SIMPLE THERMAL WAVE METHOD FOR THE DETERMINATION OF LONGITUDINAL THERMAL DIFFUSIVITY OF SiC-BASED FIBER

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INTRODUCTION

Silicon carbide fibres have proven to be an excellent reinforcement material in ceramics and other composites due to their high strength and stiffness and good thermomechanical stability. The thermal diffusivity/conductivity of SiC based fibres has been reported earlier [1]. The methods used so far for the fibres have been mainly indirect and based on using a composite of known composition and then applying a standard thermal conductivity measurement on that system. Fiber properties have further been obtained from an inversion calculation. Direct measurement data of fibre properties has not been available.

In this paper a photothermal method, three dimensional photothermal radiometry (3D-PTR), [2] is proposed for the direct measurement of longitudinal thermal diffusivity of thin, fibre-type samples. This method belongs to the thermal-wave techniques that have been successfully used for determining thermal diffusivity even in complex geometry and materials [3, 4, 5].

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THEORY

The 3D-PTR method for measuring thermal diffusivity of fibres is based on applying local, periodic heating on the fibre and observing the shape of the AC-thermal distribution that is thus created. A thin fibre can be treated as a single-dimensional “heat guide”; therefore the governing thermal diffusion equation with harmonic heat source leads to simple solutions for the phase of the thermal wave along the fibre [6]. At a distance far enough from the heat source so that the heat flow is free from the disturbance caused by the finite source size, the phase of the thermal wave along the fibre is

\[
\phi(r) = -\frac{r}{\mu} \\
\mu = \left(\frac{2\alpha}{\omega}\right)^{1/2}
\]

where \( r \) is the distance from the heating, \( \alpha \) the thermal diffusivity, \( \mu \) the thermal diffusion length and \( \omega \) the angular frequency of the excitation. The measurement principle is shown in Fig. 1. The fibre and the movement of the focusing lens are in the direction perpendicular to the page. The heating spot is moved in steps while localised radiometric detection is used to probe the thermal wave at a single point on the fibre. The phase difference between the heating produced by the modulated laser beam and the observed temperature variation is measured using a lock-in amplifier. With the movement of the heating along the fibre the thermal wave phase vs. distance relationship in Eq. 1 will be thus measured.

MEASUREMENTS AND EQUIPMENT

Two types of fibres were investigated for room temperature thermal diffusivity along the fibre direction. The fibre types on the market come from two main process types, Chemical Vapour Deposition (CVD) or from the pyrolysis of polymer precursors producing either \( \beta \)-SiC or an oxygen containing fibre [7]. Two SiC single fibres of the first type, Texton SCS-6 and AVCO SCS-6 were investigated. These first two fibres have a 33 micron carbon core that plays an important role in the heat transfer properties of the material [6]. The other samples were of Nicalon SiC-based Si-O-C fibre that has a nanocrystalline structure and thus lower thermal conductivity. A highly pure platinum wire (99.99 %) was used as the reference material for the calibration of experimental equipment.

![Figure 1. Measurement system.](image-url)
In the measurement system the laser used was a 4 W Coherent Innova 90-4 operating at 518 nm, the detector an EG&G Judson (25 µm² active area) with a DC-coupled preamplifier and a 50 mm diameter, f = 50 mm Ge-lens in a 1:2 imaging geometry. The lock-in amplifier used for phase detection was a EG&G Princeton Applied Research 5210 and the laser was modulated using an acousto-optic modulator. The laser power at the sample was about 0.1 W at a beam 1/e radius of about 100 µm. One single scan is obtained in about 30 minutes under the control of a proprietary control programme.

Fig. 2 shows the phase profiles obtained at different modulation frequencies (10, 15 and 30 Hz) on a single Textron SCS-6 fibre suspended in air. As expected, the phase has an asymptotic linear behaviour when the spacing between heating and detection centres exceeds approximately a distance of 3R, R being the 1/e-gaussian-radius of the heating beam. From the slopes of the curves that were calculated in the 500-1500 µm offset range, the thermal diffusion length was obtained for each value of the modulation frequency (Table I). The obtained values thermal diffusivity were nearly the same, with a scattering of about 1% between the highest and the lowest values.

