Int J Thermophys (2010) 31:2010–2018
DOI 10.1007/s10765-010-0847-0

Simultaneous Measurement Method of Normal Spectral Emissivity and Optical Constants of Solids at High Temperature in Vacuum

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Published online: 22 October 2010
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Abstract For the case of the thermal design of devices at high temperatures, radiative properties are important. The emissivity of materials depends on their surface conditions and temperatures. The goal of this study is to measure the emissivity of various materials with clear surface conditions. A system for simultaneous measurements of the normal spectral emissivity, optical constants, and thickness of materials at high temperatures has been developed. To determine an accurate surface temperature of specimens, a surface temperature measurement method is applied using the Christiansen effect. This article focuses on evaluations of the Christiansen effect under various conditions. Measurement results of the normal spectral emissivity of ZrO_2 from 773 K to 973 K are also presented.

Keywords Christiansen effect · Ellipsometry · Normal spectral emissivity · Optical constants · Separated blackbody · Simultaneous measurement

1 Introduction

It is essential to know the thermal radiative properties of materials for thermal design in high-temperature environments. For thermal design such as in the Space Shuttle, the emissivity of thermal protection system materials is needed to predict the surface temperature [1]. The emissivity of materials strongly depends on their surface conditions

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and temperatures. Emissivity data are needed for various materials including their surface conditions. One of the problems in emissivity measurements is to determine accurately the surface temperature of the specimens. In the past, thermocouples and thermopiles were primarily used for temperature measurements [2]. However, they are not appropriate for accurate temperature measurements for the following reasons: (1) thermocouples are expected to corrode and their performance degrade at high temperatures, and (2) thermopile measurements are difficult to use for accurate temperature measurements of materials with unknown emissivity.

The goal of this study is to develop a system for simultaneous measurements of the normal spectral emissivity, optical constants, and the thickness of materials at high temperatures. For emissivity measurements, a new surface–temperature measurement method using the Christiansen effect has been proposed [3–6]. The Christiansen effect defines the wavelength (called the Christiansen wavelength) at which the spectral emissivity is unity. This article presents an apparatus for simultaneous measurements of optical properties. In addition, we present the measurement results of the spectral reflectivity of ceramics at room temperature, the temperature dependence of the spectral reflectivity of ZrO_2 from 253 K to 373 K, the incident angle dependence of the spectral reflectivity of ZrO_2 between 10° and 70°, and the normal spectral emissivity of ZrO_2 from 773 K to 973 K. This article discusses the Christiansen effect for three types of ceramics.

2 Measurements

2.1 Measurement Methods

The measurement of the normal spectral emissivity is based on the separated blackbody method. In this method, the normal spectral emissivity ε_N is determined by comparisons of the radiation from an area of a plane surface of the specimen I_S and the radiation from a similar area of blackbody radiation I_B at the same wavelength λ and temperature T. The normal spectral emissivity of the specimen $\varepsilon_N(\lambda, T_S)$ can be expressed as

$$\varepsilon_N(\lambda, T_S) = \frac{I_S(\lambda, T_S) I_B(\lambda, T_{Amb})}{I_B(\lambda, T_B) I_B(\lambda, T_{Amb})}, \tag{1}$$

$$T_S = T_B, \tag{2}$$

where $I_S(\lambda, T_S)$ is the intensity emitted by the specimen radiation at temperature T_S, $I_B(\lambda, T_B)$ is the intensity emitted by blackbody radiation at temperature T_B, and $I_B(\lambda, T_{Amb})$ is the intensity emitted by the surrounding ambient temperature T_{Amb}. In practice, each intensity is detected by a Fourier transform infrared (FTIR) spectrometer.

In this method, one of the most important parameters is the surface temperature of the specimen. This article applies a temperature measurement method using the Christiansen effect. Thus, the radiation energy by the specimen at the Christiansen wavelength q is given by

$$q = \varepsilon_{\lambda \mathrm{Ch}} \sigma T, \tag{3}$$

where the spectral emissivity $\varepsilon_{\lambda \mathrm{Ch}}$ is equal to unity at the Christiansen wavelength, T is the specimen temperature, and σ is the Stefan–Boltzmann constant. Therefore, the surface temperature of the specimen is simply determined by measuring the radiation energy.

