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Thermal-diffusivity measurement of 3D-stitched C–SiC composites

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Abstract

Effect of siliconization conditions on thermal diffusivity of 3D-stitched fiber architecture-based C–SiC composites was investigated to select desired conditions. Several 3D-stitched C–SiC composite blocks were prepared using coal–tar pitch as a carbon precursor and siliconization was carried out at 1450 and 1650 °C for 10 and 120 min. Thermal diffusivity of blocks was investigated using laser-flash equipment in in-plane and through-thickness directions. It varies from 77 mm^2/s at room-temperature to 14.7 mm^2/s at 1500 °C in in-plane and 36–6.1 mm^2/s in through-thickness direction. A model was developed to estimate thermal diffusivity in in-plane and through-thickness directions based on the volume fraction of the constituents and porosity of the composite blocks. The estimated thermal-diffusivity values were compared with the measured values. The values were found to be close to the experimental values in entire testing temperature range at all the siliconization conditions.

Keywords: Composites; Thermal properties; Thermal conductivity; SiC; 3D-stitched C–SiC

1. Introduction

Liquid-silicon infiltration (LSI) was developed by German Aerospace Centre (DLR) as a fast and economic method of processing bi-directional (2D) carbon–silicon–carbide (C–SiC) composites[1]. Main applications of C–SiC composites are nose tips of reusable space vehicles, leading edges of hypersonic vehicles, jet-vanes and brake discs for high speed automobiles[2,3] which require uniform thermal properties in all the directions. Thermal diffusivity in the entire temperature range of operation is an important design input. Many researchers have published thermal-diffusivity data for LSI based 2D C–SiC composites.[4,5] To achieve better thermal properties in all the three directions 3D-stitched C–SiC composites are proposed for jet-vanes applications. Coal–tar pitch has been proposed to use as a carbon precursor to fabricate 3D C–SiC composites. In porous solids thermal conductivity is dependent on the porosity, the pore size, crystal size and degree of molecular orientation in case of amorphous solids.[6] For C–SiC composite it is sensitive to its constituents (carbon–fibers, carbon–matrix, silicon–carbide (SiC) and silicon) composition, microstructure which get developed during the process and also depends upon the direction in which fibers are oriented. The composition and microstructure of these composites depends upon process parameters like siliconization time, temperature and geometrical parameters.[7,8] Thermal-diffusivity data for 3D-stitched C–SiC composites is not available. Many researchers have proposed models for predicting thermal diffusivity for ceramic composites which cannot be used for 3D-stitched C–SiC composites due to different fiber architecture. Temperature of the silicon infiltration in carbon performs is reported from 1420–1600 °C[3–5,7,8,11] while time of silicon infiltration has been reported from few seconds to 2 h[5,7,8] depending on the size of the job. To select an optimum siliconization conditions for jet-vane size C–SiC composites a systematic study on the effect of siliconization conditions on thermal-diffusivity has been carried out. It was decided to carry out the following:

(1) Preparation of 3D-stitched C–SiC composite blocks at different siliconization conditions by LSI route.
(2) Thermal-diffusivity measurement in in-plane and through-thickness directions from room-temperature to 1500 °C at each siliconization condition.
(3) Empirical correlation of thermal-diffusivity data.

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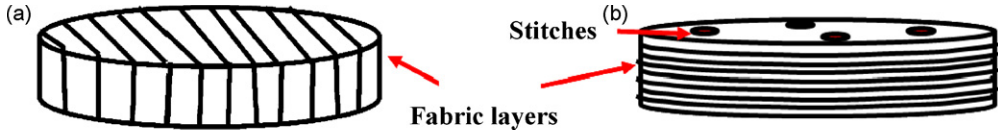

Fig. 1. Thermal diffusivity schematic: (a) in-plane parallel to fabric and (b) through-thickness.

2. Experimental

The 3D-stitched fibrous preforms were prepared by stitching several layers of the 8H satin high strength (PAN based) carbon–fabric layers with 6k (PAN based) carbon–fiber tows. The preforms were rigidized by vacuum infiltration of coal–tar pitch at 200–300 °C, followed by carbonization at 900–1000 °C and graphitization at 2400–2600 °C in inert atmosphere. The rigidized preforms were densified by impregnation of coal–tar pitch followed by hot-iso-static-pressure-impregnation–carbonization (HIPIC), to obtain C–C preform of density 1.55–1.6 g/cm^3. These C–C preform blocks were cut into 12 different blocks of sizes (75 mm × 50 mm × 20 mm to 150 mm × 50 mm × 50 mm) and termed as b-1 to b-12. b-1, b-2 and b-3 were siliconized at 1450 °C for 10 min; b-4, b-5 and b-6 at 1450 °C for 120 min; b-7, b-8 and b-9 at 1650 °C for 10 min; b-10, b-11 and b-12 at 1650 °C for 120 min to obtain 3D-C–SiC composite blocks.

