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Microscopic thermal diffusivity measurements of ceramic and metallic foams lumps in temperature

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ABSTRACT

This paper presents temperature dependent thermal diffusivity measurements of NiCrAl and mullite foam solid skeletons. The photoreflectance microscopy (PM) technique is used to characterize these skeletons at the microscopic scale. The characterized homogeneous zone area does not exceed 100 μm^2. A singular sample preparation has been realized to take into account the brittleness and the optical semi-transparency of the mullite foam lumps. This specific sample preparation as well as the details of the technique are described in this article.

1. Introduction

Metal and ceramic open-cell foams usually exhibit an open-cell structure with porosities ranging from 0.8 to 0.95. They are, now, widely used in many industrial applications where heat transfer is important, such as compact heat exchangers [1], fire barriers [2] and volumetric absorbers in receivers of concentrator solar systems [3]. They are also applied in advanced energy and combustion systems, such as low-NOx combustion burners [4–6]. In the majority of these applications, due to the high level of temperature and/or the very high porosity of the material, the total heat transfer is due to both conduction and thermal radiation. Temperature-dependent thermal conductivity of solid phase (skeleton)affects thermal energy transport through the foam [7–9]. The structural characteristics (such as porosity, shape and cell size) and solid phase properties influence the thermal transport.

Theoretical models are very useful to investigate the influence of the parameters characterizing the microstructure on the foam equivalent thermal conductivity and to optimize foam thermal performances. Some noticeable improvements have been achieved in the field recently. New models designed to predict conductive and radiative coupled heat transfer in low-density foams have been proposed. The models developed to predict the conductive [10–12] and radiative properties [13–20] try to take into account more faithfully the real morphologies. Predictive models of radiative properties take into account more and more precisely the scattering behavior of the solid matter.

However, it is difficult to find in literature, experimental data on the thermal properties of solid phase of foams, for the temperature range of interest. Thus the determination of local thermal properties remains an open problem. In this current work, to overcome this difficulty, we propose to use the photoreflectance microscopy (PM) technique to characterize the skeleton at the microscopic scale. We present temperature dependent thermal diffusivity measurements of NiCrAl and Mullite foam solid skeletons.

2. Microscopic photothermal measurement – thermal wave

Photothermal techniques consist in measuring one of the numerous secondary effects that occur after non-steady-state absorption of light energy by the sample analyzed (Fig. 1). These effects are generally used to detect an increase in the temperature of

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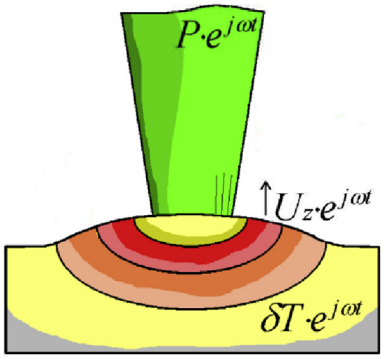

Fig. 1. Photothermal experiment. Temperature rise δT and periodic surface displacement U_z caused by the absorption of a modulated light flux P (pulse ω).

the medium studied, and thereby to characterize local thermal properties. Among the different photothermal techniques, photoreflectance [21] has the highest spatial resolution (≈1 μm), allowing for measurements at a highly local scale (a few cubic micrometers). It consists in detecting heating-induced variations of the sample's reflection coefficient. The local thermoelastic properties can also be studied with the use of an interferometer in the experimental setup, to detect the normal displacement of the sample surface due to the thermal expansion caused by the temperature increase [22,23].

When a laser beam (pump beam) with power Q and intensity-modulated at frequency $f = \omega/2\pi$, is focused on the surface of a thermally thick and isotropic sample, the periodic temperature increase observed in the medium at a distance r from the supposedly infinitesimal heat source is equal to [24]:

$$\delta T(r,t) = \frac{Q}{4\pi k r}\exp\left(-\frac{r}{\mu}\right)\cos\left(\omega t - \frac{r}{\mu}\right) \text{ where } \mu = \sqrt{\frac{\alpha}{\pi f}} \qquad (1)$$