2.2 Measurement Apparatus

A schematic diagram and photograph of the measurement system are presented in Fig. 1. This system consists of a vacuum chamber with a cooling shroud, an FTIR spectrometer with optical mirrors (normal spectral emissivity measurement system), a He–Ne laser with a polarizer and photomultiplier tube (ellipsometric measurement system), a thermopile module with the Christiansen effect filter (surface–temperature measurement system), a reference blackbody furnace and specimen, a heater control system, a power supply, and a personal computer. The inner diameter of the vacuum chamber is 255 mm and the length is 512 mm, and the vacuum chamber is maintained at 10^{-4} Pa by using a turbo-molecular pump. A cooling shroud is placed around the reference blackbody furnace and the hemispherical flange.

The hemispherical flange has seven optical windows, which can be used to measure the normal spectral emissivity at 0°, the surface temperature of the specimen with Christiansen effects at 30°, and the optical constants and thicknesses at 55° and 70°. Table 1 shows the system characteristics of the optical windows.

The normal spectral emissivity is measured by FTIR (FTS-60A/596; Bio-Rad) over the wavelength range from 1.6 μm to 22 μm. The radiation from the specimen or the reference blackbody furnace is reflected at the two optical mirrors, and it is corrected at the aperture stop of the FTIR.

The specimen is set in front of the reference blackbody furnace, and its temperature is controlled by radiation from the blackbody. The specimen is held by a ceramic screw, and the holder is vertically movable by the linear motion component in Fig. 2.

The cylindrical blackbody cavity has two parts of the reference blackbody furnace and the heating unit as shown in Fig. 3. The length is 70 mm, and the diameter is 20 mm. The open area ratio of the blackbody is five, and the effective emissivity is greater than 0.99 [7–10]. The shape of the reference blackbody furnace is a cylindrical blackbody cavity, which is made of carbon. The heating unit is made by forming a cylindrical insulating tube and passing a tantalum wire through the insulating tube. The outside of the heating unit is wrapped with insulation materials and titanium foil. The heating unit can be controlled from 373 K to 1673 K.

To determine an accurate surface temperature, a method using the Christiansen effect is presented. The surface–temperature measurement system consists of a thermopile module (A1TPMI; Perkin Elmer, Inc.) with a narrow bandpass filter which has a peak wavelength of 11.96 μm and the half width is 0.97 μm. The thermopile module is set in front of the KRS-5 optical window at an incident angle of 30°.

The ellipsometric system consists of a He–Ne laser at 0.6328 μm (05LHR691, Melles Griot), two polarizers (Gran-Thompson polarizing prisms), a quarter-wave

