Spectral emissivity inversion of quartz composite ceramics under hypersonic airflow collision

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Abstract
In this paper, the radiation spectra of quartz composite ceramic thermal protective materials in a pneumatic environment were measured in a plasma arc wind tunnel experiment. Spectral emissivities and material temperature at varying airflow speeds were calculated based on the algorithm of slow variation properties of emissivity. The inversion results show that the spectral emissivity reaches its maximum at a maximum airflow velocity of Mach 10. Emissivity uncertainty caused by the spectral measurement was analyzed. Relative error was determined by comparing real and calculated emissivities from Standard blackbody radiation spectrums at 2298 K in the wavelength range of 420–900 nm and 1573 K for 1200–2400 nm. Results obtained by the algorithm of slow variation properties for emissivity show that the maximum relative error in 420–900 nm is 3.3% and the average relative error is 2.7%; the maximum relative error for 1400–2400 nm is 4.1% and the average relative error is 2.1%. This provides a new method for the study of material emissivity under hypersonic flow collision aerodynamic heating conditions.

1. Introduction

Spacecraft reentry capsules are hypersonic aircraft in near space high-speed flight and have strong collisional friction and compression with the atmosphere. Impacts with atmospheric molecules cause a surface temperature surge and an ionization of the atmospheric molecules impacting the surface and those in the high temperature region that forms a plasma sheath along the leading edge of the aircraft. The plasma sheath will shield the communication signal of aircraft and seriously affect the spacecraft safety. The thermal protection material of aircraft shell needs to withstand high temperature, oxidation, cold and heat shock and so on under extreme environment. As an outstanding structural material, ceramics are widely used. Quartz ceramic composites are used for spacecraft return cabin coatings and thermal insulation tiles. Aerodynamic heating in these extreme environments causes a sharp rise in the material temperature, with radiative heat transfer providing the primary heat dissipation path. The heating dissipation rate is controlled by the materials emissivity. Therefore, accurate measurement of high-temperature emissivity of pneumatic environmental ceramic material is essential for the evaluation performance of thermal protection materials [1–6].

Determining the true temperature of materials through utilizing a multispectral temperature measurement is largely dependent on an accurate emissivity. However, spectral emissivity is reliant not only on wavelength and temperature but also on surface roughness, oxidation, impurities as well as other factors [7–13]. At present, there are usually two main types of measurements, the direct method and the indirect method [10, 14, 15]. In the direct measurement method, the test that is used to measure the same temperature, wavelength and light path under the case of the sample radiation and blackbody radiation ratio is emissivity. Meanwhile, the indirect measurement method is based on Kirchhoff’s law, and the emissivity is calculated through experimentally measuring the reflectance and transmittance of the sample, which is able to avoid blackbody radiation measurement. Notably, the integrating sphere is usually used to obtain the material transmittance or reflectance. However, it should be noted that it is difficult to achieve this under a high temperature heating environment [9].

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Therefore, direct measurement of spectral emissivity has been widely studied. Guo Y M et al have developed a directional spectral emissivity measurement device, including sample temperature control heater, blackbody and Fourier infrared transform spectrometer, etc. Specifically, this can achieve a wavelength of 1.28–28.6 μm and a maximum temperature 1400 K sample emissivity measurement [16]. Additionally, the University of Wisconsin built a spectral emission measurement system for ceramic materials and calibrated the system based on the U.S. DoE quality assurance standards. It was used to measure the spectral emissivity of high temperature reactor materials under high temperature and surface composition changes [17]. Campo developed a device for measuring the emissivity of controlled atmosphere-oriented spectral emission. Under such circumstances, the vacuum chamber can be filled with different gases, which can be used to measure the spectral emissivity of samples with a temperature range of 500–1050 K and a wavelength range of 2.5–22 μm [18]. Notably, Christian et al used the measurement equipment of the German Institute of Technical Physics to calculate the directional spectral emissivity in the temperature range of 80 °C to 400 °C, the wavelength range of 4–40 μm, and the observation angle of 0°–60° relative to the normal [19].

The sample spectral radiation can be obtained in a direct measurement of the spectral emissivity under dynamic heating at a high temperature environment. Despite so however, it is very difficult to achieve the blackbody spectral radiation measurement under the same conditions. Consequently, scholars have engaged in further studies on the measurement method and emissivity algorithm model. Pidan S used pyrometer, dual probe and Pyrex temperature sensor to measure and calculate the emissivity of high-temperature ceramics in further studies on the measurement method and emissivity algorithm model. Pidan S used pyrometer, dual probe and Pyrex temperature sensor to measure and calculate the emissivity of high-temperature ceramics in PWK2 plasma wind tunnel, the temperature range from about 1200 to 1820 K [4]. Wang Hui et al proposed a new technique of emissivity and surface temperature under high-temperature dynamic heating conditions, the spectrometer measured the spectral radiation of the sample. Moreover, the temperature of the sample was calculated according to the two-color temperature measurement method and the multi-spectral fitting method, respectively, the results approaching 2081.3 K [3]. Monteverde et al applied an optical pyrometer to determine the spectral emissivity of ultra-high temperature ceramic ZrB2-SiC under the conditions of pneumatic heating and high enthalpy plasma impact, the material surface temperature reaches 2100 K. However, the optical pyrometer measures a certain error between the brightness temperature and the true target temperature [2].