k represents the thermal conductivity of the material and α its thermal diffusivity. Equation (1) shows that the decrease in periodic temperature rise is exponential. The temperature rise is confined to a volume with a characteristic dimension μ (thermal diffusion length) that decreases as the thermal diffusivity of the medium decreases or the modulation frequency f increases. For example, at a frequency of 2 MHz (corresponding to the passband of the instrument), μ does not exceed 5 μm. A concentrated heat source (i.e. a highly localized laser beam) associated with a high operating frequency will therefore confine the temperature increase to a microscopic volume. Moreover, it is observed that the phase shift between the local temperature rise and the periodic heating decreases linearly with r. The slope of this linear variation is equal to $-1/\mu$. This propagating behavior explains why the term 'thermal wave' is used to describe the phenomenon. It allows to simply use temperature measurements obtained by photothermal microscopy. The thermal diffusivity of the isotropic homogeneous materials is derived from the slope of the phase shift using equation (1).

Practically, to reach the required spatial resolution, the convolution effects due to the energy distributions of probe and pump beams must be taken into account in our analysis. Assuming gaussian energy profiles, numerical calculations are required to estimate the exact periodic temperature increase i.e. to convolve the Green solution (1) with the energy distribution of laser beams.

As example Fig. 2 shows the pertinence of this procedure in the case of a tantalum sample photoreflectance characterization. Several measurements are performed at various frequencies. Experimental data points are then automatically fitted using a least square minimization procedure by adjusting both the thermal diffusivity of the sample and the gaussian radii of pump and probe beams. The Levenberg-Marcquard algorithm is used to minimize the function χ:

$$\chi^2 = W\sum_{mes}\sum_{i}\left(A_{cal}(R_i) - A_i - \Delta A_{mes}\right)^2 + \sum_{mes} \times \sum_{i}\left(\varphi_{cal}(R_i) - \varphi_i - \Delta\varphi_{mes}\right)^2 \qquad (2)$$

with

$$R_i^2 = (r_i - \Delta r_{mes})^2 + d_{mes}^2$$

A and φ correspond respectively to the attenuation and the phase of the signal, and Δr_{mes}, ΔA_{mes}, $\Delta\varphi_{mes}$ and d_{mes} are additional optimization parameters taken into account notably micro positioning.

We must mention that the minimization procedure attributes a greater weight to phase estimation (directly diffusivity dependent) rather than attenuation (factor W). It means that larger discrepancies can appear between attenuation measurement and its least square adjustment.

Usually, the relative uncertainty on the thermal diffusivity estimation is less than few percent and the identified equivalent gaussian radius at e^{-1} is smaller than 1 μm [25].

3. Photoreflectance measurement principle

To perform a photothermal experiment at such a local scale, the only available technique is photoreflectance microscopy. It consists in measuring, with a photodiode, the light flux of a continuous laser beam (intensity I_0), called probe beam, after it has been reflected on the sample surface in the periodically heated zone. If the temperature variation is small, the reflection coefficient, R of the surface can be assumed to vary linearly with temperature according to relation (3):

$$\delta R(r,t) = R_0\left[1 + \frac{1}{R_0}\frac{\partial R}{\partial T}\delta T(r,t)\right] \qquad (3)$$

After reflection, the a.c. component of the probe beam intensity $\delta I(r,t)$ is directly proportional to the temperature increase.

$$\delta I(r,t) = R_0.I_0.\left[\frac{1}{R_0}\frac{\partial R}{\partial T}\delta T(r,t)\right] \qquad (4)$$

These intensity variations can be detected with a photodiode whose signal is filtered by a lock-in amplifier. If the amplitude of the signal depends on both thermal and optical parameters, on the other hand the phase term is independent of optical artefacts and only depends on the thermal diffusivity of the material.

4. Description of the experimental setup

The photothermal microscope conceived to perform these measurements is shown in Fig. 3. It must ensure four functions:

- position and focus the pump beam,
- measure the variations of the reflection coefficient,
- Detect by interferometry the normal component of the sample surface displacement
- Control measurement automation and data storage.

The pump beam is produced by a 2 W Coherent Verdi laser (wavelength 532 nm). Its power is modulated by an acousto-optical modulator, at a frequency fixed by a function generator. The beam is