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Study of IR absorption properties of fumed silica-opacifier composites

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ABSTRACT

Mineral powders were added into fumed silica as IR opacifers in order to develop opacified thermal insulating composites for application at high temperatures up to 1000 °C. The opacifying effect was evaluated with effective specific extinction. The results indicated that opacifiers could effectively block IR radiation heat transfer and improve thermal insulating properties of fumed silica. Among four opacifiers investigated in this paper, SiC was proved to be the best one for excellent extincting performance. When SiC was adopted with average particle size of 3.029 μm and mass ratio of 25%, the values of effective specific extinction increased from less than 12.6 m^2/kg to about 55 m^2/kg in the wavelength range from 2.5 to 7 μm. It was demonstrated that fumed silica-opacifier composites are efficient high temperature insulating materials.

1. Introduction

Fumed silica is a versatile fine particle that delivers superior performance for diverse applications, such as fillers for rubbers and plastics, coatings, adhesives, etc. In recent years, fumed silica has been used as thermal insulators with high specific surface area, porosity and low density [1,2]. It has been demonstrated that fumed silica is an effective thermal insulating material with low thermal conductivity of about 0.030 W/m K under ambient conditions (25 °C and atmospheric pressure). However, at high temperatures (<1000 °C) its thermal conductivity rises so rapidly that consequently prevents its development and application. Three primary contributions to thermal conductivity arise from solid conduction, gas convection and radiation. The solid conduction is little dependent on temperature but determined by density. The nanoporous structure of fumed silica restricts free convecting air. Meanwhile, fumed silica shows low radiation extinction for wavelengths below 7 μm like silica aerogel [3–7]. Therefore, a surge of thermal conductivity mainly lies in radiation heat transfer at high temperatures.

To solve the problem of applications at high temperatures, mineral powders are introduced into fumed silica as IR opacifiers. The particles of opacifiers can cause radiation waves to be redirected from their original path, in other words, thermal insulating properties will be significantly improved due to low radiation transmittance. In this paper, four opacifiers were adopted and the effects of particle size and mass ratio of SiC on effective specific extinction were investigated.

2. Experimental

2.1. Materials

Fumed silica, AEROSIL200 from Degussa, was used as matrix material with specific surface area (BET) of 200 m^2/g and average primary particle size of 12 nm. E-glass fiber was mixed into fumed silica to increase mechanical strength, and its average diameter was 7 μm, length 5–10 mm. Generally opacifiers should be with properties of high refraction index and superior chemical stability at high temperatures. In this experiment, SiC, $ZrSiO_4$, KT_6 and BN were selected as IR opacifiers, whose refraction index and average particle size were shown in Table 1.

2.2. Preparation of composites

Fumed silica, E-glass and opacifiers with appropriate mass ratios were mixed together with MJ-250 high-speed multifunctional disintegrator for 60 s. Although a homogeneous mixture would give better results, excessive grinding of the mixture may possibly break up fibers and lead to the decrease of mechanical strength. The mixture was treated with vapor comprising water such that the vapor was absorbed into fumed silica, so as to produce a vapor-treated composite [8]. The presence of water in composites could reduce the volume springback. After dried at 150 °C for 2 h, the mixture was pressed into films, which should be homogenous and smooth in appearance. So the samples for IR

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Table 1
Properties of IR opacifiers adopted in experiments.

Opacifier		Refraction index	Average particle size (μm)	Sources
SiC	SiC_1	2.468–2.691	0.877	Beijing Venus Ultrafine Materials Co., Ltd.
	SiC_2		1.969	
	SiC_3		3.029	
	SiC_4		4.314	
$ZrSiO_4$		1.93–2.01	2.216	Zibo Yongbang Zirconium Co., Ltd.
KT_6		2.21	10	USTB
BN		1.53	0.837	USTB

The uncertainty of average particle size is less than 1%.

transmittance measurement were prepared, and the data for each sample should be recorded clearly including the mass, thickness and diameter.

2.3. Characterization

Generally the radiative conductivity, $k_{rad}(T)$, is considered as a basic parameter, through which we can clearly understand the degree of radiative heat transfer. When opacifiers added into fumed silica, $k_{rad}(T)$ would obviously be changed due to scattering or reflection. These effects were described by effective specific extinction, e^*. The relation between $k_{rad}(T)$ and e^* may be expressed as [3]

$$k_{rad}(T) = \frac{16n^2\sigma}{3e^*(T)\rho}T_m^3, \quad (1)$$

where n is the effective refractive index, σ is the Stefan–Boltzmann constant, $\sigma^* = 5.669 \times 10^{-8}$ W/m^2 K^4, ρ is sample density, and T_m is the mean temperature. For an exact evaluation of IR extinction performance of opacifiers, e^* was adopted in this paper.