In summary, the direct measurement method of spectral emissivity is the most popular method for measuring emissivity at high temperatures. However, in a wind tunnel experiment with dynamically heated high-temperature environments, the blackbody spectral radiation cannot be obtained under the same conditions. At present, the measurement methods also experience similar problems of complex equipment and high cost. Therefore, the multispectral method is used to measure the radiation spectrum of quartz ceramic composite materials during wind tunnel ablation. Inversion of temperature and material spectral emissivity is based on the emissivity algorithm of slow variation properties. The experimental setup accurately simulates the reentry aerodynamic heating process. The article has two important innovative meanings: one is that the emissivity calculation algorithm avoids the problem of determining blackbody radiation emission when calculating the material emissivity; another is that the joint calculation of emissivity and temperature improves the accuracy of temperature measurement.

2. Method for calculating temperature and spectral emissivity

The Planck equation used to relate emissivity and temperature is shown in equation (1). For n wavelengths, $\lambda_i$, there are n equations for the spectral radiance $L_i(\lambda_i, T)$ [20, 21].

$$L_i(\lambda_i, T) = \varepsilon_i(\lambda_i, T) \cdot \frac{2hc^2}{\lambda_i^5(e^{hc/kT} - 1)} \quad (i = 1, \ldots, n)$$

In equation (1), Planck’s constant is $h = 6.625 \times 10^{-34}$ J · s, the speed of light is $c = 3 \times 10^8$ m s$^{-1}$, and Boltzmann’s constant is $k = 1.38 \times 10^{-23}$ J/K. The equation has n + 1 unknowns, where n unknowns are emissivities, $\varepsilon_i(\lambda_i, T)$, and one unknown is the temperature value T. This type of system of equations is classified as abnormal state equations and is not solvable. A large number of studies on the emissivity have found that the emissivity spectrum of most materials changes relatively slowly with wavelength [22, 23]. Therefore, we assume that the emissivity at adjacent wavelengths are equal, as shown in equation (2). This assumption reduces equation (1) from n + 1 to n/2 + 1 unknowns, and the resulting system becomes solvable.

$$\begin{align*}
\varepsilon(\lambda_0, T) &= \varepsilon(\lambda_1, T) \\
\varepsilon(\lambda_2, T) &= \varepsilon(\lambda_3, T) \\
\varepsilon(\lambda_{n-1}, T) &= \varepsilon(\lambda_n, T)
\end{align*}$$
Using the above assumptions, we arrive at the following equation:

\[
\frac{L(\lambda_{i-1}, T)}{L(\lambda_i, T)} = \frac{\lambda_{i-1}^5 \cdot e^{\frac{hc}{\lambda_i kT}} - \lambda_{i-1}^5}{\lambda_{i-1}^5 \cdot e^{\frac{hc}{\lambda_{i-1} kT}} - \lambda_{i-1}^5} \quad (i = 1, 2, \cdots, n)
\]  

(3)

The only unknown quantity in equation (3) is the temperature $T$. There are $n/2$ such equations, and they can all be solved for $T$, and the true material temperature is then given by the average. Emissivity is then obtained by substituting the average temperature into equation (4):

\[
\varepsilon_i(\lambda_i, T) = \frac{L_i(\lambda_i, T)}{2hc^2 / \lambda_i^5 (e^{hc/kT} - 1)}
\]  

(4)

This new algorithm, named a similar wavelength method (SWM), is able to solve the abnormal state equations for emissivity.

3. Experimental procedures

3.1. Experimental installation

The pneumatic heating experiment was performed in a plasma arc wind tunnel system, as shown in figure 1. The quartz ceramic sample was affixed onto a support frame such that the surface normal was perpendicular to the airflow velocity. The optical system realized a Casseille Green system, and the spectrometer probe was placed 3 meters from the wind tunnel window. Before the experiment, the spectrometer probe was switched to the laser collimation system to adjust the optical path and probe position to ensure that the detection area completely covers the sample surface. The radiation spectrum signal was directly coupled into the optical system, and the collected optical signal was transmitted to the detection system through an optical fiber. Spectral detection was carried out by a QE65Pro grating spectrometer and an Avantes 2.5–HSC–EVD near–infrared spectrometer. The two spectrometers determine the maximum unsaturated spectral intensity during the integration period of the high–speed scanning mode, and simultaneously record the spectral information.

