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A Method of Measurement of the Multilayer Woven Ceramic Matrix Composite Panel In-plane Thermal Diffusivity

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Abstract

A method of determination of the in-plane thermal diffusivity of multilayer woven ceramic matrix composite (CMC) panels was developed at CEAT. The laser flash method was used on a cylindrical laminate specimen made of CMC and a ceramic cement. The thermal diffusivity of the CMC is derived by applying a correction to the value obtained with the laminate specimen in order to account for the effect of the cement. This method was first validated on POCO graphite and then applied to a multilayer woven SiC/SiC material between 20 and 1200°C. © 1997 Elsevier Science Limited.

1 Introduction

High temperature metallic materials presently used in the hottest parts of military fighter engines have reached their temperature limit and ceramic matrix composites (CMCs) are potential candidates for low-stress parts in severe environments (afterburner duct, nozzle flaps ...). Amongst these composites, SiC/SiC (silicon carbide continuous fibres in a silicon carbide matrix) is a promising material. Its thermal diffusivity is a paramount parameter which must be taken into account by the research department for parts design. Indeed, the thermal stresses that build in high temperature gradient parts depends on the thermal diffusivity of their constituents.

The thermal diffusivity of opaque materials is generally measured by the laser flash method.[1,2] In this method, the face of a thin cylindrical specimen is subjected to an instantaneous laser flash and the temperature variation of the opposite face of the specimen is recorded (thermogram); the thermal diffusivity of the material in the direction of the laser beam is computed from this temperature history.[1] Experimentally, the thermal diffusivity of specimens reaches a constant limit value, called limit diffusivity, with increasing thicknesses.[3,4]

The use of this method for composite materials implies that it is considered as thermally homogeneous. Also, the surface area of the laser spot should be greater than the unit mesh of the material in order to get a value representative of the whole composite [see Fig. 1(b)]. SiC/SiC material is often orthotropic; thus, its thermal diffusivity must be measured in the three principal directions [x, y and z in Fig. 1 (a)]. A few authors[5–7] have used the laser flash method on a thin SiC/SiC plate in order to measure the in-plane thermal diffusivity [Fig. 1(c)]. However, this method may not be adapted to the latest generation of thin multilayer woven CMCs with a unit mesh of more than 1 cm^2. To overcome this point, a few authors[8–10] have proposed the derivation of the radial, in-plane thermal diffusivity of material from the *heterogeneous* temperature history of a circular specimen subjected to an out-of-plane laser flash.

This paper introduces a new method of CMC in-plane thermal diffusivity assessment between 20 and 1200°C developed at CEAT.

2 Experimental Procedure and Method

2.1 Experimental procedure

In this study, the thermal diffusivity was measured in a vacuum by the laser flash method on 12 mm dia. specimens using a PC-computer monitored SOPRA 2000 equipment. The laser flash had a wavelength of 1054 μm, a pulse time of 300 μs and an energy of up to 60 J. Two infrared detectors have been used to measure the thermal history of

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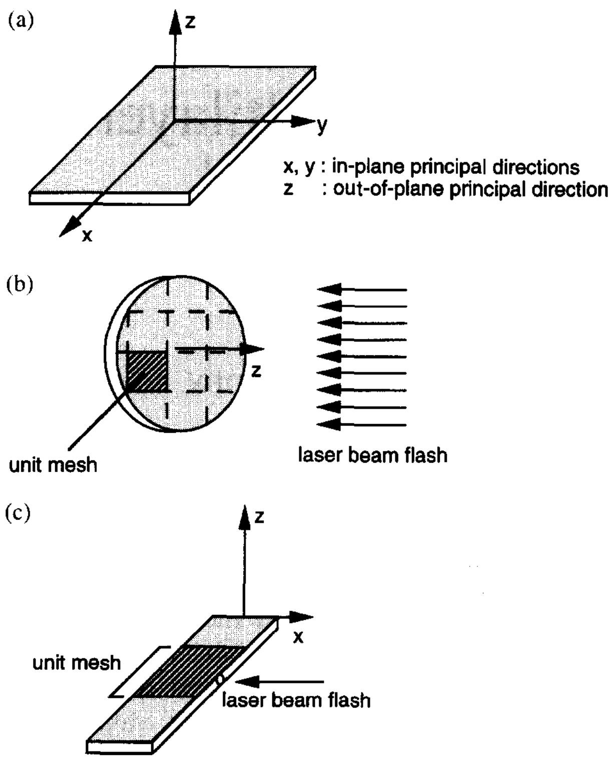

Fig. 1. Thermal diffusivity measurement by the laser flash method. For the in-plane measurement, the laser-exposed area is smaller than the unit mesh.

the face of the specimens: HgCdTe from 20 to 600°C and Ge up to 1200°C.

