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Spectral properties of an UV fused silica within 0.8 to 5 μ m at elevated temperatures

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A radiative inverse method was proposed to derive spectral properties.

A FTIR system to measure high-temperature transmittance has been developed.

The wavelength and temperature dependence of spectral properties were acquired.

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ARSTRACT

A thermal radiative inverse method was used to determine the high-temperature spectral properties of an ultraviolet fused silica from transmittance data for wavelengths from 0.8 to 5 μ m. A developed FTIR system used to measure apparent transmittances of the fused silica sample has been designed and built. In order to reduce the system error caused by detector emission and stray radiation, a measurement strategy at high temperatures was proposed. For deriving spectral transport properties from experimental transmittances, the parameter identification principle was described. The results show that spectral properties are both wavelength dependent and temperature dependent. Spectral refractive indexes rise with increasing temperature and decrease with wavelength. Three absorption peaks of spectral absorptive indices respectively at about $1.4 \mu m$, $2.22 \mu m$ and $2.75 \mu m$ shift toward the far infrared region and vary differently with increasing temperature. In addition, three absorption bands all become broader for temperatures from 20 \degree C to 900 \degree C.

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1. Introduction

Fused silica is one kind of typical semitransparent noncrystalline material. Its spectral properties are very fundamental to estimate the transfer and absorption of optical or thermal radiation $[1-4]$, especially at a higher temperature. Both refractive indexes and absorptive indices are wavelength-dependent and temperature-dependent in the infrared wavelength region. The actual values of spectral properties in some spectrums are always sensitive to the glass manufacturing process and to the presence of impurities, point defects, inclusions, bubbles, etc. [\[5\].](#page--1-0) However, it is often difficult to choose a set of validly spectral data of a fused silica produced by unknown manufacturing processes. Therefore, an update method is always necessary for determining the spectral properties of fused silica for corresponding applications.

The spectral properties of fused silica have been the subject of numerous studies [\[6–9\]](#page--1-0), especially at room temperature [\[5\].](#page--1-0) There are several well-known methods to estimate the spectral transport properties. Usually, the prism method is used to determine the refractive index with great precision in the high transparency region [\[10\]](#page--1-0). The method of Kramers-Kronig analysis on reflectance data with moderate accuracy is applied in the strong absorptive ultraviolet and infrared regions [\[11,12\]](#page--1-0). The interferometry method with an accuracy depending on the determination of the sample thickness and positions of the interference fringes is used in weak absorptive infrared regions [\[13–16\].](#page--1-0) In addition, this method mainly focuses on deriving the refractive index. In comparison, the thermal radiative inversion method [\[17–21\]](#page--1-0) can be applied to a widely spectral range regardless of the absorption strength. This method needs to measure the apparent radiative variables such as emittance, reflectance, transmittance or absorptance [\[22\]](#page--1-0). References [\[23–26\]](#page--1-0) proposed the potential methods for measuring apparent emittances of semitransparent materials.

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References [\[18,27–29\]](#page--1-0) reported methods of transmittance measurement for semitransparent materials at elevated temperatures. More recently, a two-substrate method [\[30,31\]](#page--1-0) was proposed to simultaneous determine emittance, transmittance, and reflectance of semitransparent materials. Although the measurement strategies in references are very different, we can even conclude two main challenges. One unavoidable challenge in all measurements is how to construct a careful correction to obtain the accurate apparent property at an elevated temperature, since the detected radiation includes stray emissions from the heating source, surroundings, or the sample itself. The other unescapable challenge is how to demonstrate the assumptions in a measurement model is reasonable. Therefore, a new measurement strategy or assumption is always in great demand.