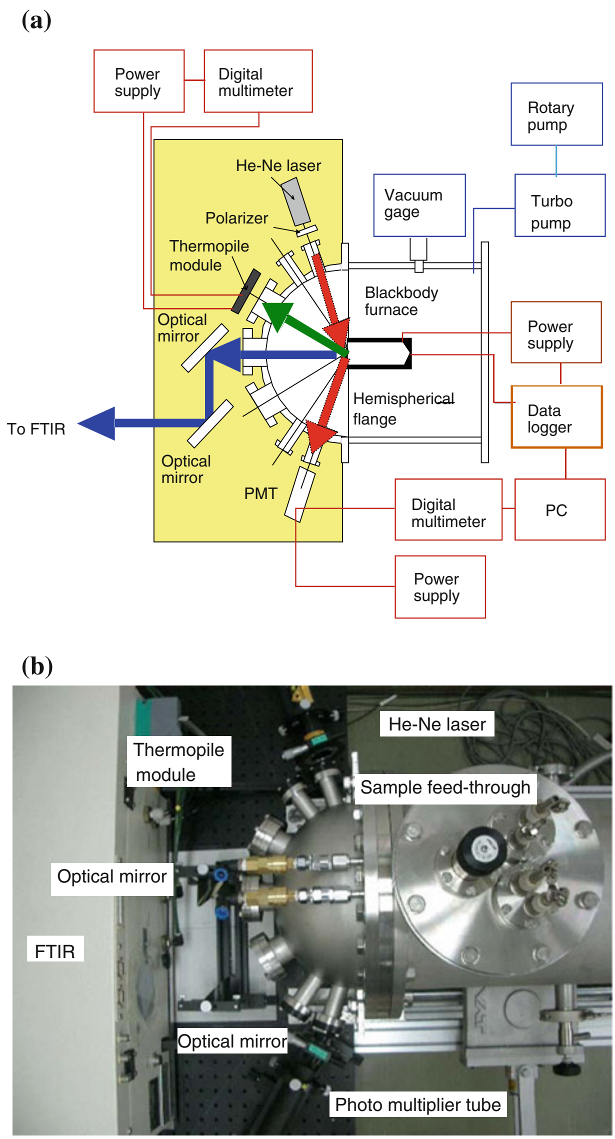

Fig. 1 Simultaneous measurement system for normal spectral emissivity, optical constants, and thickness of materials at high temperature: (a) schematic diagram and (b) photograph

Table 1 System characteristics of the optical windows

Measurement	Incident angle	Measurement wavelength (μm)	Optical windows
Emissivity	0°	1.6–22	KRS-5
Surface temperature	30°	11.96	KRS-5
Optical constants	55°, 70°	0.6328	CaF_2

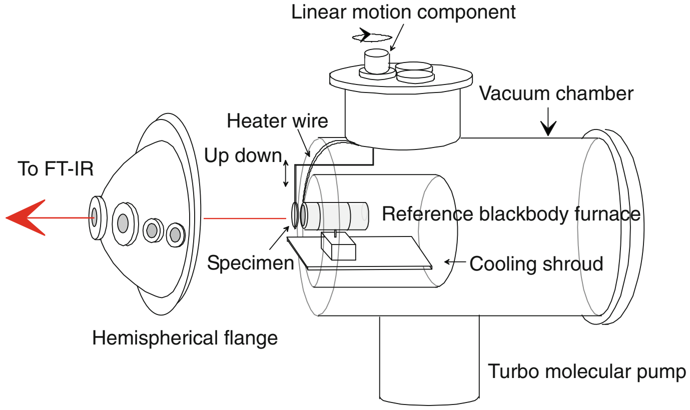

Fig. 2 Installation of specimen and reference blackbody furnace in vacuum chamber

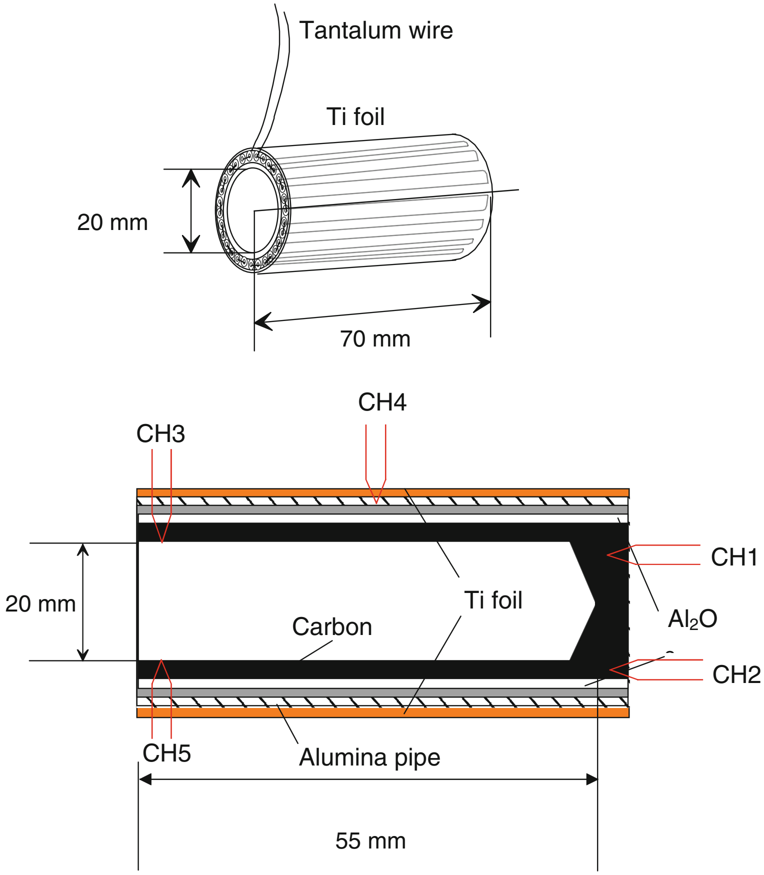

Fig. 3 Configuration of reference blackbody furnace

plate (Mica retardation plate), and a photomultiplier tube (R1104, Hamamatsu Photonics K.K.).