The material studied here was a 9-ply, multilayer woven CVI SiC/SiC composite fabricated by SEP (Société Européenne de Propulsion) using Nicalon NLM 202 fibres. Its unit mesh size was 1·5 cm× 0·5 cm×thickness. The panel was about 3·2 ± 0·1 mm thick and its density was 2·47 ± 0·01 g cm^{-3}.

Laminate specimens were prepared as follows (see Fig. 2):

(a) In order to suppress surface defects and assure a good thermal coupling the rough panels were diamond ground to a thickness of 2·6 ± 0·1 mm.
(b) Small pieces with dimensions 12 mm×length ×thickness were cut in the panels with the length parallel to the weft direction.
(c) The pieces were stacked and cemented using Cotronics alumina 903 HP cement cured for 2 h at 120°C and 4 h at 370°C.
(d) The laminate was diamond machined to obtain a 12 mm dia cylinder which was cut to slices with a thickness of 2, 4 or 6 mm, using a diamond wafering blade.

Figure 3 shows a picture of a SiC/SiC laminate specimen.

Cylindrical specimens of isotropic Unocal-Poco AXM 5Q1 POCO graphite were machined to serve as standard specimens. The comparison between the thermal diffusivity of POCO and laminate POCO specimens permits to evaluate the difference due to the cement joints, to verify that the thermograms obtained for laminate specimens are exploitable and to derive a correction to apply to a laminate specimen in order to calculate the diffusivity of its main component. Alumina cement cylindrical specimens were also fabricated to measure their thermal diffusivity. Table 1 lists the types and thicknesses of specimens which were tested.

2.2 Method

From the thermal diffusivity of POCO, the cement and POCO laminate specimens, we derive a correction which is applied to a SiC/SiC composite. The correction sought on diffusivity is actually established from the thermal conductivity of the components. The thermal conductivity λ and diffusivity a are related by eqn (1) where ρ is the density and c is the specific heat capacity:

$$\lambda = a\rho c \qquad (1)$$

In the direction of fibres the conductivity of the composite λ_3 can be derived if one assumes that the rule of mixture applies:

$$\lambda_3 = \lambda_1 \cdot V_1 + \lambda_2 \cdot V_2 \qquad (2)$$

where 1, 2 and 3 stands respectively for the main phase (POCO or SiC/SiC in our case), the cement and the laminate specimen and V is the volume fraction. The assessment of cement volume fraction ($V_2 = 3\%$) must be precise. It was derived from the cement surface area fraction which was measured on the specimens with an optical microscope. The rule of mixture also applies to the heat capacity per unit volume of the laminate:

$$\rho_3 \cdot c_3 = \rho_1 \cdot c_1 \cdot V_1 + \rho_2 \cdot c_2 \cdot V_2. \qquad (3)$$

Equation (1) is used to determine the thermal conductivity of the main phase λ_1:

$$\lambda_1 = a_1\rho_1 c_1 \qquad (4)$$

Equations (1) and (3) are used to derive the thermal conductivity of the laminate specimen λ_3:

$$\lambda_3 = a_3(\rho_1 \cdot c_1 \cdot V_1 + \rho_2 \cdot c_2 \cdot V_2) \qquad (5)$$

Finally, eqns (1)–(3) are used to derive a value for the conductivity of the main phase, λ_1', called corrected conductivity:

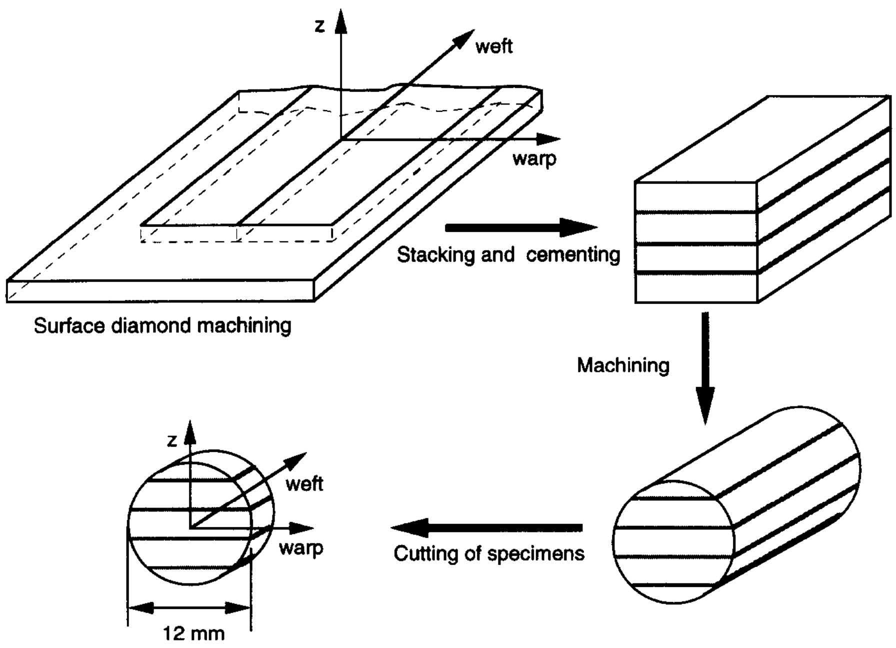

Fig. 2. Fabrication procedure of laminate specimens.

