

## Radiation effects on candidate fiber optic sensors for nuclear applications

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#### Nuclear applications for fiber optics (not a comprehensive list)



[1] H.C. Hyer et al., Additive Manufacturing, 52 (2022), 102681. [2] C.M. Petrie et al., Journal of Nuclear Materials 552 (2021) 153012. [3] D.C. Sweeney et al., "Analog Front End Digitizer using Optical Pulse-Width Modulation for Nuclear Applications," IEEE Trans. Instrum. Meas. (under review) [4] A. Birri and T.E. Blue, Progress in Nuclear Energy 130 (2020) 103552.

[5] D.C. Sweeney, A.M. Schrell, and C.M. Petrie, IEEE Trans. Instrum. Meas. 70 (2021) 1-10. [6] 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.

Heater

Centering rods

Sheath

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

#### Fiber optic sensor materials

#### Fused silica glass (a-SiO<sub>2</sub>)

- Maximum temperature: 1000°C (long-term devitrification)
- Cost: As low as ~\$0.20 per meter
- Core diameter: ~8–10 µm (singlemode)
- Cladding: Routinely accomplished via chemical dopants in a-SiO<sub>2</sub>
- Maximum continuous length: >> kilometers
- Typical intrinsic attenuation: ~dB/km

#### Singe-crystal sapphire ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>)

- Maximum temperature : 1700–1800°C demonstrated, close to ~2000°C melting point
- Cost: ~\$1k per meter
- Diameter: 75–500 µm
- Cladding: None
  - Active R&D targeting singlemode operation
- Maximum continuous length: ~meters
- Typical intrinsic attenuation: ~dB/m
  - Dominated by scattering losses due to lack of cladding
- Must be single-crystalline to avoid scattering losses at grain boundaries
- High loss and short lengths generally require non-trivial splicing to a-SiO<sub>2</sub> leads





### a-SiO<sub>2</sub>: High temperature degradation

- Polymer-based coatings burn at ~200–300°C (requires metal coatings or sheaths for protection)
- Increased OH absorption near 1400 nm and > 1800 nm in presence of  $H_2$  or  $H_2O$  at > ~600°C (manageable)
- Increased O mobility at > 600°C
  - No significant effect on transmission
  - Can alter local defect distributions and challenge distributed temperature sensors relying on Rayleigh backscatter
  - Can be addressed by altering fiber or signal processing
- Dopant diffusion at > 1000°C
- Devitrification, or crystallization, at temperatures of ~900°C and above (rate depends on temperature)
  - 850°C: Indefinite?
  - 900°C: Days to weeks?
  - 1000°C: Days
  - 1100°C: <1 day</p>

[1] A.H. Rose, "Devitrification in Annealed Optical Fiber," Journal of Lightwave Technology 15(1997) 808–814.



#### $\alpha$ -Al<sub>2</sub>O<sub>3</sub>: High-temperature degradation

- Large increases in attenuation in air at ~1400°C
  - Formation of surface bubbles, likely aluminum hydroxide  $AI(OH)_3$
- Attenuation and bubbles not observed in an inert environment
  - Promising for nuclear applications that can locate sapphire sensors in a metal sheath or fuel cladding backfilled with an inert gas
  - Successful operation up to 1500°C



C.M. Petrie and T.E. Blue, "In-situ Thermally Induced Attenuation in Sapphire Optical Fibers Heated to 1400°C," Journal of the American Ceramic Society 98 (2014) 483-489.
B.A. Wilson et al., "High Temperature Effects on the Light Transmission through Sapphire Optical Fiber," Journal of the American Ceramic Society 101 (2018) 3452-3459.