3 Preliminary Measurement Results and Discussion

3.1 Evaluation of Christiansen Effect

The spectral reflectivities of three ceramics (ZrO_2, Al_2O_3, Si_3N_4) at room temperature are shown in Fig. 4. The spectral reflectivity of each ceramic is zero around 10 µm at an incident angle of 20°, and this wavelength is defined as the Christiansen wavelength. The Christiansen wavelengths of ZrO_2, Al_2O_3, and Si_3N_4 are 12.7 µm, 9.5 µm, and 8.6 µm, respectively.

Figure 5 shows the temperature dependence of the spectral reflectivity and spectral transmissivity of ZrO_2 over the wavelength range from 1.6 µm to 22 µm. The results of measurements of the spectral reflectivity in the temperature range from 253 K to 373 K are almost zero, and the spectral transmissivity is zero over the wavelength range from 12 µm to 22 µm. Therefore, the Christiansen wavelength for ZrO_2 is accepted as 12.7 µm, and this effect does not depend on temperature from 253 K to 373 K.

Figure 6 shows the incident angle dependence of the spectral reflectivity of ZrO_2 over the wavelength range from 1.6 µm to 22 µm. From the results, the Christiansen effect is confirmed to be within the incident angle range of 40° Thus, the optical window of the surface-temperature measurement is selected to be at an incident angle of 30° in this system.

