°C and are located outside the furnace on both ends, with air cooling.
• Figure shows an expanded plan view of the custom flange used to support the UltraTorr seals, along with the pumping ports for the sample (at centerline) and outer volume (at bottom).
• Both ends of the tube assembly will be similar, and will provide for mounting of the sample outside the furnace.
• A feedthrough will be incorporated into one end for the sample thermocouple.

Expanded view of custom flanges used at each end of tube assembly. Custom UltraTorr O-ring fittings are used to seal to both inner alumina tubes and outer quartz tube.
• SiC sample successfully sealed at 1000°C
• 3 weeks running at 1000°C for the 1 mm thick sample (at 1/2 atm on the upstream side).
• Temperature increased to 1100°C on same sample and additional week of time at temperature put on sample.
• Permeability was very low and closely correlated with Causey et al.
Computerized X-ray microtomography was completed at Sandia on Ultramet supplied foam samples. The smaller files were translated from stereolithography format to ACIS solid modeling format.

The solid models of the detailed foam microstructure were meshed and analyzed by commercial computational fluid dynamics (CFD) codes (CFDesign and CCM+) to determine the effective permeability and ligamental heat conduction.

Foam models derived from computerized x-ray tomography were closed with facesheets of SiC on one side and steel on the other inside CCM+.

The CCM+ tet mesh was exported to Abaqus for stress analysis.

Deflections, inelastic strains and stresses were computed in the ligaments and permeation barrier facesheets using Abaqus.

X-ray microtomography file of carbon foam after translation to ACIS solid modeling format (left). CFD conjugate heat transfer analysis (right).
Modeling of SiC Foam

Temperature

Vertical Deformed

Mises All

Maximum

Over 500

Over 3000

45ppi, 10%

45ppi, 15%

65ppi, 10%
Modeling of SiC Foam

Temperature

Vertical Deformed

Mises All

Maximum

Over 500

Over 3000

65ppi, 25%

100ppi, 10%

100ppi, 20%

plates
Modeling of Nb Foam

Temperature

Vertical Deformed

Mises All

Maximum

Over 500

Over 700

45 ppi, 10%

45 ppi, 15%

65 ppi, 15%
Modeling of Nb Foam

Temperature

Vertical Deformed

Mises All

Maximum

Over 500

Over 700

65ppi, 25%

100ppi, 10%

100ppi, 20%

plates
Modeling Conclusions

- Nb foam with 45 ppi 10% dense has the lowest thermal stress of all.
- The SiC foam with 65 ppi 10% density has the lowest stress for the SiC foams. The 100 ppi cases were higher.
- Stresses increased with ppi and density.
- In all cases the foam reduces the stress in the faceplates as expected, the Nb foam by an order of magnitude.
- The SiC foam was not as effective because of higher stresses in the ligaments, but stresses in the faceplates were still very low.

Plates (over 3000)  65ppi, 10% SiC foam (over 3000)  45ppi, 10% Nb foam (over 700)
Foam Development

- Process was established for CVD of Vanadium
- Deposition rate was impractically slow, only low density foam was fabricated.
- Cost and schedule would not allow development of V foam in the project.
- Vanadium attempted because it is a preferred material in fusion applications.
- Niobium (with similar physical properties) substituted to demonstrate prototype fabrication and testing.

SEM images of vanadium deposit on molybdenum substrate

Optical microscopy images of vanadium deposits on RVC foam
• Successfully developed processing for brazing of niobium and SiC foam to steel

Optical micrographs of SiC foam bonded to low-activation steel with Cusil ABA

Optical micrographs of Nicoro braze joint between niobium foam and steel

High mag SiC foam-LAS with Cusil ABA

Optical microscopy images of vanadium deposits on RVC foam
• Dense CVD SiC coatings can be deposited directly on foam or separately and post brazed.
• Depositing SiC TPB on niobium foam not possible because brittle silicides form and warpage.
• SiC TPB and Nb foam must be made separately and then joined by brazing.
• Currently the main programmatic focus is confirming Mo coating on SiC\textsuperscript{1} will allow brazing with Nicoro.
• Based on modeling results, 45 ppi/10\% and 100 ppi/10\% Nb foam are best candidates. 4 additional foam candidates will be fabricated into ~1\times1” strain-gauge instrumented coupons to be tested using the EB-60 e-beam at the PMTF.
• The dense SiC TPB layer will be heated to 800\degree C while the steel backside will be passively cooled to 550\degree C.
• Results of coupon testing will determine foam materials used for prototype tube fabrication and hermeticity evaluation after thermal cycling.
• D/T plasma testing at INL STAR anticipated for Q1 2011.

\textsuperscript{1}B.V. Cockeram, "The Diffusion Bonding of Silicon Carbide and Boron Carbide Using Refractory Metals", USDOE Contract No. DE-AC1 1-98 PN38206