3.2. Experimental condition

Three aerodynamic states were used during the experiment, and the relevant parameters are shown in table 1. The wind speed was increased from Mach 5 in state 1 to a maximum speed of Mach 10 in state 3.

| Experimental state | State 1 | State 2 | State 3 |
|--------------------|--------|--------|--------|
| Operating time (s) | 0–10   | 10–20  | 20–30  |
| Mach number (Ma)   | 5      | 7      | 10     |

Table 1. Parameters setting in the wind tunnel experimental.
4. Results and discussion

When the hypersonic airflow collides with the ceramic target, the airflow energy is rapidly transformed into internal energy of the ceramic material surface, thereby intensifying the materials molecular motion. The molecules moving at high speed then collide with each other, breaking chemical bonds and ionizing atoms. This created a line radiation spectrum that was superimposed on the continuum spectrum. The continuum spectrum consisted of blackbody radiation, electron bremsstrahlung radiation and composite radiation from the plasma caused by high speed collisions.

The spectral data collected by the spectrometer in this experiment include environmental background information and spectrometer transfer function information. This baseline spectrum was then used to preprocess the spectral data before inverting the temperature and emissivity by SWM algorithm. Spectral data measured by the QE63Pro spectrometer and the Avantes 2.5–HSC–EVD spectrometer are shown in figures 2(a) and (b), respectively. They both show a nonlinearly increasing radiation intensity with Mach number; the ratios of peak radiation intensities are much greater than the ratios in Mach number in the various experimental states. The state 3 spectrum in figure 2(a) clearly shows the presence of a superimposed line radiation spectrum, in contrast to the spectrum in figure 2(b). Because in general, the plasma spectral line in the range of 200–1000 nm is easier to detect [24], and continuous spectrum central wavelength locations vary according to the Wien displacement law such that the center wavelength position moves toward the short wavelength direction as the temperature increases [25]. According to the National Institute of Standards and Technology (NIST) standard atomic spectroscopy database, the line spectrum in figure 2(a) is designated. It was found that the silicon atomic spectrum line Si I 557.7 nm, oxygen ion spectrum O II 509.1 nm and O II 323.6 nm, nitrogen ion spectrum line N II 588.7 nm. This indicates that Si atoms have transitioned during the collision, oxygen atoms and nitrogen atoms have been ionized. In figure 2(b), there are strong absorption peaks around 1366 nm and 1900 nm, which are caused by the water vapor absorption zone [26].

The spectrometer transfer functions are shown in figures 3(a) and (b), respectively, and baseline corrected spectral data is presented in figures 4(a) and (b). Using this processed spectral data by the SWM algorithm resulted in the calculated temperatures summarized in table 2, those are within a range of the corresponding wind tunnel temperature measurements. As expected, the calculated material temperature increased with airflow velocity.

In figure 5(a), the emissivity of the spectral band of 600–880 nm decreases with wavelength in all three experimental states. The near–infrared spectral band between 1200–2400 nm, shown in figure 5(b), also has an emissivity that increases with wavelength across all states. As the airflow was velocity increased, the temperature increased, and the emissivity increased from 600–750 nm. The emissivity reached a maximum when the air velocity in state 3 reached Mach 10. It may be that a long time airflow erosion heat accumulation and changes in surface roughness due to material oxidation. Figure 6(a) shows the spectrum at six time points, taken at 20 ms intervals after the airflow was terminated. The intensity of the spectrum decreases sharply in time. The maximum value of the spectrums relative intensity decreased to 2760 arb. u. in 100 ms, which was close to the relative intensity of the ambient background spectrum as show in figure 6(b). Figure 6(c) shows the baseline corrected spectrum, and figure 6(d) shows the spectral emissivity obtained by the SWM algorithm. The spectral emissivity increases with wavelength and significantly decreases in time. This rapid decrease can be attributed to
the termination of the hypersonic airflow, which stopped the energy injection at the sample surface. Once the material surface collisions stopped, the thermal radiation spectral intensity rapidly reduced, and the spectral emissivity significantly decreased may be that the subsequent solidification and crystallization at the surface of the hot melt material.