All the experimental data on different fibres are listed on Table II. The thermal conductivity was calculated by the well-known relation

\[ k = \rho c_p \alpha \]  

(3)

where the bulk density \( \rho \) was measured using Archimedes’ method and the constant pressure specific heat \( c_p \) was taken from the literature.

![Figure 2. Photothermal phase vs. heating and detection area centre offset measured on Textron SCS-6 fibre at different modulation frequencies (10, 15, and 30 Hz).]
The thermal diffusivity of the platinum wire, obtained using the 3D PTR method, is in agreement with the data reported in the literature for pure bulk platinum [8]. This value was obtained from the phase slope profile at a modulation frequency of 10 Hz that corresponds to a thermal wave diffusion length of about 890 μm. With regard to the nanocrystalline SiC-fibre (SiC Nicalon), measurements using both 3D-PTR and the standard laser-flash method [9] were performed. As the sample for the laser-flash needed to be in the shape of a cylinder, many of the fibre bundles were held together (diameter 5 mm) using heat shrink tubing.

<table>
<thead>
<tr>
<th>Material</th>
<th>Method</th>
<th>α (cm²/s)</th>
<th>ρ (g/cm³)</th>
<th>k (W/mK)</th>
<th>diameter (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Platinum wire</td>
<td>3D PTR</td>
<td>2.53</td>
<td>21.5</td>
<td>66.0</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>Literature 8</td>
<td>2.51</td>
<td>21.5</td>
<td>66.9</td>
<td>bulk</td>
</tr>
<tr>
<td>SiC Fibre (Nicalon™)</td>
<td>Laser-Flash</td>
<td>0.0107</td>
<td>2.55</td>
<td>1.82</td>
<td>15 (fibre)</td>
</tr>
<tr>
<td>SiC Fibre (Nicalon™)</td>
<td>Laser-Flash</td>
<td>0.0110</td>
<td>2.55</td>
<td>1.87</td>
<td>15 (fibre)</td>
</tr>
<tr>
<td>SiC Fibre (Nicalon™)</td>
<td>3D PTR</td>
<td>0.0104</td>
<td>2.55</td>
<td>1.77</td>
<td>15 (fibre)</td>
</tr>
<tr>
<td>SiC/C Fibre (Textron SCS-6)</td>
<td>3D PTR</td>
<td>0.085</td>
<td>3.15</td>
<td>17.8</td>
<td>140 (fibre)</td>
</tr>
<tr>
<td>SiC/C Fibre (AVCO SCS-6)</td>
<td>3D PTR</td>
<td>0.12</td>
<td>3.01</td>
<td>24.0</td>
<td>33 (core)</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>80 (fibre)</td>
</tr>
</tbody>
</table>

Table II. Experimental results

<table>
<thead>
<tr>
<th>f (Hz)</th>
<th>μ (μm)</th>
<th>α (cm²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>522</td>
<td>0.0855</td>
</tr>
<tr>
<td>15</td>
<td>425</td>
<td>0.0851</td>
</tr>
<tr>
<td>30</td>
<td>302</td>
<td>0.0860</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>α (cm²/s)</th>
<th>Δα (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0855</td>
<td>1</td>
</tr>
</tbody>
</table>

common to electronics use and cut dry to two length, and lengths 4.50 mm and 2.45 mm. By contrast, the measurement performed using 3D-PTR was carried out on a single fibre bundles suspended in air and containing about 250 fibres. The comparison of these results shows good agreement (~ 5%) between the two different methods. The thermal properties of SiC Nicalon fibres found in this work (0.0107 cm²/s, 1.8 W/mK) are somewhat higher than those obtained by Brennan et al. using an indirect method based on the application of a composite theory [10]. They found a longitudinal thermal diffusivity for Nicalon fibres in the range 0.00669-0.00899 cm²/s at 300 K, that corresponds to a thermal conductivity within 1.14-1.53 W/mK. Since heat transfer properties are structure-sensitive, especially for SiC-based materials for which a variation of two and a half orders of magnitude has been reported [10], this difference is not surprising and it could be explained in terms of slight differences in the material composition.
CONCLUSIONS

The proposed 3D-PTR method allows for an accurate determination of the longitudinal thermal diffusivity of fibre-type samples. Together with the use of a simple composite model for the two phase fibres, 3D-PTR results give new information on the thermal transport properties of SiC-fibres.

REFERENCES