

### Radiation-Induced Attenuation and Nonlinear Optical Properties of Fused Silica and Single-Crystal Sapphire

Chris Petrie Milos Burger 4/17/2024

Contributions from T. Birri, T. Dixon, and K. Everett

ORNL is managed by UT-Battelle LLC for the US Department of Energy





### Radiation-Induced Attenuation and Nonlinear Optical Properties Post-Irradiation Annealing in Fused Silica and Single-Crystal Sapphire

#### **Chris Petrie**

**Milos Burger** 4/17/2024

\* Samples were determined to be too thin for nonlinear measurements after project award

Contributions from T. Birri, T. Dixon, and K. Everett

ORNL is managed by UT-Battelle LLC for the US Department of Energy





Nuclear applications for fiber optics (not a comprehensive list)

### Embedded sensors for pressure, corrosion, or acoustic emissions [4, 5]

H.C. Hyer et al., *Additive Manufacturing*, **52** (2022), 102681.
C.M. Petrie et al., *Journal of Nuclear Materials* **552** (2021) 153012.
A. Birri and T.E. Blue, *Progress in Nuclear Energy* **130** (2020) 103552.
D.C. Sweeney, A.M. Schrell, and C.M. Petrie, *IEEE Trans. Instrum. Meas.* **70** (2021) 1-10.



Trace O and H detection in SFRs, other impurities in MSRs

Local temperature measurements in an experiment simulating gascooled reactor core outlet mixing [6]





[5] C.M. Petrie, D.C. Sweeney, and Y. Liu, US Non-Provisional Patent No. US 2021/0033479 A1, Application No. 16/865,475, published February 4, 2021.

[6] H.C. Hyer et al., "Toward Local Core Outlet Temperature Monitoring in Gas -Cooled Nuclear Reactors Using Distributed Fiber-Optic Temperature Sensors," *Applied Thermal Engineering* (under review).

### Limitations for a-SiO<sub>2</sub> and $\alpha$ -Al<sub>2</sub>O<sub>3</sub> fiber-based sensors

- Both sensors suffer from signal attenuation and drift under neutron irradiation
  - Need to quantify at higher dose and temperature





#### Low-temperature (<100°C) attenuation in $a-SiO_2$ [1]

CAK RIDGE

[1] G. Cheymol et al., "High Level Gamma and Neutron Irradiation of Silica Optical Fibers in CEA OSIRIS Nuclear Reactor," IEEE Trans. Nucl. Sci. 55 (2008) 2252-2258 [2] W. Primak, "Fast-Neutron-Induced Changes in Quartz and Vitreous Silica," Phys. Rev. 110 (1958) 1240-1254.

### Inexpensive, passive HFIR irradiation tests

- "Slab" specimens irradiated in the High Flux Isotope Reactor (HFIR)
  - 4 low-OH a-SiO\_2, 4 high-OH SiO\_2, and 4  $\alpha\text{-Al}_2\text{O}_3$  per capsule
  - 16 mm long × 5 mm wide × 0.85 mm thick
  - Design temperatures of 100, 300, and 600°C confirmed by passive SiC temperature monitors (TMs)

#### Irradiation test matrix

Fast neutron fluence (n/cm²)	Measured specimen temperatures (°C)		
	Target 100°C	Target 300°C	Target 600°C
2.4×10 <sup>21</sup>	95	298	688
9.6×10 <sup>21</sup>	88	N/A	592



Capsule parts before (top) and after (bottom) assembly



## Post-irradiation examination

- Optical transmission measured through specimen thickness
- Dimensional changes measured using micrometer
- Dilatometry to measure recovery of radiationinduced dimensional changes



#### Post-irradiation specimen pictures





Vis = visible; NIR = near-infrared region; UV = ultraviolet

#### Schematic (a) and picture (b) of optical measurement system

#### **Optical measurements**

CAK RIDGE

Reprinted from "High-dose temperature-dependent neutron irradiation effects on the optical transmission and dimensional stability of amorphous fused silica," *Journal of Non-Crystalline Solids* Vol 525 (2019), C.M. Petrie, A. Birri, and T.E. Blue, with permission from Elsevier en slide master to edit

### a-SiO<sub>2</sub> radiationinduced dimensional change

#### New data compared to previously developed model

Good agreement • at 592°C

• Further emphasizes missing physics at <100°C (swelling after peak compaction)

**CAK RIDGE** National Laboratory



### Post-irradiation annealing of a-SiO<sub>2</sub> radiation-induced compaction

- Initial recovery of swelling caused by low-temperature gamma irradiation after reactor shutdown?
- Significant recovery of radiation-induced compaction, particularly for samples irradiated at <300°C and heated to >500°C
- Start of heating, relative to preirradiation length
- X End of heating , relative to preirradiation length



More significant in high-OH samples

### $\alpha$ -Al<sub>2</sub>O<sub>3</sub> radiation-induced dimensional changes

- Unfortunately we couldn't accurately measure caxis changes
  - Sapphire fibers are grown along c-axis
- Low temperature a-axis data consistent with literature
- High temperature a-axis data from this and previous RTE question validity of some literature data
  - Our data suggest anisotropic swelling is less than previously reported





### Post-irradiation annealing of $\alpha$ -Al<sub>2</sub>O<sub>3</sub> radiation-induced swelling

- Minimal recovery of radiation-induced swelling
  - Even in samples irradiated at <100°C and then heated to >800°C
  - Good in the sense that once the sensor drifts it wouldn't change much during temperature transients





### a-SiO<sub>2</sub> transmission

- Increases in attenuation at higher dose are minor, particularly in near-infrared (>1,000 nm) range
  - Broadband increases likely due to chemical interactions at higher temperatures, higher dose (time)
  - Origins of increases >1,600 nm are unclear (compaction effects should be lower at higher temperatures)



### $\alpha$ -Al<sub>2</sub>O<sub>3</sub> transmission

- Higher dose did not significantly affect attenuation after irradiation at ~90°C
- Prohibitive attenuation after 3.2 dpa at 688°C also observed after 12 dpa at 592°C
  - Advanced Sensors and Instrumentation (ASI) program supporting transmission electron microscopy: Could Rayleigh scattering from radiationinduced voids could provide an explanation?
  - Unclear what would cause the attenuation at > 1600 nm





### **Summary and conclusions**

- Signal attenuation and drift are critical issues for both fused silica and single crystal sapphire optical fiber based sensors
  - Data is needed at higher temperatures and neutron fluence
- Examined bulk a-SiO\_2 and a-Al\_2O\_3 samples irradiated in HFIR to 9.6×10<sup>21</sup> n/cm<sup>2</sup> at ~100, and 600°C
- a-SiO<sub>2</sub> attenuation appears manageable but compaction-induced drift is significant and current models are not accurate at low temperatures and high dose
- $\alpha$ -Al<sub>2</sub>O<sub>3</sub> attenuation is much higher than a-SiO<sub>2</sub>, particularly at higher temperatures (~600–700°C)
  - ASI activity trying to identify potential mechanism



Post-irradiation pictures of a-SiO<sub>2</sub> and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> specimens



# Questions? Chris Petrie, petriecm@ornl.gov

#### Chris Petrie

Scholar Group Leader, ORNL petriecm@ornl.gov W (865) 576-0827 | C (419) 410-4135 ORCID: 0000-0003-1167-3545 https://www.ornl.gov/staff-profile/christian-m-petrie