C–SiC composite blocks contain C–C (carbon fibers and un-reacted carbon matrix), SiC and residual silicon. Weight fractions of these constituents were determined by acid digestion method[11] where a part of each siliconized block was crushed and converted into powder. The C–SiC composite powder (about 5 g) was digested into acid (HF:HNO$_3$, 4:1) mixture of about 50 ml for 24 h. Residual silicon in the powder dissolves into acid while the amount of carbon and SiC powder was obtained by filtering the acid. Amount of SiC powder was obtained by heating the carbon and SiC powder in air at about 600 °C. The composition by weight was subsequently expressed in terms of volume fraction of each constituent. To convert weight fractions into volume fraction densities for constituents are taken as: C–C, 2.2 g/cm^3; silicon, 2.25 g/cm^3; SiC, 3.2 g/cm.[3,12] Open porosity of C–SiC blocks was measured by Mercury porosity-meter (Quantachrome instrument).

2.1. *Thermal-diffusivity determination*

Cylindrical specimens of diameter 12.5 mm and thickness (L) 2.5 mm were cut from each of the C–SiC composite block in in-plane and transverse directions (Fig. 1). Thermal-diffusivity (α) was determined from room-temperature to 1500 °C using Laser-flash apparatus LFA 427 (NETZSCH Company) as per ASTM E-1461-92 in argon atmosphere at pre-decided temperatures. In this method the specimen is subjected to a short duration thermal pulse. The energy of the pulse absorbed at front face resulting in rear face temperature rise is measured. Thermal diffusivity is calculated from the specimen thickness and the time ($t_{1/2}$) required for the rear face temperature to reach one half of its maximum value. For each specimen thermal diffusivity was measured ($\alpha = 0.1388L^2/t_{1/2}$) thrice at a pre-decided temperature. Arithmetic mean average of three measurements is reported as thermal-diffusivity of each specimen for a given temperature.

3. Results and discussion

3.1. *Composition of C–SiC composite blocks*

The density and composition of the C–SiC composites blocks are given in Table 1. Density was found to be the highest for the blocks siliconized at 1450 °C for 120 min and the least for the blocks siliconized at 1650 °C for 120 min. Composition of the fabricated C–SiC composites blocks was determined by acid digestion. SiC fraction is found to be the highest for the blocks siliconized at 1650 °C for 120 min (b-10, b-11 and b-12) and the least for the blocks siliconized at 1450 °C for 10 min (b-1,

Table 1
Density and composition of C–SiC composite blocks

S. no.	Measured					Calculated			
	Density of C–SiC	Open porosity	Carbon (%, w/w)	SiC (%, w/w)	Silicon (%, w/w)	Carbon (vol%)	SiC (vol%)	Silicon (vol%)	Total porosity
b-1	2.19	4.86	0.568	0.335	0.097	56.62	22.96	9.45	10.97
b-2	2.19	4.81	0.592	0.335	0.073	58.93	22.93	7.11	11.04
b-3	2.19	4.89	0.574	0.335	0.091	57.24	22.97	8.87	10.91
b-4	2.33	3.71	0.538	0.359	0.102	56.98	26.14	10.60	6.32
b-5	2.30	3.68	0.530	0.359	0.111	55.41	25.80	11.30	7.44
b-6	2.34	3.64	0.532	0.359	0.109	56.59	26.25	11.30	5.83
b-7	2.23	5.31	0.582	0.348	0.069	58.99	24.25	6.84	9.92
b-8	2.17	5.29	0.599	0.348	0.053	58.97	23.56	5.10	12.37
b-9	2.20	5.27	0.524	0.348	0.127	52.4	23.93	12.40	11.26
b-10	2.19	5.42	0.563	0.384	0.053	56.17	26.34	5.17	12.32
b-11	2.20	5.46	0.559	0.384	0.057	55.9	26.40	5.57	12.13
b-12	2.26	5.49	0.563	0.384	0.080	57.76	27.08	8.02	7.13