#### Single crystal sapphire $(\alpha - AI_2O_3)$ vs. amorphous silicon dioxide $(a - SiO_2)$

- Sapphire: Ordered single crystalline structure (corundum)
  - Hexagonal close packing of O (anion sublattice), Al occupies 2/3 of octahedral interstices
  - No grain boundaries to serve as sinks for point defects
  - Point defects -> trapping states in band gap -> increased optical absorption at energies corresponding to band transitions
  - Aggregation of point defects can result in microstructural and dimensional changes that cause drift of Bragg gratings or other sensors that rely on changes in refractive index or thermal expansion
- Silica: Amorphous structure (distribution of Si-O-Si bond angles, no long-range order)
  - Less sensitive to point defects
  - Expected to be more tolerant to radiation damage

#### Ordered crystal structure of $\alpha$ -Al<sub>2</sub>O<sub>3</sub>

#### Amorphous structure of a-SiO<sub>2</sub>





## Radiation-induced dimensional changes

## Sapphire swells >4% under neutron irradiation [1]

- No evidence of saturation up to ~2.3×10<sup>22</sup> n/cm<sup>2</sup>
- Swelling higher at higher temperatures



## Fused silica compacts ~2% under neutron irradiation [2]

- Saturates after ~10<sup>20</sup> n/cm<sup>2</sup>
- Equilibrium compaction lower at higher temperatures

1E+22

[1] C.M. Petrie et al., "Optical transmission and dimensional stability of single-crystal sapphire after high-dose neutron irradiation at various temperatures up to 688°C," Journal of Nuclear Materials 559 (2022) 153432.

[2] C.M. Petrie et al., "High-Dose Temperature-Dependent Neutron Irradiation Effects on the Optical Transmission and Dimensional Stability of Amorphous Fused Silica," Journal of Non-Crystalline Solids 525 (2019) 119668.

#### High neutron fluence ( $\sim 10^{22} n_{fast}/cm^2 = 12-20 dpa$ ) measurements of bulk optical properties



$$5688^{\circ}C: a-SiO_{2}$$
  
95°C: a-SiO<sub>2</sub>  $505^{\circ}C: α-Al_{2}O_{3}$   
95°C: α-SiO<sub>2</sub>  $505^{\circ}C: α-Al_{2}O_{3}$ 



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[2] C.M. Petrie et al., "High-Dose Temperature-Dependent Neutron Irradiation Effects on the Optical Transmission and Dimensional Stability of Amorphous Fused Silica," Journal of Non-Crystalline Solids 525 (2019) 119668.

#### Comparison to previous low neutron fluence in situ measurements

- High neutron fluence testing [1]
  - Post-irradiation attenuation (or optical density, OD) measurement
  - OD at 650 nm normalized to value measured after irradiation at 95°C
  - 1.1×10<sup>15</sup> n/cm<sup>2</sup>/s fast flux
  - 2.4×10<sup>21</sup> n/cm<sup>2</sup> fast fluence
- Previous low neutron fluence testing [2]
  - In situ measurement
  - Attenuation rates (Å) at 650 nm normalized to value during irradiation at 56°C
  - 6.3×10<sup>10</sup> n/cm<sup>2</sup>/s fast flux
  - 6.9×10<sup>15</sup> n/cm<sup>2</sup> fast fluence
- Clearly very different temperature trends, suggesting different phenomena at low vs. high neutron fluence



[1] C.M. Petrie et al., "Optical transmission and dimensional stability of single-crystal sapphire after high-dose neutron irradiation at various temperatures up to 688°C," Journal of Nuclear Materials 559 (2022) 153432.

[2] C.M. Petrie and T.E. Blue, "In-situ reactor radiation-induced attenuation in sapphire optical fibers heated up to 1000°C," Nuclear Instruments and Methods in Physics Research B: Beam Interactions with Materials and Atoms 342 (2015) 91-97.

#### Current theory: Rayleigh scattering losses from radiationinduced voids that occur at high dose and temperature

298°C: Dislocation loops, no voids

688°C: Voids aligned along caxis TEM courtesy of Keyou Mao (ORNL)



- Observations of voids oriented along c-axis consistent with previous literature
  - Requires temperatures >500°C for fluence tested in HFIR
- Void diameter (~3 nm) <<  $\lambda$  (~1  $\mu$ m), n<sub>void</sub>  $\approx$  1
- Observed  $\lambda^{-4}$  attenuation dependence consistent with theory



[1] C. Kinoshita and S.J. Zinkle, "Potential and limitations of ceramics in terms of structural and electrical integrity in fusion environments," *Journal of Nuclear Materials* 233–237 (1996) 100–110.

