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Linear and Non-linear Optical Characterization of Glass and Sapphire for Optical Instrumentation of Advanced Fission Reactors

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INTRODUCTION

The linear and nonlinear optical properties of glass and sapphire are investigated to determine the material’s suitability to support optical instrumentation for advanced fission reactors. A post-irradiation examination (PIE) experiment consisting of a Z-scan, measuring nonlinear optical absorption and refraction, and a spectrometer, measuring linear optical absorption was developed and used [1, 2]. Sample irradiations and measurements in a mixed gamma and neutron flux were performed at The Ohio State University Nuclear Reactor Laboratory (NRL). The NRL facilities allow for the near-in-situ evaluation of materials under both post-irradiation and concurrent-irradiation thermal annealing conditions. Radiation-induced negative nonlinear optical absorption (NLA) was observed for the first time in fused silica, sapphire, and borosilicate glass under both types of thermal annealing conditions [3]. The NLA was observed in conjunction with linear optical absorption, and the effects of thermal annealing are reported.

Experiment Description and Testing Plan

The nonlinear and linear optical absorption of bulk optical materials were measured using the PIE system developed and validated at the University of Michigan, consisting of a Z-scan and spectroscopy experiment [4]. The system is self-contained and mobile so that the experiment can be established at the point of irradiation to reduce time delays between sample irradiation, thermal annealing, and examination to provide near in-situ performance data of the optical materials. The PIE was established in a dedicated optics and radiation laboratory at the NRL where irradiation and thermal annealing of samples was performed.

The samples examined included high-OH content fused silica (Spectrosil 2000), low-OH content fused silica (Infrasil 302), optical grade sapphire, and borosilicate glass including a cerium doped radiation-resistant borosilicate glass (NBK7 and BK7G18). The samples were irradiated within the NRL nuclear reactor while suspended within the custom-fabricated thermal annealing furnaces to ensure uniform radiation exposure between post- and concurrent-irradiation thermal annealing cases. The three radiation doses administered to the sample sets are provided in Table I, with the neutron dose representing the total fluence (fast and thermal). Examples of the optical samples irradiated to n-Dose 1 are provided in Fig 1.

TABLE I. Neutron and gamma doses administered to optical samples for evaluation.

<table>
<thead>
<tr>
<th>Dose</th>
<th>Total Neutron Fluence (γ Dose)</th>
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<tr>
<td>n-Dose 1</td>
<td>$3.4 \times 10^{16} \text{ n \cdot cm}^{-2}$ (42 Mrad γ)</td>
</tr>
<tr>
<td>n-Dose 2</td>
<td>$1.7 \times 10^{17} \text{ n \cdot cm}^{-2}$ (212 Mrad γ)</td>
</tr>
<tr>
<td>n-Dose 3</td>
<td>$3.6 \times 10^{17} \text{ n \cdot cm}^{-2}$ (443 Mrad γ)</td>
</tr>
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Fig. 1. Neutron and gamma irradiated optical samples. (a) From left to right: Spectrosil 2000, Infrasil 302, and sapphire. (b) Left: BK7G18 and right: NBK7.

Two separate sets of window samples were either thermally annealed post-irradiation, or annealed concurrently with irradiation, for each radiation dose. This was done to compare the rate of annealing to the rate of radiation damage in each sample type. Samples annealed post-irradiation were heated to 200 °C, 400 °C, 600 °C, and 800 °C, for 30 minutes at each temperature, and evaluated with the PIE system after
irradiation and after each annealing temperature. Samples annealed concurrently with irradiation were heated to 800 °C for the duration of the irradiation and evaluated with the PIE system after irradiation. The borosilicate glass samples were only heated to 400 °C and were not included in the concurrent-irradiation thermal annealing experiment, because of the melting point of borosilicate glass.