5. Measurement uncertainty and error

To determine the uncertainty of emissivity, the influence of each parameter should be considered [27–29]. Spectral emissivity and temperature of the sample in the wind tunnel are calculated by collecting the radiation spectrum of the sample and applying a SWM algorithm. In equation (5) $L_\lambda(T)$ is the measured radiation spectrum, $G(\lambda_i)$ is the spectral response function of the spectrometer, $\varepsilon(\lambda_i, T)$ and $L_b(\lambda_i, T)$ are the material spectral emissivity and blackbody radiation spectrum at temperature $T$, respectively. $\varepsilon'(\lambda_i, T')$ and $L_b(\lambda_i, T')$ are the environment spectral emissivity and blackbody radiation spectrum at temperature $T'$, respectively. However, because the
Experimental temperature exceeded 2000 K, and the measurement band of the spectrometer was less than 2.5 μm, environmental radiation can be ignored.

\[
L_i(\lambda_i, T) = [\varepsilon_i(\lambda_i, T) \cdot L_0(\lambda_i, T) + \varepsilon_i'(\lambda_i, T') \cdot L_0(\lambda_i, T')] \cdot G(\lambda_i)
\]

\[
L_i(\lambda_i, T) = \varepsilon_i(\lambda_i, T) \cdot L_0(\lambda_i, T) \cdot G(\lambda_i)
\]

Figure 5. Emissivity inversion result (a) visible band (b) near-infrared band.

Figure 6. Spectrum result (a) after the hypersonic airflow was stopped (b) ambient background spectrum (c) processed spectrum (d) inversion of spectral emissivity.

The input of the SWM algorithm is the measured spectral radiation information, particular concerns are the accuracy of the wavelength of the spectrometer $S_\lambda$, time stability of the measurement system $s_t$ and uncertainty.
of the material temperature \( T \) obtained by inversion. According to GUM (the guide to the expression of uncertainty in measurement), the combined uncertainty of emissivity can be expressed with the following equation (6) [30]. The relevant parameters of measurement uncertainty are illustrated in Table 3.

\[
u[e_i(\lambda, T)] = \left( \frac{\partial e_i}{\partial L_\lambda(\lambda, T)} \frac{\partial L_\lambda(\lambda, T)}{\partial (S_\lambda)} \right)^2 u^2(S_\lambda) + \left( \frac{\partial e_i}{\partial L_\lambda(\lambda, T)} \frac{\partial L_\lambda(\lambda, T)}{\partial (S_i)} \right)^2 u^2(S_i) + \left( \frac{\partial e_i}{\partial L_\lambda(\lambda, T)} \frac{\partial L_\lambda(\lambda, T)}{\partial T} \right)^2 u^2(T) \right)^{1/2} \tag{6}\]

To determine the relative error in the emissivity measurement system and algorithm, blackbody radiation spectrums at 2298 K and 1573 K were measured by the QE65Pro and Avantes 2.5–HSC–EVD spectrometers, respectively. According to standard blackbody furnace spectral emissivity 0.995, as shown by the red curves in figures 7(a) and (b). The measured spectrums were used to calculate emissivities using the SWM algorithm are shown in black. In the 420–900 nm range, the average emissivity is 0.968, and the average relative error is 2.7%. In the 1200–2400 nm range, the average emissivity is 0.976, and the average relative error is 2.1%.

6. Conclusion

In this paper, an arc plasma wind tunnel was used to simulate the aerodynamic heating process experienced by the outer layer of thermal protection material during spacecraft reentry. The materials radiation spectrum was collected by a grating spectrometer system. The spectral emissivity and temperature of quartz composite ceramics under different Mach numbers and different wavelength bands was inverted with the SWM algorithm. The measured spectral emissivities in the 600–750 nm wavelength range decreased with increasing wavelength and increased with increasing airflow velocity. The emissivity of the 1200–2400 nm wavelength range also increased with wavelength, and the spectral emissivity reached a maximum at the peak airflow velocity of Mach

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Table 3. Uncertainty of the emissivity measurements.

| Uncertainty contributions | Sub components | \( \lambda = 650 \text{ nm} \) | \( \lambda = 850 \text{ nm} \) | \( \lambda = 1400 \text{ nm} \) | \( \lambda = 2000 \text{ nm} \) |
|---------------------------|----------------|----------------|----------------|----------------|----------------|
| Spectrometer measurement signal | Accuracy of spectrometer wavelength | 0.0179 | 0.0105 | 0.0331 | 0.0162 |
| Time stability apparatus | 1.5179 | 2.6775 | 1.9766 | 3.4163 |
| Sample Temperature | Calculated temperature | 2.4724 | 2.9666 | 0.6302 | 0.2535 |
| Combined uncertainty (%) | / | 2.901 | 3.9962 | 2.0749 | 3.4257 |
10. After the airflow stops, the surface temperature drops sharply and the spectral radiation decreases accordingly. Emissivity uncertainty is mainly in the measurement of the radiation spectrum. Standard blackbody emissions were used to calculate the emissivity error of the system and the average relative error was 2.7% for the 420–900 nm wavelength region and 2.1% for 1200–2400 nm. This method can be used to study the spectral emissivity of materials in extreme cases when blackbody radiation spectra cannot be obtained under the same conditions, and to improve the accuracy of temperature measurement by the joint calculation of emissivity and temperature.

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