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Effect of surface morphology and densification on the infrared emissivity of C/SiC composites

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ABSTRACT

The effects of surface morphology and densification on the infrared emissivity of 2D C/SiC composites were investigated in 6–16 μm from 1000 °C to 1600 °C. As the sample surface was polished, the reflection and scattering for the electromagnetic waves of thermal radiation were reduced, causing a sustained decrease in the infrared emissivity. The space-variant polarizations caused by the cauliflower-like microstructure were enervated in the smooth surface, which enhanced the reduction trendy in the infrared emissivity. In densification process, the increasing SiC content and the growing amount of the cauliflower-like microstructure on sample surface improved the infrared emissivity of C/SiC composites, while the decreasing porosity decreased it. Due to the greater positive effects on the thermal radiation during the densification process, the infrared emissivity of C/SiC composites increased successively with density.

1. Introduction

As an important ceramic matrix composites (CMCs), C/SiC composites are the most promising candidate materials in the spacecraft reentry thermal protection system (TPS) applications, such as sharp leading edges or nozzle ramps, on the next generation hypersonic reentry vehicles [1,2]. C/SiC composites exhibit the good performance under severe environments, especially under high temperatures over 1000 °C. For the thermostructural composites, the radiative heat transfer is very important to dissipate aeroheating with rising temperature levels and may be totally dominant over conduction and convection at high temperatures. The same reasons also make the radiative heat transfer the only method in outer space applications for heating and cooling. Therefore it is necessary to investigate the thermal radiation properties of C/SiC composites, especially under high temperatures, which is crucial for calculating temperature distributions and the heat transfer in service. A relative measure of the radiative properties of materials is the emissivity, ε. The emissivity is defined as the ratio of the energy intensity radiated by a actual material to that radiated by a black body at the same conditions, which determines the radiative performance of the real body [3,4]. The analysis of thermal radiation is further complicated by the behavior of the radiative properties of materials. Numerous methods are usually applied to study the infrared emissivity of materials. One method of determining the emissivity is to measure the reflectivity, and the emissivity and the reflectivity is complementary to each other in the opaque materials according to Kirchhoff's law [5]. Another way is to directly compare the radiation intensity from the materials with that from a perfect emitter (black body), the ratio of which yields the emissivity value [6–8]. In the high temperature measurements, the direction method is usually applied.

As an important thermostructural material, many studies have been done to know the properties of C/SiC composites, but most researches for C/SiC composites are mainly focused on the mechanical properties and oxidation resistance. So far, few works have been done to know the relationship between the radiation heat transfer and the microstructure of C/SiC composites, especially under vacuum and high temperatures [9]. The studies about C/SiC with SiC coating have presented that SiC coating has an obvious effect on the spectral emissivity, but causes just about 5% difference in the total emissivity [10]. Other studies about C/SiC in the temperature range 1000–1600 °C display high emissivity with an average value of about 0.7, and the pressure changing from 4 Pa to 200 Pa has limited effect on the emissivity due to the limited oxidation on SiC coating [11]. The influence of the microstructure evolution on the radiative heat transfer of C/SiC composites is still unclear. More experiments are required to investigate the relationship between

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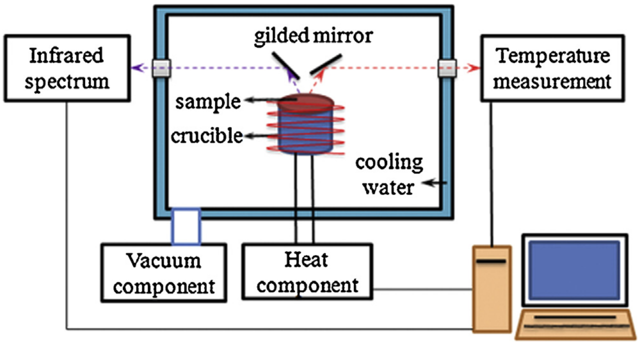

Fig. 1. Schematic diagram of the infrared emissivity measurement system.

surface morphology, microstructure and infrared emissivity properties of C/SiC composites.

In our present work, the effects of surface morphology and densification process on the infrared emissivity of C/SiC composites are investigated through the direct method. The propagation of electromagnetic waves on the different surface morphology is deeply analyzed. The microstructure changings during densification process, such as chemical component, surface morphology and porosity are analyzed to know their effects on the infrared emissivity properties of samples.

2. Experimental details

2.1. Samples preparation

The stacking plain weave carbon cloths (T300) were used as the preforms. The preforms were fixed in a perforated graphite holder which were tightened to obtain the desired fiber volume fraction (generally 40 vol%). Pyrolytic carbon (PyC) interphase and SiC matrix were deposited by low pressure chemical vapor infiltration (LPCVI) process to prepare 2D C/SiC composites. Propylene (C_3H_6) and Methyltrichlorosilane (MTS, CH_3–$SiCl_3$) were used as the carbon source and the SiC precursor for deposition of PyC and SiC matrix, respectively. Argon was employed as the dilute gas to slow down the chemical reaction rate of deposition. The more details about the deposition conditions could be seen in the previous papers [10,12]. The as-produced C/SiC composites were then machined and polished to the specimens with a dimension of $6.0 \times 6.0 \times 3.0\,\mathrm{mm}^3$. SiC coating was prepared on the samples surface under the same conditions with that of SiC matrix.