RESULTS

Figures 2, 3, and 4 show the NLA measurements for Spectrosil 2000, Infrasil 302, and sapphire, respectively, for the cases of post- and concurrent-irradiation thermal annealing. Figures 5 and 6 show the NLA measurements for NBK7 and BK7G18, respectively, for the case of

Fig. 2. NLA measurements of Spectrosil 2000 after (a) irradiation to n-Dose 1 and post-irradiation thermal annealing through 400 °C, (b) irradiation to n-Dose 2 and post-irradiation thermal annealing through 400 °C, (c) irradiation to n-Dose 3, (d) irradiation to n-Dose 1 with concurrent-irradiation thermal annealing at 800 °C, (e) irradiation to n-Dose 2 with concurrent-irradiation thermal annealing at 800 °C, and (f) irradiation to n-Dose 3 with concurrent-irradiation thermal annealing at 800 °C.

Fig. 3. NLA measurements of Infrasil 302 after (a) irradiation to n-Dose 1 and post-irradiation thermal annealing through 400 °C, (b) irradiation to n-Dose 2 and post-irradiation thermal annealing through 600 °C, (c) irradiation to n-Dose 3, (d) irradiation to n-Dose 1 with concurrent-irradiation thermal annealing at 800 °C, (e) irradiation to n-Dose 2 with concurrent-irradiation thermal annealing at 800 °C, and (f) irradiation to n-Dose 3 with concurrent-irradiation thermal annealing at 800 °C.
Fig. 4. NLA measurements of sapphire after (a) irradiation to n-Dose 1 and post-irradiation thermal annealing through 400 °C, (b) irradiation to n-Dose 2 and post-irradiation thermal annealing through 400 °C, (c) irradiation to n-Dose 3, (d) irradiation to n-Dose 1 with concurrent-irradiation thermal annealing at 800 °C, (e) irradiation to n-Dose 2 with concurrent-irradiation thermal annealing at 800 °C, and (f) irradiation to n-Dose 3 with concurrent-irradiation thermal annealing at 800 °C.

Fig. 5. NLA measurements of NBK7 after (a) irradiation to n-Dose 1 and post-irradiation thermal annealing through 400 °C, (b) irradiation to n-Dose 2 and post-irradiation thermal annealing through 400 °C, and (c) irradiation to n-Dose 3.

Fig. 6. NLA measurements of BK7G18 after (a) irradiation to n-Dose 1 and post-irradiation thermal annealing through 400 °C, (b) irradiation to n-Dose 2 and post-irradiation thermal annealing through 400 °C, and (c) irradiation to n-Dose 3.
post-irradiation thermal annealing. Figures 2, 3, and 4 show an increasing negative NLA effect with increasing dose in the upper plots (a, b, and c), especially for the case of sapphire where the NLA does not readily decay after irradiation. Spectrosil 2000 and sapphire are shown to anneal at 400 °C, and Infrasil 302 is shown to anneal at 600 °C to pre-irradiation transmission during post-irradiation thermal annealing. The lower three plots (d, e, and f) in Figures 2, 3, and 4 show that Spectrosil 2000 and sapphire do not develop negative NLA under concurrent-irradiation thermal annealing at 800 °C, but Infrasil 302 does develop negative NLA under concurrent-irradiation thermal annealing. Figures 5 and 6 show that both borosilicate glass types do develop negative NLA of increasing magnitude with increasing dose and that the samples anneal to the pre-irradiation transmission after post-irradiation annealing at 400 °C.

The observed negative nonlinear absorption for all five materials may be attributed to saturable absorption (SA), which competes with two-photon absorption and limits optical absorption in the material as laser intensity increases [3]. The negative nonlinear absorption is observed to counteract radiation-induced linear optical absorption, which is reported in our previous work [5]. The SA in Spectrosil 2000 and Infrasil 302 is attributed to the presence of aluminum, which when exposed to gamma irradiation produces Al-oxygen-hole centers that absorb at 537 nm (FWHM 152 nm). Spectrosil 2000 is more resistant to radiation damage and readily thermally anneals likely because of the high-OH content which passivates E’ absorption centers. SA observed in sapphire is attributable to composite V center defects that thermally anneal via thermal charge recombination. Finally, the SA observed in the borosilicate glass samples is also attributed to aluminum impurities, and the resistance to radiation damage in BK7G18 is attributed to the cerium doping. The negative NLA coefficients are estimated through simulation of the Z-scan curves, and these values are found in our previous work [3].

REFERENCES