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Determination of thermal diffusivity of ceramics by means of photothermal beam deflection

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Abstract. The non-contact and non-destructive photothermal beam deflection (PBD) method has been used for the characterization of the thermal properties of Alumina (Al_2O_3), Mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) and Oxidic-Bonded Silicon Carbide (OBSiC) samples often used in chemical engineering. Measurements of thermal diffusivity κ have been performed. One obtains $\kappa = 7.96 \cdot 10^{-6} \frac{\text{m}^2}{\text{s}}$ for Alumina, $\kappa = 1.94 \cdot 10^{-6} \frac{\text{m}^2}{\text{s}}$ for Mullite and $\kappa = 3.91 \cdot 10^{-6} \frac{\text{m}^2}{\text{s}}$ for OBSiC.

1. Introduction

Solid ceramic sponges are open-celled foams with porosities up to 85 % and were developed for the purification of liquid metals. Actually, ceramic sponges are of high interest due to their advantageous properties like low pressure drop, large specific surface area and good radial heat conductivity compared to packed beds of spherical particles for example. Therefore, possible application fields in the future could be burners for combustion, catalytic reactors or packings in columns. For designing such equipment, the knowledge of the thermal diffusivity κ of the solid ceramic network body of the sponges is essential (see [1]). Due to impurities in the ceramic suspension, literature data can not be used. In this contribution, a non-contact and non-destructive method for the determination of the thermal diffusivity of samples made of Alumina, Mullite and Oxidic-Bonded Silicon Carbide (OBSiC) are presented. The method used was the photothermal beam deflection (PBD).

An alternative method often used for the determination of diffusivities is the 3 D photothermal radiometry (PTR), which has been successfully applied for example on diffusivity measurements of SiC-based fibres [2]. However, the classical 3 D PTR technique has the limitation, that IR radiation not coming from the sample cannot be distinguished from the heat radiation originating from the sample of interest. Radiation coming from deeper layers of the sample decreases rapidly below the detection limit of the mercury cadmium tellurite (MCT) detector.

For the verification of the results presented in this contribution, laser flash as standard method for diffusivity measurements was applied [3]. However, the disadvantage of the laser flash method compared with PBD is the higher sample standard.

2. Theoretical background

By heating a sample with a focused continuous wave (cw) laser beam, a thermal wave in dependence of the material properties develops. This thermal wave changes the properties of the air directly above the sample. In the case of PBD a second laser beam, the probe laser, is focused parallel and close to the sample surface. The refractive index gradient in the air causes a deflection of this laser beam (Figure 1), whereas the deflection angle is given by the following line integral [4]:

$$\phi = -\int_P \frac{1}{n} \cdot \frac{dn}{dT} \cdot \nabla T_g \times dl \quad (1)$$

where P is the probe beam path, n the refraction index of air, T_g the periodic temperature distribution in the air, and dl an incremental distance along the pathway P . In the case of small temperature changes, the gradient of the refraction index is assumed to be constant and can be removed from the integral.

The deflection ϕ can be resolved into two components: beam deflection normal to the sample surface, ϕ_n , and beam deflection parallel to the sample surface, ϕ_t , [4]. In this contribution, thermal diffusivity measurements of different ceramic materials have been performed by considering only the transverse deflection ϕ_t of the probe beam due to the experimental set-up. Using a lock-in amplifier one obtains from the signal of ϕ_t the amplitude and the phase due to the modulation of the pump beam. Applying the zero-crossing (ZC) method afterwards, the real part (Re) and the imaginary part (Im) of ϕ_t were determined by the following equations:

$$Re(\phi_t) = \text{amplitude}(PBD) \cdot \cos\left(\frac{(\text{phase}(PBD) + \Delta) \cdot \pi}{180^\circ}\right) \quad (2)$$

$$Im(\phi_t) = \text{amplitude}(PBD) \cdot \sin\left(\frac{(\text{phase}(PBD) + \Delta) \cdot \pi}{180^\circ}\right) \quad (3)$$

The real part of ϕ_t determined with the lock-in amplifier in the experiment is not related to a phase shift of 90° at the central zero crossing due to the finite height H of the probe beam above the sample surface (see Figure 1). For obtaining the correct real part of ϕ_t considering the phase shift of 90° (called true zero crossing), the phase must be corrected by Δ applying a correction procedure [5, 6, 7]. The correction procedure is described in section 4.

The ZC method for the determination of the thermal diffusivity is based on the linear relation between the first non-central crossing distance d of the real part of ϕ_t (see equation 2) divided by the amplitude (PBD) against the inverse root of the modulation frequency $f^{-0.5}$. The slope m of the linear relation between d and $f^{-0.5}$ is related to the thermal diffusivity κ expressed by the following equation [6]:

$$\kappa = \frac{m^2}{\gamma \cdot \pi} \quad \text{with } \gamma = 1.44 \quad (4)$$

where γ is a parameter which depends on the bulk thermo-optical properties of the material.