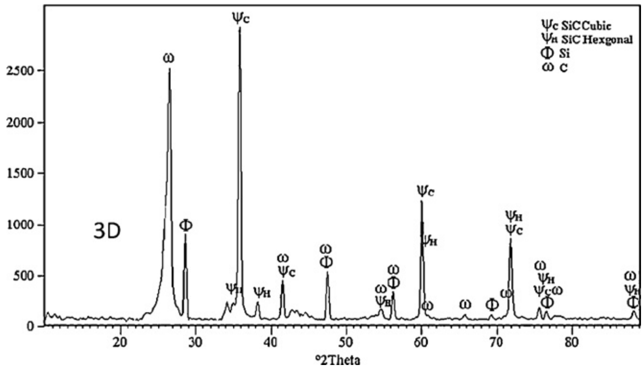

Fig. 2. XRD pattern of C–SiC composite block-1.

b-2 and b-3). Silicon found to be the highest for the blocks siliconized at 1450 °C for 120 min and the least for b-10, b-11 and b-12. At 1450 °C, silicon fraction in composite blocks increases with time while at 1650 °C it decreases. Composite blocks were further analyzed by XRD to determine crystalline phases present and microstructure was analyzed by optical microscope. A typically XRD pattern of block 1 is shown in Fig. 2 and an image of optical micrograph is shown in Fig. 3. XRD pattern confirms the presence of three phases, viz. SiC, carbon and silicon. It could also be seen from Fig. 3 that carbon fibers/matrix, SiC and residual silicon phases are uniformly distributed. Porosity was found to be the least at 1450 °C and highest at 1650 °C for infiltration time of 120 min. Porosity and composition data was used to interpret and estimate the thermal diffusivity.

3.2. Thermal-diffusivity investigations

Average values of thermal diffusivities at each siliconization condition in both the testing direction (in-plane (||) and through-thickness (T-T)) are presented in Fig. 4. Average thermal diffusivity at a specific siliconization condition is obtained by taking arithmetic mean average of thermal diffusivities of three specimens cut from different C–SiC blocks of same siliconization conditions. It decreases with increase in testing temperature in both the directions at all the conditions. The average values are two-three times higher in in-plane than the values in through-thickness direction in the entire temperature range. Above 727 °C the effect of siliconization conditions is negligible in both the testing directions. Thermal diffusivity trend is explained in the following lines.

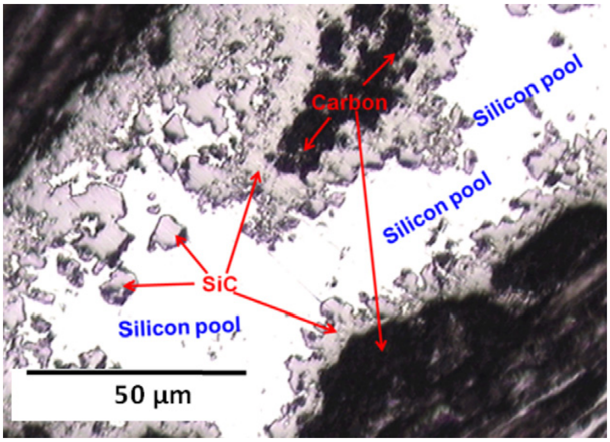

Fig. 3. Optical micrograph of 3D-stitched C–SiC composite block.

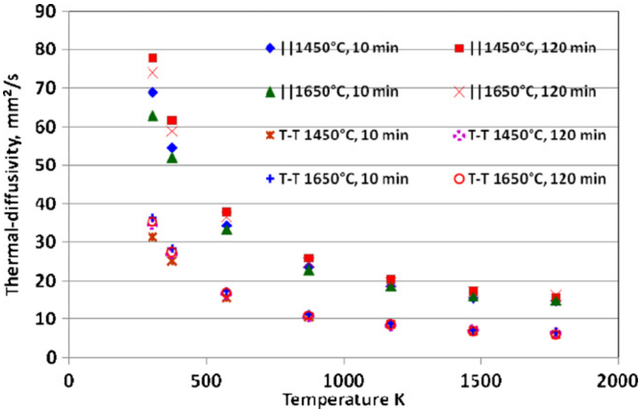

Fig. 4. Effect of testing temperature on transverse thermal-diffusivity.