## WIRE-21 experiment

- Primary goal: Evaluate effects of high neutron fluence on WEC's wireless sensors in the High Flux Isotope Reactor (HFIR) at LWR temperatures (~300°C)
- Secondary goal: Characterize temperature and flux distributions with additional instrumentation
  - Thermocouples
  - Distributed fiber optic temperature sensors
  - Self-powered neutron detectors (V emitter)
  - Passive SiC TMs and flux wires
- Most highly instrumented experiment in HFIR's history
- High neutron flux:  $\sim 5 \times 10^{14} n_{fast}/cm^2/s$
- ~50 cm length within fueled region of the core



## In situ measurements of a-SiO<sub>2</sub> fiber sensors

- Primarily focused on radiation-hardened fibers
  - Pure SiO<sub>2</sub> core, F-doped SiO<sub>2</sub> cladding
  - Hollow core photonic crystal fibers
  - Ge-doped core fibers as a reference
- Fibers included with and without fiber Bragg gratings (FBGs)
  - Type I and Type II
- Optical backscatter reflectometry measurements made every 5 minutes
  - 1530–1572 nm

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- Allows measurement of spatial variations in reflected amplitude (attenuation) and local spectral shifts
- Data processed using previously-established adaptive reference techniques to continuously resolve shifts relative to start of experiment

Fiber	Description	Gratings
1	Pure $SiO_2$ core, F-doped $SiO_2$ cladding	N/A
2	Ge-doped SiO <sub>2</sub> core, pure SiO <sub>2</sub> cladding	
3	F-doped SiO <sub>2</sub> core and cladding with 6–9 Type II gratings	~1% reflectivity, ~65 mm spacing
4		
5	Pure SiO <sub>2</sub> core, F-doped SiO <sub>2</sub> cladding with 28 Type II gratings	~3% reflectivity, ~10 mm spacing
6	Ge-doped SiO <sub>2</sub> core, pure SiO <sub>2</sub> cladding with ~120 Type I gratings	<0.1% reflectivity, 10.5 mm spacing
7	Hollow core photonic crystal fiber	N/A
8		

## Reflected amplitudes

14

- Ge-doped fiber amplitudes quickly reduced to noise floor .
- F-doped fiber amplitudes reach equilibrium values higher than preirradiation values
- Amplitudes from Type II FBGs in F-doped fiber approach those of • non-grated fiber



-70

-80

-90

-100

-110

F-FBG: 0.0 EFPD

F-FBG: 2.3 EFPD F-FBG: 9.6 EFPD **F-FBG: 12.5 EFPD** 

F-FBG: 25.3 EFPD

## Spectral shifts

- Drift in a-SiO<sub>2</sub> fibers expected due to compaction
  - Predictive models developed previously [1]
  - Negative dL/L, positive dn/n, net blue shift (lower wavelengths)
  - Should reach equilibrium below 10<sup>20</sup> n<sub>fast</sub>/cm<sup>2</sup>
- Model accurately captures drift up to ~3×10<sup>19</sup> n<sub>fast</sub>/cm<sup>2</sup>
- Unsaturated drift begins at higher fluences
  - Cannot be explained by previous measurements of compaction and refractive index at even higher neutron fluence



[1] C.M. Petrie et al., "High-Dose Temperature-Dependent Neutron Irradiation Effects on the Optical Transmission and Dimensional Stability of Amorphous Fused Silica," Journal of Non-Crystalline Solids 525 (2019) 119668.

[2] C.M. Petrie and D.C. Sweeney, "Enhanced backscatter and unsaturated blue wavelength shifts in F-doped fused silica optical fibers exposed to extreme neutron radiation damage," Optica (under review).

# Similar linear drift observed previously in FBGs in random air-line fibers

- Consistent trends observed in fibers irradiated with/without FBGs in both F-doped SiO<sub>2</sub> fibers and SiO<sub>2</sub> fibers with random air-lines
- Fluence at which unsaturated drift begins could depend on temperature, dose rate, fiber type, FBG type, etc.



[1] M.A.S. Zaghloul et al., "Radiation resistant fiber Bragg grating in random air-line fibers for sensing applications in nuclear reactor cores," Optics Express 26 (2018) 11775.

### Windows of operation





17

## Ouestions? Chris Petrie, petriecm@ornl.gov

