Equipment for the Spectral Characterization of High-Temperature Particles¹

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The spectral radiant characteristics of plume particles of a solid rocket engine are important in the design of the engine specific impulse, ablative material, and plume flame hiding. These parameters are measured from tests of the engine. Some equipment has been established to realize particle heating, uniform particle distribution, and measurements based on an FTIR spectral instrument. The equipment is based on SiC heating and is divided into a warm-up chamber and a measurement chamber to improve the particle temperature stability. A special design of uniform particle distribution combined with an acoustic levitation device is used to determine the particle falling speed. The spectral characteristics and the transmission rate of the particles have been measured by using the system including a standard blackbody, an assembled optical system, and an FTIR spectrometer. The measurements of particle concentration and temperature are given in detail. The instrument specifications are as follows: temperature range - 60-1500 °C; spectral range - 0.60-25 µm; and particle dimension range - 10-500 µm.

KEY WORDS: FTIR spectrum; particle levitation; particle radiation; particle uniform distribution.

1. INTRODUCTION

Thermal radiation from particulate-laden media often plays an appreciable role in industrial and atmospheric research involving fluidized beds, oil and gas-fired furnaces, radiative burners, solid propellant rockets, gas

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turbine combustors, internal combustion engines, natural fires, clouds, fog, and dust. The plume of a solid propellant rocket engine is full of high temperature particles that include Al_2O_3 , MgO, and ZrO₂ and emission gases with H_2O , CO_2 , and CO. The spectral properties of the plume of a solid rocket engine play an important role in the design of the engine specific impulse, ablative material, and plume flame hiding. The following methods have been applied in this area of research.

(a) Theoretical calculation: According to electromagnetic theory, the spectral complex refractive index of particles is first calculated, then by putting in the parameters of density, temperature, and dimension of the particles, the attenuation, scattering, and absorption coefficients can be calculated on the basis of the Mie scattering theory.

(b) Experimental measurement: The effective emissivity and the attenuation of a plume with solid particles can be determined by measuring the absorption and radiation in situ or in the laboratory [1-5]. The optical constant (complex refractive index) of the material is a basic physical property that is related to the composition, temperature, and surface condition. The surface of the particle material is much larger than that of the bulk material, and the high-temperature particles are easy to accumulate into a mass. Consequently, the surface of the high temperature particles is very complicated, and the complex refractive index of particles is much different from that of bulk material [6]. The radiation properties of materials and especially the radiation properties of particles must be given more and more attention. However, the experimental equipment for research on high-temperature optical properties of particles is complicated and difficult, so research on the complex refractive index of particles is mainly done at ambient temperature. Data of the complex refractive index of high-temperature particles are urgently needed in many fields. This work describes some equipment for realizing a flow with high-temperature particles and for measuring their properties at the same time.

2. MEASUREMENT PRINCIPLE

2.1. Transmittance and Emissivity Measurements

The transmittance of particles, τ_{ν} , is the fraction of radiation transmitted, the subscript indicating a quantity that varies with wavelength, λ :

$$\tau_{\lambda} = I_{\lambda} / I_{0\lambda} \tag{1}$$

where $I_{0\lambda}$ and I_{λ} are the incident and transmitted radiation, respectively.

The spectral emissivity of a particle material is the ratio of the measured spectral radiation of the particle sample and the spectral radiation of a blackbody at the same temperature and wavelength,

$$\varepsilon(\lambda, \mathbf{T}) = M(\lambda, T) / M^0(\lambda, \mathbf{T})$$
⁽²⁾

where $\varepsilon(\lambda, T)$ is the spectral emissivity; $M(\lambda, T)$ is the spectral radiation of the particle sample at temperature *T*; and $M^0(\lambda, T)$ is the spectral radiation of a black body at the same temperature *T*. We can get the spectral emissivity of particles at temperature *T* by measuring the spectral radiation of the particle material and the spectral radiation of a blackbody [7].

2.2. Theory and Method to Determine the Complex Refractive Index of Particles

Scattering and absorption of a solid particle with incident radiation is restricted by the boundary conditions of electromagnetism equations and its joint boundary surface. The system can be described with some physical modeling. The most general assumptions include: spherical particles, even quality, no magnetic field, smooth particles, and radiation from every particle. Mie derived his theory under the above-mentioned assumptions [8]. If the diameter of a particle is D, the complex refractive index of particle m = n - ik with a single wavelength (λ), then the incident radiation can be obtained by using the Mie theory. The equations are

$$Q_{\text{ext},\lambda} = \frac{4}{x^2} \operatorname{Re} \{S_0\} \\ = \frac{2}{x^2} \operatorname{Re} \left\{ \sum_{n=1}^{\infty} (2n+1)(a_n+b_n) \right\} = f(m,x)$$
(3)

$$Q_{s,\lambda} = \frac{2}{x^2} \sum_{n=1}^{\infty} (2n+1)(|a_n|^2 + |b_n|^2)$$
(4)