For getting different surface morphologies, the composites surface was polished by a series of abrasive papers with increasing fine grit. In the paper, 400# grit SiC polished samples, 1500# grit SiC polished samples, and W0.5# emery grinding grease polished samples were denoted as sample A–C, respectively. By adjusting the preparation time, C/SiC composites with different density were prepared. Those samples with increasing density were denoted as sample D1–D4, respectively.

2.2. Characterizations and measurements

In this paper, for directly getting the infrared emissivity of samples, a special measurement system was applied as shown in Fig. 1. In the experimental process, the sample was placed in a tungsten crucible which located in the center of a inductance furnace. For eliminating the air disturbance, a turbomolecular pump in series with a mechanical pump was equipped to provide ultrahigh vacuum environment (~10^{-4} mbar). The sample was heated by the inductance furnace and the sample temperature was monitored by an ultraviolet photon pyrometer which had been calibrated by a Pt–Rh/Pt thermocouple. When the sample was heated to the desired temperature, the infrared spectrum $M(\lambda,T)$ of sample was detected by a Fourier-transform infrared (FTIR) spectrometer and a liquid nitrogen-cooled mercury–cadmium–telluride (MCT) detector from 6 μm to 16 μm. C/SiC composites as the thermostructural materials were usually suffered high temperatures. Therefore, the chosen testing temperature range to the infrared emissivity should approximately contain main service temperature scope, i.e. 1000–1600 °C.

According to the definition of the direction method, the spectral emissivity of sample was determined as follows:

$$\varepsilon(\lambda, T) = \frac{L_s(\lambda, T)}{L_b(\lambda, T)} \tag{1}$$

where, $L_s(\lambda,T)$ and $L_b(\lambda,T)$ represented the thermal radiation intensity of the sample and the blackbody at given wavelength λ and temperature T, respectively. The thermal radiation intensity emitted by a blackbody at given temperature T could be calculated by Planck's law:

$$L_b(\lambda, T) = \frac{C_1}{\lambda^5\left[\exp\left(C_2/\lambda T\right) - 1\right]} \tag{2}$$

where $C_1 = 3.742 \times 10^{-16}\,\mathrm{W{\cdot}m^2}$ was the first radiation constant and $C_2 = 1.439 \times 10^{-2}\,\mathrm{W{\cdot}K}$ was the second radiation constant.

It should be noted that the infrared spectrum of samples $M(\lambda,T)$ measured in the tests was affected by the detectivity of detector, the relative position of equipments and other nonlinear responses. Those factors made the experimental data deviate the actual thermal radiation intensity of samples. So a system function taking into account those factors was needed. In this system, a reference blackbody was measured at several temperatures under the same condition as samples to get the spectral response function of system $F(\lambda,T)$. Therefore, the thermal radiation intensity of sample $L_s(\lambda,T)$ could be expressed as:

$$L_S(\lambda, T) = F(\lambda, T)M(\lambda, T) \tag{3}$$

The final measurement equation used to determine the spectral emissivity is

$$\varepsilon(\lambda, T) = F(\lambda, T)\frac{M(\lambda, T)}{L_b(\lambda, T)} \tag{4}$$

The more details about the measure principle of this system could be got in Ref. [13].

The total emissivity at a given temperature ε_T could be calculated from the spectral emissivity by the integral method over all the considered wavelengths range λ_1–λ_2 [14]:

$$\varepsilon_T = \frac{\int_{\lambda_1}^{\lambda_2} \varepsilon_\lambda E_{b\lambda}\,d\lambda}{\int_{\lambda_1}^{\lambda_2} E_{b\lambda}\,d\lambda} = \frac{\sum_{\lambda_1}^{\lambda_2} \varepsilon_\lambda E_{b\lambda}\,\Delta\lambda}{\sum_{\lambda_1}^{\lambda_2} E_{b\lambda}\,\Delta\lambda} \tag{5}$$

where $E_{b\lambda}$ represented the thermal radiation intensity of the ideal blackbody at wavelength λ and temperature T.

The samples were characterized with regard to their surface microstructure by scanning electron microscopy (SEM, HITACHI S4700).

3. Results and discussion

3.1. Effect of surface morphology

Fig. 2 presents the spectral emissivity of C/SiC composites with different surface morphologies at 1000 °C and 1300 °C. At the two measurement temperatures as seen in Fig. 2, the spectral emissivity curves of all three types samples showed a special "*V*" shape during 10–14 μm. This appearance in the spectral emissivity was a typical feature of infrared emissivity for SiC. According to the oscillator