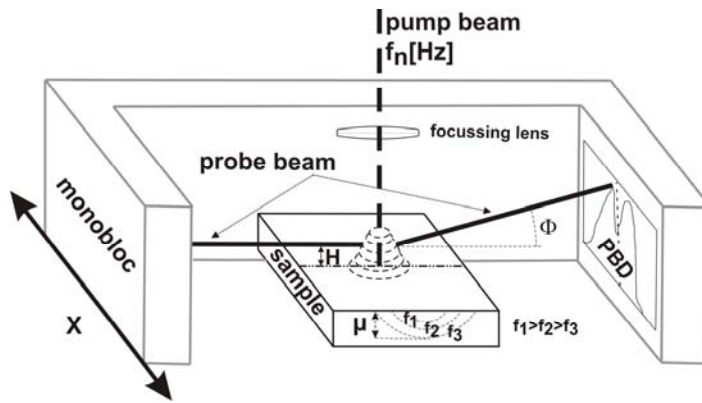
3. Experiments

3.1. Investigated ceramic samples

The investigated ceramic samples were opaque cylinders of 12 mm in diameter and 3 mm in height. They were made of Alumina (Al_2O_3), Mullite ($3 \text{Al}_2\text{O}_3 \cdot 2 \text{SiO}_2$) and Oxidic-Bonded Silicon Carbide (OBSiC). The cylinders were prepared by pressing the powder and sintering it afterwards. The exact manufacturing process of the samples is described in Dietrich et al. [1]. To optimize the absorption of the pump laser, the samples were blacked with a marker.

3.2. Experimental set-up

The experimental set-up used in this work is shown in Figure 1. The sample was heated by a focused Nd-YAG laser, called pump laser in the following (wavelength 532 nm, cw power 60 mW, model DPY315II, ADLAS Luebeck, Germany). The laser beam was guided perpendicular to the sample and modulated by a mechanical chopper (from 4 Hz to 46 Hz). The focus point of the laser beam had a diameter of about 100 μm . A He-Ne laser (wavelength 633 nm, power 1 mW, model 1101P, JDS Uniphase, Mantea, USA) has been used as probe beam source. The probe beam has been focused parallel to the sample surface with a distance H of maximal 100 μm . The transversal probe beam deflection signal (transverse component ϕ_t) was detected by a four-quadrant position detector and processed by means of a lock-in amplifier. For scanning the complete geometry of the thermal wave, the probe beam and the detector were integrated in the PBD apparatus [7]. In all experiments the diffusion length (see equation 5) was twice as large as the grain size in the minimum [1].



μ is identified with the diffusion length of the pump beam calculated by

$$\mu = \sqrt{\frac{\kappa}{\pi \cdot f}} \quad (5)$$

Figure 1. Experimental set-up of PBD method

3.3. Experimental procedure

After switching-on the pump laser, the experiments were started after 30 minutes stabilisation delay with a chopper frequency of 46 Hz. The beam deflection of the probe beam was recorded at the detector in a step size of 50 μm through the thermal wave (integration period 1 sec.). Therewith, values for the phase shifting and the amplitude in dependency of the position in the thermal wave could be determined. After crossing the complete wave the next lower frequency was selected. In literature, the method used in this work is often called “skimming method” [6].

4. Experimental results

First, the real part $Re(\phi_t)$ and the imaginary part $Im(\phi_t)$ of ϕ_t (transversal deflection) were determined (see equations 2 and 3). As described above the phase shift Δ had to be found out by a correction procedure, because Δ cannot be determined experimentally. The correction procedure used in this contribution is presented in Figure 2.

If $\Delta = 0^\circ$ is assumed, one obtains the Argand diagram ($Im(\phi_t)$ versus $Re(\phi_t)$) for all investigated frequencies as shown in Figure 2 top left exemplary for OBSiC. The diversification of the graphs in all four quadrants is caused by the phase shift Δ . Therefore the real part of ϕ_t divided by the amplitude of PBD Signal versus the monobloc position x was plotted (see Figure 2 bottom, step 1). Δ was varied until the peaks (marked with the large black points) matched the values 1 and -1 respectively (equal to a phase shift of $+90^\circ/-90^\circ$, as described in literature [7]). After this

procedure, a further Argand diagram was plotted and one obtains all graphs orientated in one direction and enclosing themselves (see Figure 2 top right, step 2). Afterwards the zero-crossing distance d can be determined in the diagram (bottom, step 3).

