Pores in highly densified silica glass by positron annihilation spectroscopy

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Pores are greatly related to the properties of silica glasses. We have previously examined voids in silica glasses with different fictive temperatures using positron annihilation lifetime spectroscopy (PALS) [1]. The pick-off annihilation lifetime of o-Ps increased with the fictive temperature, $T_f$, although the density increased. Further, it was found that this result was very similar to that of the Rayleigh scattering coefficient. High $T_f$ leads to a low degree of network polymerization, resulting in a large density fluctuation. Therefore, the largest pores appeared in the silica glass at high $T_f$ and o-Ps was able to detect such large pores. In this work, highly densified silica glass specimens were produced using the hot isostatic pressure (HIP) method. PALS and Rayleigh scattering measurements were then carried out on those samples.

Highly densified silica glass specimens with $\phi$ 50 mm and 70 mm length were manufactured using the HIP method. The temperature was 2073 K and the atmosphere was Ar gas. The pressure was varied from 0.1 MPa to 200 MPa with a preparation time of up to 4 h. For the sample prepared at 200 MPa, the refractive index and the density saturated within 1 h, while the o-Ps lifetime and Rayleigh scattering coefficient gradually decreased within the preparation time of up to 4 h. This meant that the macroscopic properties did not change for more than 1 h, but the relaxation of the microscopic pore structures continued for a longer time. The plot of Rayleigh scattering intensity versus pore radius estimated from the o-Ps lifetime is summarized in the figure below. This figure shows a good correlation between the two. It was found that the highly densified silica glass showed a small density fluctuation and the pores detected by o-Ps became smaller. We, thus, conclude that PALS is a suitable method for estimating the optical loss in silica glasses such as a glass fiber.

![Rayleigh scattering intensity versus pore radius estimated from the o-Ps lifetime.](image)