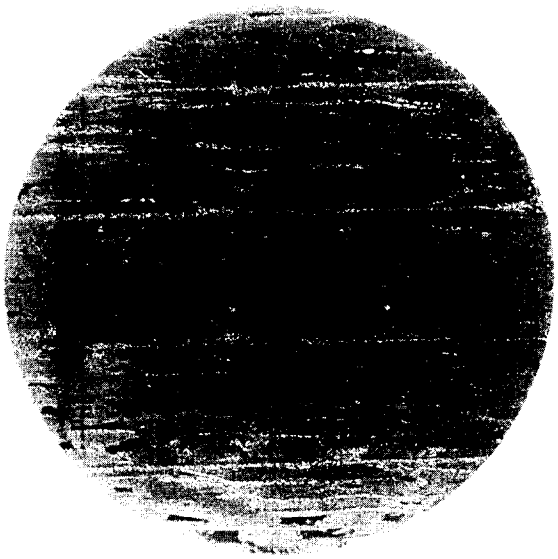

Fig. 3. SiC/SiC laminate specimen. Note the alumina cement joints.

$$\lambda_1' = \frac{\lambda_3 - \lambda_2 \cdot V_2}{V_1} = \frac{a_3 \rho_3 c_3 - \lambda_2 \cdot V_2}{V_1} \tag{6}$$

from which a corrected diffusivity a_1' is derived:

$$a_1' = \frac{\lambda_1'}{\rho_1 c_1} \tag{7}$$

3 Results and Discussion

3.1 POCO laminates

The thermal diffusivity of alumina as a function of temperature is summarised in Table 2.

Table 1. Types of tested specimens

Materials	*Specimen thickness (mm)*		
Poco AXM 5Q1	2	4	6
Alumina cement-POCO laminate	2	4	6
SiC/SiC direction 3	approx. 2·7		
Alumina cement-SiC/SiC laminate in-plane direction	2	4	6
Alumina cement	approx. 2·5		

Table 2. Average thermal diffusivity of alumina cement

	Alumina cement	
Temperature (°C)	a_{ave} *(cm² s⁻¹)*	*CV (%)*
20	0·0237	21·6
290	0·0118	14·0
477	0·0102	13·0
655	0·0089	12·7
837	0·0082	10·5
1019	0·0076	9·5
1206	0·0073	12·7

Table 3 summarises the average thermal diffusivity of POCO graphite specimens of all thicknesses and POCO laminates with a thickness of 2, 4 and 6 mm. The values for monolithic POCO are similar to those found in refs 11 and 12 ; the small coefficient of variation indicates a good homogeneity of the values despite the use of three different thicknesses of specimens.

The thermograms of POCO laminates have shown that the determination of thermal diffusivity

Table 3. Average values of experimental thermal diffusivity of POCO and POCO laminates. For monolithic POCO graphite, the CV was calculated using the 6 measurement values (2 for each thickness)

	monolithic POCO (6 measurements)		*6 mm laminate (6 measurements)*		*4 mm laminate (3 measurements)*		*2 mm laminate (6 measurements)*	
Temperature (°C)	a_{ave} (cm^2s^{-1})	*CV (%)*	a_{ave} (cm^2s^{-1})	*CV (%)*	a_{ave} (cm^2s^{-1})	*CV (%)*	a_{ave} (cm^2s^{-1})	*CV (%)*
20	0·698	0·6	0·658	0·9	0·660	1·6	0·671	0·9
290	0·302	1·2	0·292	1·4	0·293	0·4	0·289	0·7
477	0·230	1·8	0·221	1·0	0·225	2·1	0·223	0·9
655	0·191	1·3	0·186	0·5	0·187	0·8	0·188	0·6
837	0·165	0·7	0·161	0·6	0·160	0·4	0·162	0·6
1019	0·145	1·2	0·142	0·0	0·141	0·0	0·144	0·3
1206	0·133	0·9	0·128	1·2	—	—	0·129	0·6

Table 4. Thermal conductivity of POCO and POCO laminates. λ_1 is derived from eqn (4), λ_3 from eqn (5) and λ'_1 from eqn (6)

