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MEASUREMENT OF THERMAL DIFFUSIVITY OF REACTION BONDED SILICON NITRIDE

J.-J. SERRA, M. JAYMES and M. CANTAREL

E.T.C.A., 16 bis Avenue Prieur de la Côte d'Or, F-94114 Arcueil Cedex, France

Abstract-The thermal diffusivity of samples of reaction bonded silicon nitride from different sources, was measured using the ETCA solar furnace. Two measurement methods, suitable for use with this type of equipment, allowed the different material origins to be determined. This installation accepts relatively large samples which are therefore representative of the bulk material.

1 - INTRODUCTION

The thermal diffusivity of structural materials is an essential parameter in the definition of their thermomechanical properties. This parameter can be determined, at high temperature using various dynamic methods on generally thin samples.

The utilization of a 45 kW solar furnace, such as the ETCA furnace, allows relatively thick samples to be used which are more representative of the bulk material. In measurements at high temperature the modulated incident flux method is used and, around ambient temperature, a method is used based upon material response to a flux window. These methods were applied to the calculation of the thermal diffusivity of discs of reaction bonded silicon nitride.

2 - EXPERIMENTAL EQUIPMENT

2.1 Generation of thermal flux

The front of the samples is subject to a light flux from a compound solar furnace, a description of which has already been published /1/.

The furnace comprises three main elements:

- a heliostat: a plane reflector 17.4 x 13.2 m, the purpose of which is to reflect the sun's rays in the direction of the optical axis of a concave mirror.
- a concentrating mirror, focal length 10.75 m, the projection of which covers an area of 10 m x 10 m.

- an attenuator which acts as a servo-controlled flux modulator and varies the flux density reaching the concentrator focus between 0 and 500 W/cm². This attenuator comprises a system of vertical flaps placed on the parallel rays, between the heliostat and the concentrator. This configuration conserves the same distribution of energy in space, in the focal plane.

2.2 Mounting of samples

The samples, in the form of discs, are held at three points by a concentric grip clip with ceramic pads. The assembly is placed behind a water-cooled diaphragm of the same diameter as the sample, which protects surrounding experimental equipment.

2.3 Temperature measurement

The high temperatures on both sides of the samples are measured by optical pyrometry (Barnes 12.550) in the infra-red area, above 4 μm. This avoids errors due to random reflections of the sun's rays, as the radiation spectrum leaving the overall optical system is limited to wavelengths less than 2.8 μm. Around ambient temperature, only the temperature on the rear face of the samples needs to be measured. It is measured by a thermocouple. Signals from the temperature sensors are recorded by an automatic data collection system (HP 30528) and transferred to a computer (HP 9826A) for processing.

3 - PROCESSING OF RESULTS

3.1 Principle of methods used

The methods used to interpret the thermograms for the calculation of the diffusivity are based upon the model of the plane wall of finite thickness (cf. Fig. 1). The sample of thickness e receives, on one side \((x = e)\), a thermal flux, the time variation of which is known. Consideration is then given to the temperature variation in relation to the temperature existing at the beginning of the flux pulse: \( \Theta (x, t) = T(x, t) - T(x, 0) \). This flux law may be either applied to a wall which was not penetrated by any flux, or superimposed on a wall already penetrated by a constant flux. The heat exchange of the two sides will be defined by local Biot numbers \( \beta_0 \) and \( \beta_e \) (\( \beta x = e h x / \lambda \), where \( \lambda \) is the thermal conductivity and \( h x \) the heat loss parameter at \( x = 0 \) and \( x = e \)).

![Figure 1: Schematic drawing of the sample arrangement](image)
3.2 Flux sinusoidal wave modulation /2/

At high temperature, the wall is subjected to a sinusoidal wave variation in flux, pulsing $\omega$. This then results in a stabilized temperature cycle situation on each side. The sinusoidal wave signal on the unexposed side ($x = 0$) is out of phase in relation to that of the exposed side ($x = e$) by an amount $\Delta$ and its amplitude is attenuated in a ratio $P$ (figure 2). These two values are function of the two intermediate variables $\beta_0$ and $b$, where $b = e\sqrt{\omega/2a}$, and $a$ is the thermal diffusivity /3/. Thus, by measuring $\Delta$ and $P$, it is possible to calculate $b$ and $a$ (as $\omega$ and $e$ are known) /4/.

3.3 Flux window /5/

In the medium temperature range, a thermal pulse will be used, in a flux window, duration $\tau$, such that the term $a\tau/e^2$ will be greater than 0.5. At the end of the window, during cooling, loss coefficients will be assumed to be the same on both sides. Under these conditions, material temperature during cooling tends towards a negative exponential function in time. The semi-logarithmic representation of function $N = \theta(0, t)/\theta(0, \tau)$ accepts an asymptote (figure 3). In this case, measuring the parameters of this straight line will give the Biot number and diffusivity values.

![Figure 2: Measurement principle for sinusoidal wave flux method](image1.png)

![Figure 3: Measurement principle for flux window method](image2.png)

4 - EXPERIMENTAL RESULTS

Samples of reaction bonded silicon nitride from three different sources were studied. The samples were in the form of discs, 50 mm diameter and 5 mm thick. Their physical and mechanical characteristics are shown in Table 1.

Results concerning variations of thermal diffusivity with temperature are shown in figure 4. It will be seen that the values obtained by the two methods agree quite well (the critical point between the two methods occurring around 300° C).

The results show a considerable difference between the diffusivities of the three materials, especially at low temperatures. Values obtained for $A$ and $B$ become the
same at high temperatures. The physical and mechanical properties shown in Table 1 give no explanation for this difference in behaviour. Conversely, there are considerable differences in sample composition, determined using the method described in Ref. 17. Depending upon the sample concerned, this method showed two or three phases (α and β Si₃N₄ and Si₃ON₂). Materials A contained 21% β Si₃N₄, the remainder comprising α Si₃N₄ and a small quantity of Si₃ON₂. Samples B contained 91% β Si₃N₄, with the remaining 9% formed by α Si₃N₄ and Si₃ON₂, in equal proportions. Concerning the C samples, these contained 33% β Si₃N₄ and 67% α Si₃N₄, without any apparent silicon oxynitride. The differences observed in thermal diffusivity would therefore appear to be the result of differences in composition.

Table 1: Main characteristics of samples tested.

* Manufacturer's data  
** Parameters measured by ourselves (method described in Ref. 6).

<table>
<thead>
<tr>
<th>Apparent density (g/cm³)</th>
<th>Open porosity (%)*</th>
<th>Poisson's coefficient (***)</th>
<th>Young's modulus (GPa) ***</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2.45</td>
<td>25</td>
<td>0.235</td>
</tr>
<tr>
<td>B</td>
<td>2.35</td>
<td>17 - 20</td>
<td>0.225</td>
</tr>
<tr>
<td>C</td>
<td>2.55</td>
<td>15 - 25</td>
<td>0.235</td>
</tr>
</tbody>
</table>

Figure 4: Comparison between diffusivity results obtained for the three types of material.
5 - CONCLUSION

Due to its thermal flux modulation system, the ETCA solar furnace is well suited for the determination of diffusivity. Two measuring methods were developed to study variations of this parameter, from ambient temperature up to high temperatures. Installation dimensions allow relatively large samples to be tested (up to more than 10 mm thick). Results obtained for reaction bonded silicon nitride clearly showed the difference between three materials from different sources. These results thus demonstrate the installation's capabilities in the investigation of material characteristics at high temperature.

REFERENCES

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