For calculating the values of e^*, IR transmittance, τ, of samples should be measured firstly by NEXUS-670 Fourier transform infrared spectrometer (FT-IR, spectral range 4000–400 cm^{-1}). The equation for IR transmittance can be written as [9]

$$\tau = \frac{I}{I_0} = e^{-\alpha h}, \quad (2)$$

where I and I_0 are incident IR intensity and transmitted IR intensity, respectively, h is sample thickness and α is specific extinction. In terms of specific extinction, it can be replaced by

$$\alpha = e^*\rho, \quad (3)$$

where ρ is related to sample mass, m, and volume, V. Therefore, according to Eqs. (2) and (3), the effective specific extinction, e^*, can be deduced as

$$e^* = -\frac{V}{mh}ln\left(\frac{I}{I_0}\right). \quad (4)$$

Thermal analysis of the composites was carried out using STA409–QMS derivatograph (Netzsch). Other operating conditions were as follows: the heating rate was 10 K/min in the air atmosphere; sensitivities of differential thermal analysis (DTA) and thermogravimetric analysis were 0.1 μV and 0.1 μg, respectively.

3. Results

As the investigations described here are mainly performed in order to find an effective opacifier for fumed silica thermal insulators, the samples are evaluated according to effective specific extinction, e^*, in the wavelength range from 2.5 to 7 μm. SiC used in this experiment was a mixture of SiC_1, SiC_2, SiC_3 and SiC_4 with the same mass ratio. In Fig. 1, the values of e^* for no opacifier are very low just only in the range from 1.9 to 12.6 m^2/kg. The result indicates that fumed silica without opacifiers is nearly transparent

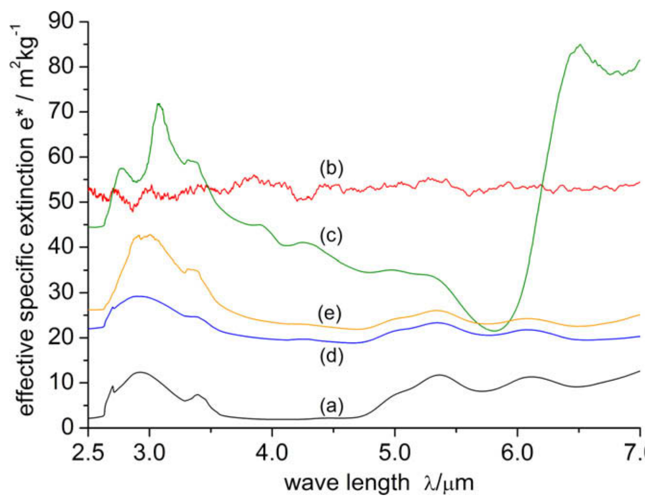

Fig. 1. Effective specific extinction, e^*, versus wavelength, λ, of fumed silica opacified with 20 wt% of various mineral powders: (a) no opacifier; (b) SiC; (C) BN; (d) $ZrSiO_4$; (e) KT_6.

for IR radiation in this wavelength range. In other words, radiation heat flux can freely transfer through fumed silica in this wavelength range. However, when opacifiers are adopted, the values of e^* can be significantly improved, especially for SiC. The values of e^* for the sample with SiC get up to about 50 m^2/kg and keep little changed. It indicates that SiC as an opacifier can scatter IR radiation more efficiently compared with other three powders. Though the values of e^* for BN are higher than those of SiC in the wavelength from 2.5 to 3.5 μm, they rapidly decrease in the wavelength from 3.5 to 6.5 μm. Therefore, SiC is an ideal opacifier for fumed silica because of excellent extincting ability and constant stability in a wide wavelength range.

Fig. 2 shows TG–DTA curves of fumed silica-SiC composites. The TG curve shows that there is little mass loss from 18 to 910 °C, and the biggest loss is about 1.37% due to the evaporation of water contained in the composites. The DTA curve reveals that there exists a single endothermic peak from 100 to 488 °C, which may be ascribed to the high heat capacity of fumed silica. TG–DTA curves also indicate that there is no phase transformation in the temperature range. Therefore, it is concluded that fumed silica-SiC composites have an excellent chemical and thermal stability in applications at high temperatures.

In order to investigate the influence of particle size on e^*, four SiC powders were introduced into fumed silica. The values of e^* in Fig. 3 show a trend of first increase and then decrease with the increase of particle size. For shorter wavelengths, the values of e^* keep high for all four SiC powders. However, for larger wavelengths, the situations will greatly change. The values of e^* for SiC_3 and SiC_4 can keep constantly stable, but for SiC_1 and SiC_2 there have been drastic fluctuations of e^*. From results in Fig. 3, we can