	Monolithic POCO	*Laminates*					
		6 mm		*4 mm*		*2 mm*	
Temperature (°C)	λ_1 ($W\,m^{-1}\,K^{-1}$)	λ_3 ($W\,m^{-1}\,K^{-1}$)	λ'_1 ($W\,m^{-1}\,K^{-1}$)	λ_3 ($W\,m^{-1}\,K^{-1}$)	λ'_1 ($W\,m^{-1}\,K^{-1}$)	λ_3 ($W\,m^{-1}\,K^{-1}$)	λ'_1 ($W\,m^{-1}\,K^{-1}$)
20	84·3	81·0	83·4	80·6	83·0	81·3	83·6
290	69·2	67·4	69·4	67·1	69·0	65·6	67·5
477	63·9	61·6	63·4	62·2	64·0	60·9	62·7
655	58·3	56·7	58·4	56·8	58·5	56·6	58·3
837	53·0	51·7	53·2	51·3	52·8	51·3	52·8
1019	48·8	47·9	49·3	47·2	48·6	47·8	49·2

Table 5. Average thermal conductivity difference between POCO and POCO laminates

	6 mm laminate		*4 mm laminate*		*2 mm laminate*	
	$(\lambda_3-\lambda_1)/\lambda_1$	$(\lambda_3-\lambda'_1)/\lambda_1$	$(\lambda_3-\lambda_1)/\lambda_1$	$(\lambda_3-\lambda'_1)/\lambda_1$	$(\lambda_3-\lambda_1)/\lambda_1$	$(\lambda_3-\lambda'_1)/\lambda_1$
Average difference (%)	−2·8	0·0	−3·2	−0·3	−3·6	−0·8

by the laser flash method is appropriate even for the 2 mm thick specimens. From Table 3, it can be deduced that cement joints induce a decrease of 2–6% of the thermal diffusivity of POCO, with a greater effect at a low temperature. Moreover, the values are almost identical for all thicknesses.

To calculate λ_1, λ_3, and λ'_1 from eqns (1)–(6) for POCO + alumina cement laminates, ρ_1, ρ_2, ρ_3, a_1 and a_3 have been experimentally measured whereas c_1 was taken from ref. 13 and c_2 is the specific heat capacity of sapphire. Table 4 summarises the values of λ_1, λ_3 and λ'_1 for the three specimen thicknesses and Table 5 shows the average values of the relative difference of λ_3 and λ'_1 compared to λ_1. It can be seen that the correction on the conductivity is very efficient for 6 and 4 mm specimens as compared to that of the monolithic POCO specimens since the difference is less than 0·5%. The larger difference for the 2 mm thick specimens is probably due to an increase of relative uncertainty on the time and thickness measurements.

3.2 Application to SiC/SiC laminates

The thermal diffusivity a_3 of 2, 4 and 6 mm thick SiC/SiC laminates was measured. The thermograms for the 2 mm thick specimens could not be used since the laser flash would sometimes cross the laminate due to its high porosity. Equation (4) shows the average value of measurements made on all 4 and 6 mm thick specimens as they were very similar. Figure 4 also shows the corrected average

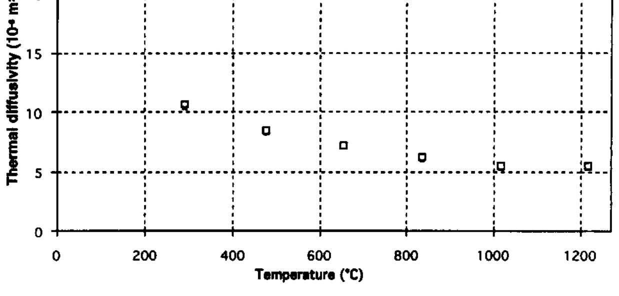

Fig. 4. Thermal diffusivity of SiC/SiC and SiC/SiC laminates.

diffusivity a'_1 of 4 and 6 mm thick specimens as defined in eqns (6) and (7). The correction to apply to a_3 due to the cement joints is between 2·7% and 3·4%. This correction is all the smaller that the thickness of the cement joints (or V_2) is minimal and their diffusivity a_2 is close to that of the main component (a_1).

4 Conclusion

The measurements made in this study on laminate specimens aimed at investigating a method of determination of the in-plane thermal diffusivity of multilayer woven CMC thin panels. The results obtained on laminate POCO or laminate SiC/SiC specimens using alumina cement joints showed that:

—the specimens can be considered thermally homogeneous since the quality of the recorded thermograms and the stability of the diffusivity for different thicknesses were good, and

—a correction which depends on the cement volume fraction and thermal characteristics needs to be applied to the laminate thermal diffusivity in order to derive the main component's diffusivity.

Acknowledgement

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