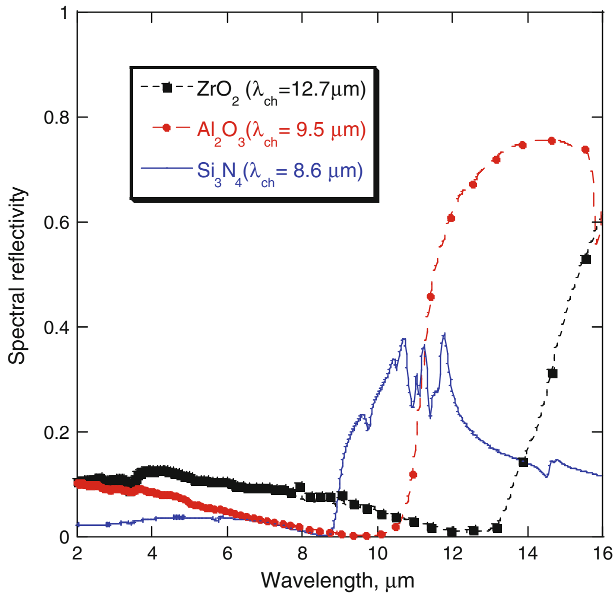

Fig. 4 Spectral reflectivity of three ceramics at room temperature

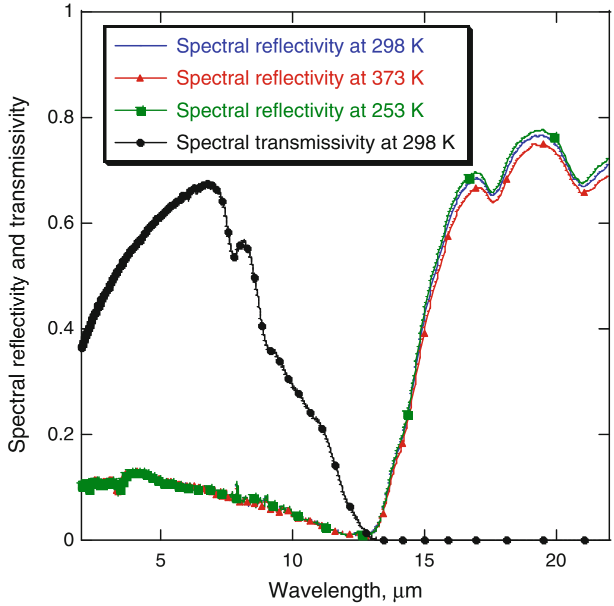

Fig. 5 Temperature dependence of ZrO_2 at incident angle of 20°

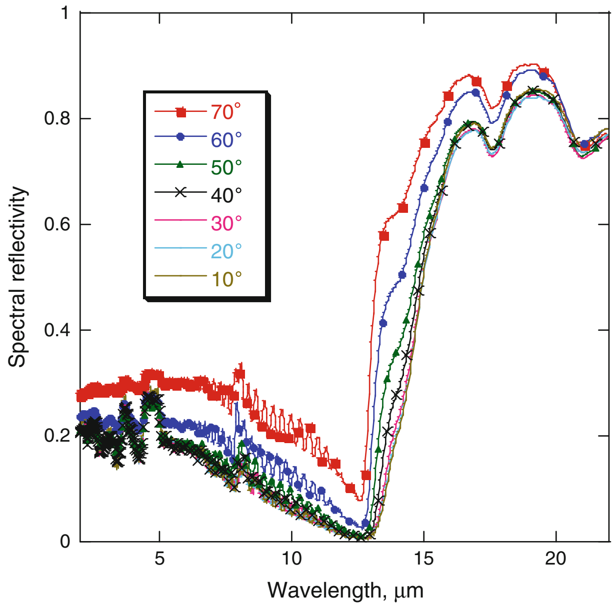

Fig. 6 Incident angle dependence of ZrO_2 at room temperature

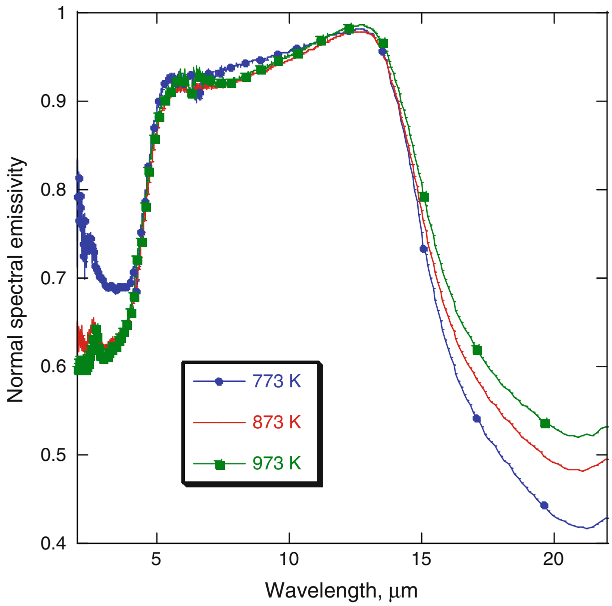

Fig. 7 Normal spectral emissivity of ZrO2

3.2 Normal Spectral Emissivity of ZrO_2

The normal spectral emissivity of ZrO_2 was measured in the wavelength range from 1.6 µm to 22 µm at temperatures of 773 K, 873 K, and 973 K. Figure 7 shows the normal spectral emissivity of ZrO_2. The determination of the surface temperature of ZrO_2 compares the specimen radiation energy with the blackbody radiation energy at the Christiansen wavelength using FTIR. This gives a Christiansen wavelength of 12.7 µm, and this effect appears at high temperatures. In the wavelength range from 12.7 µm to 22 µm, the normal spectral emissivity tends to increase with temperature. On the other hand, in the wavelength from 1.6 µm to 12.7 µm, the normal spectral emissivity tends to decrease with temperature.

4 Conclusions

A system for simultaneous measurements of the normal spectral emissivity and optical constants at high temperature in vacuum was developed. The surface–temperature measurement method with the Christiansen effect was used and evaluated for ceramic specimens. It was clear that the Christiansen effect of the ZrO_2 specimen appears from room temperature to high temperature within an incident angle of 40°. Future research will involve the development of a system for simultaneous measurements of emissivity and optical constants.

Acknowledgment We would like to thank Tanikawa Fund Promotion of Thermal Technology.

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