$$Q_{a,\lambda} = Q_{ext,\lambda} - Q_{s,\lambda} \tag{5}$$

where $Q_{\text{ext},\lambda}$, $Q_{\text{s},\lambda}$, and $Q_{\text{a},\lambda}$ are, respectively, the monochromatic extinction, scatting, and absorption coefficients of a particle. $\chi = \pi D/\lambda$ is a size parameter, S_0 is the forward scattering function, and a_n and b_n represent the Mie scattering efficiency. When particles can be considered a monodisperse system, the scattering of particles is independent with no reciprocal impact. When the incident light is parallel, the monochromatic transmission rate of particles τ_{λ} is

$$\tau_{\lambda} = \exp(-\kappa_{\lambda} \cdot L) \tag{6}$$

where L is the path length. We use the monochromatic extinction efficient, k_{λ} , to take the place of the monochromatic absorption efficient,

$$\kappa_{\lambda} = N \int_{D_{\min}}^{D_{\max}} \frac{1}{4} \pi D^2 \cdot P(D) \cdot Q_{\text{ext},\lambda}(m,x) dD$$
(7)

where N is the particle number density, $[D_{\min}, D_{\max}]$ is the diameter scale of particles, and P(D) is the particle size frequency distribution function. We adopt a log-normal distribution. According to Eq. (3), if P(D) and χ are given, then $Q_{\text{ext},\lambda}$ is only related to the complex refractive index m. However, since m is a complex number, the real and imaginary parts cannot be obtained simultaneously with only Eq. (3), so additional conditions are needed. According to the theory of dielectric dispersion, the complex refractive index $m(\lambda) = n(\lambda) - ik(\lambda)$ satisfies Kramers–Kronig relations,

$$n(\lambda) = 1 + \frac{2\lambda^2}{\pi} p \int_0^\infty \frac{k(\lambda_0)}{\lambda_0 \left(\lambda^2 - \lambda_0^2\right)} d\lambda_0 \tag{8}$$

$$k(\lambda) = \frac{2\lambda}{\pi} P \int_0^\infty \frac{n(\lambda_0) - 1}{\lambda^2 - \lambda_0^2} d\lambda_0$$
(9)

where *P* indicates the Cauchy principal value of the integral. When the $k(\lambda)$ spectrum is known, $n(\lambda)$ can be obtained from Eq. (8). So Eqs. (8) and (9) are the additional conditions for limiting the values of *n* and *k*. Combining Eqs. (3), (7), and (8), a unique *m* can be calculated [9–12].

From the above mentioned conditions, we can deduce the optical constants (the complex refractive indices) of small particles from the measured transmittance spectrum of a dilute polydispersion, by using the precise Mie scattering theory and the Kramers–Kronig dielectric dispersion relation.

3. EXPERIMENTAL EQUIPMENT

The equipment for measuring high-temperature spectral radiation characteristics of particles consists of a FTIR infrared spectrometer, a light source, a computer, a particle heating system, a temperature control system, a blackbody, a supplementary light path, a suspension system, and a particle distribution system. The equipment is shown in Fig. 1.



Fig. 1. Schematic diagram of meaurement system.

3.1. FTIR Spectrometer

The instrument is a JASCO FTIR-6100. The detector is DLATGS (silicon-photodiode detector) with a temperature regulator. The resolution is 0.5 cm^{-1} , and the measurement range is $15000.0-400.0 \text{ cm}^{-1}$.

3.2. Particle Warm-up and Measurement Chambers

The particles are heated through a preheating room with a carbonization silicon pipe within the temperature range from ambient to 1500°C. The particle pre-heating chamber and the particle measurements are in the same chamber, the temperature is monitored by a thermocouple and a pyrometer, and the heating section has appropriate temperature control to assure that the temperature of the particles in the pre-heating chamber and in the measurement chamber consistently do not change.

3.3. Optical System

The optical system uses collimated light. The radiation source area is larger than the detector surface area. The infrared spectrometer has transmittance and emissivity measurement paths. In transmittance measurements, the light through the interferometer which passes through the particles and reaches the detector is amplitude modulated. We can get the absorption spectrum of particles through the Fourier transform using the computer. But the emission light from particle radiation is not interfered.

3.4. Particle Distribution and Acoustic Levitation Device

The screening and the uniform distribution of particles are obtained by a two-level screen machine. The screen machine is driven and controlled by an electromotor. In order to guarantee uniform distribution of particles, an acoustic levitation device is adopted to reduce the flow drop speed. To prevent overheat of the acoustic transducer, we use a water-cooling system to control the temperature of the transducer.

3.5. Test Measurement

The laboratory equipment is currently under development; so we do not yet have any experiment data. However, the transmittance of plastic film and spectral distribution of acetylene flame have been measured with FTIR, as shown in Figs. 2 and 3.

4. CONCLUSION

We have described some specific laboratory equipment built for studying the high-temperature optical properties of particulate materials. The infrared emissivity and transmittance of particles (such as Al_2O_3) can be



Fig. 2. Transmittance of plastic film.



Fig. 3. Emissivity of acetylene flame.

measured from 60 to 1500 °C over the spectral range of 0.60–25 μ m with particle dimensions ranging from 10–500 μ m.

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