This work proposed a radiative inverse method to determine the high-temperature spectral properties of an ultraviolet fused silica within 0.8 to 5 μ m. A developed FTIR system for spectral transmittance measurement from room temperature to elevated temperatures was reported. In order to reduce system error caused by stray radiation and detector emission, a measurement model based on the experimental setup was deduced. The spectral transmittances were measured for several samples of ultraviolet fused silica at temperatures 20 °C, 300 °C, 600 °C and 900 °C. For deriving spectral properties from transmittances, an identifying principle was described. The results show that spectral properties are both wavelength dependent and temperature dependent. Spectral refractive indexes rise with increasing temperature and decrease with wavelength. Three absorption peaks of spectral absorptive indices respectively at about 1.4 μ m, 2.22 μ m and 2.75 μ m shift toward the far infrared region and vary differently with increasing temperature. In addition, three absorption bands all become broader for temperatures from 20 \degree C to 900 \degree C.

2. Experimental principle and uncertainty

2.1. Experimental principle

The major components of the apparatus include a vacuum-Argon furnace and a developed Fourier Transform Infrared Spectrometer (FTIR). Of course, some assistant elements (diaphragms, parabolic mirror, etc.) in the light path are also very necessary. A schematic diagram of the experimental apparatus is shown in Fig. 1.

A vacuum-Argon furnace with a uniform temperature volume of Φ 65 mm \times 90 mm was used to heat a sample. In the execution, the whole chamber of the furnace was evacuated to achieve a vacuum level of \sim 10 Pa, and then Argon was filled into the chamber to keep the absolute pressure in the range from 100 kPa to 110 kPa. These steps minimize spectral contamination from atmospheric carbon dioxide and water vapor, and make an argon environment for the heating elements (tantalum alloy) which are easily oxidized at a high temperature atmosphere. Two W-Re3/25 thermocouples were used in measuring the temperatures of the sample and the uniform temperature volume. The measured results can compare with each other and verify the temperature uniformity of heating volume. Beside, a pyrometer attached on the flank of furnace to check against thermocouple readings.

The Fourier Transform Infrared Spectrometer (Bruker Vertex 70) offers high throughput, high resolution, brief data collection time with broad spectral coverage, and because of computer control, easy data manipulation. We removed its IR detector out to align the furnace into the transmissive light path. The IR detector is mounted on focus of a gold-plated parabolic mirror that collects transmission or source radiation. The strong stray radiation from the furnace usually causes the detector saturated or overflowed. One part directly enters the view of IR detector in combination with the transmissive radiation. The other part backward penetrates into FTIR spectrometer and then become incident radiation illuminating on a sample, after transmission, is finally received by the IR detector. So two diaphragms aligned beside the furnace are used to attenuate the stray radiation through reducing the stray-radiation solid angle. On practice, the stray radiation from possible hot surfaces decrease obviously.

The transmittance of sample is defined as

$$
\tau_{slab}(\lambda) = \frac{I(\lambda)}{I_o(\lambda)}\tag{1}
$$

where, $I_0(\lambda)$ is the radiative intensity of light source; $I(\lambda)$ is the transmissive intensity. However, the output of IR detector is the relevant voltage of irradiance, not the absolute value of energy. The systemic response are usually linear. So the signal of light source can be defined by

$$
S_o(\lambda) = R(\lambda)I_o(\lambda) + N(\lambda). \tag{2}
$$

where, $R(\lambda)$ denotes the systemic response function; $N(\lambda)$ is the signal offset caused by emission of the IR detector. When the sample chamber is heated to the aimed temperature, the signal of light source combined with stray radiation from the high-temperature wall can be expressed as

$$
S'_{o}(\lambda) = R(\lambda)(I_{o}(\lambda) + I_{s}(\lambda)) + N(\lambda)
$$
\n(3)

where, $I_s(\lambda)$ is the intensity of stray radiation. In the same temperature level, the transmissive signal when a sample is mounted in the heating chamber is

$$
S(\lambda) = R(\lambda)(I(\lambda) + I'_{s}(\lambda)) + N(\lambda)
$$
\n(4)

Fig. 1. Schematic diagram of the experimental apparatus.

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