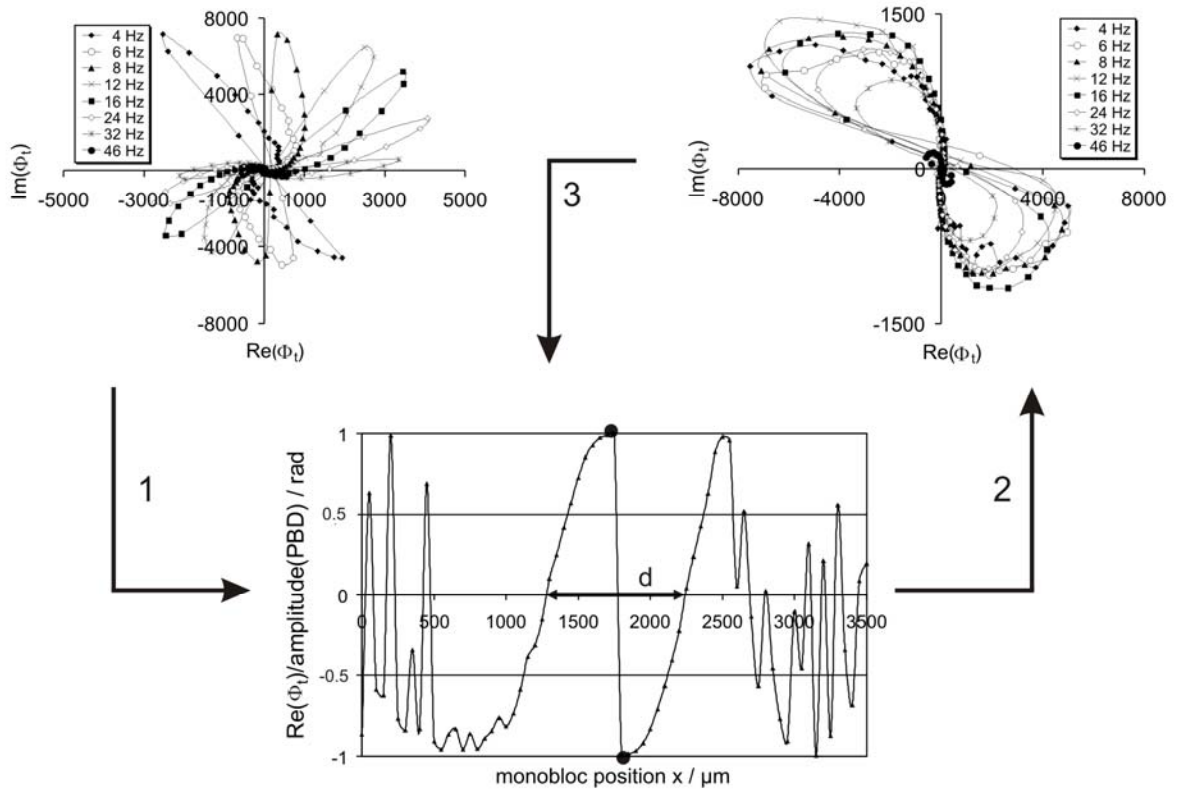
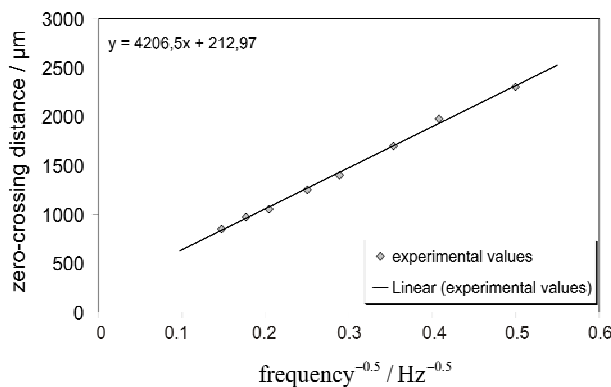


Figure 2. Procedure for the determination of the zero-crossing distance (exemplary shown for OBSiC)

Finally, the thermal diffusivity can be obtained from the slope of the experimental values in a diagram zero-crossing distance versus the square root of the inverse chopper frequency (see Figure 3) and equation 4. One obtains $\kappa = 7.96 \cdot 10^{-6} \frac{m^2}{s}$ for Alumina, $\kappa = 1.94 \cdot 10^{-6} \frac{m^2}{s}$ for Mullite and $\kappa = 3.91 \cdot 10^{-6} \frac{m^2}{s}$ for OBSiC. Control measurements with laser flash showed a deviation of 9 % with respect to these values (laser flash method and values, see [1]).



zero-crossing distance:

$$d = \frac{1}{\sqrt{f}} \cdot m + c$$

Figure 3. zero-crossing distance versus the reciprocal square root of the frequency for OBSiC

5. Conclusion

By means of photothermal beam deflection measurements of the thermal diffusivity of ceramic samples made of Alumina, Mullite and OBSiC were successful performed. The method used for obtaining the results from the experimental data was the zero-crossing method. The results show a deviation of 9 % with respect to laser flash experiments.

6. Acknowledgment

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