Thermal-diffusivity of the constituents of C–SiC composites is reported to be decreased with temperature.[3] For fabric based 2D C–C composites it varies from 108–22 mm^2/s in direction in-plane and 17–3.8 mm^2/s in through-thickness direction; the highest value is at 25 °C and the lowest at 1500 °C.[13] For 6H SiC single crystal it varies from 152–9.3 mm^2/s in the temperature range of −243 to 1500 °C.[14] For silicon it varies in the range of 78–11.8 mm^2/s in temperature range of 27–1127 °C.[15]

In case of the in-plane thermal-diffusivity measurement heat flows in direction parallel to the warp while perpendicular to the weft and the third direction carbon fibers. In-plane thermal-diffusivity of C–C composites is higher than that in the transverse direction and the other constituents of the C–SiC composites. Therefore resistance to heat flow would be the least along the axis of fibers. However resistances (due to fibers, due to matrix) are in parallel. It is expected that the heat preferentially passes through material which offers least resistance. Hence it is the in-plane thermal diffusivity of the carbon–fibers which perhaps controls the overall in-plane thermal-diffusivity of the specimen.

In case of the through-thickness direction measurement the resistances to heat flow are in series where heat has to pass through the thickness of the fabric and matrix. Hence it is the cumulative effect of the through-thickness thermal diffusivity of the carbon fabric, along the axis of third direction carbon fiber tows and combined thermal diffusivity of SiC, residual silicon and un-reacted carbon matrix. The constituent having least thermal diffusivity would control its overall magnitude. As mentioned through-thickness thermal diffusivity of 2D C–C composites is much less than that of any of the other constituents of the matrix. Through-thickness direction thermal diffusivity would be in between the values of 2D C–C composite and SiC/silicon. Thermal diffusivity of C–SiC composite and its constituents decrease with temperature. Thermal diffusivity of solids depends on the lattice vibration and microstructure (micro-cracks, grain boundary, porosity, etc.), its dependence on temperature can be closely approximated by the following rela-

Table 2
Slope and intercept of plots a^{-1} versus T

S. no.	Siliconization conditions	Testing direction	A ($\times 10^{-5}$ s K/mm^2)	B ($\times 10^{-3}$ s/mm^2)	R^2
1	1450 °C, 10 min	||	4.327	2.900	0.994
2	1450 °C, 120 min	||	4.063	5.397	0.986
3	1650 °C, 10 min	||	3.838	2.877	0.992
4	1650 °C, 120 min	||	4.000	3.179	0.987
5	2D C–C composite	||	3.243	0.997	0.987
6	1450 °C, 10 min	T-T	9.300	7.467	0.992
8	1450 °C, 120 min	T-T	9.715	2.839	0.992
7	1650 °C, 10 min	T-T	9.573	0.763	0.997
9	1650 °C, 120 min	T-T	9.725	1.432	0.996
10	2D C–C composite	T-T	14.370	1.000	0.988
11	Silicon	Iso-tropic	9.176	−16.990	0.991
12	SiC	Iso-tropic	6.849	−14.180	1.0

tionship in terms of lattice characteristics and microstructure.[16]

$$\frac{1}{\alpha} = AT + B \qquad (1)$$

where α is the thermal diffusivity and A and B are constants. The magnitude of A is solely determined by the intrinsic lattice characteristics, while magnitude of B depends on the impurities and the microstructure in addition to the intrinsic lattice characteristics.

Eq. (1) is valid for SiC above 105 °C. Since the behavior of the C–SiC composites is similar to SiC, thermal diffusivity of C–SiC composites blocks can also be analyzed with the same equation. In order to apply the above theory, the average values of measured data were plotted (a^{-1} versus T) and fitted with a straight line. Values of constants A (slope) and B (intercept) were determined along with regression coefficient 'R^2' for C–SiC composite blocks at each siliconization condition and for both the measurement directions. For comparison, thermal-diffusivity data of silicon, SiC and 2D C–C composite were also treated in similar way and the values of constants were determined. The values of constants are given in Table 2.

Magnitude of A indicates the rate at which thermal-diffusivity decreases with temperature. The values of these are in very narrow band (3.838×10^{-5} to 4.3×10^{-5} s K/mm^2) and (9.3×10^{-5} to 9.7×10^{-5} s K/mm^2) for in-plane and through-thickness directions, respectively (Table 2). In direction in-plane, it is the fibers which control the thermal diffusivity, since fiber volume fraction is similar in all the C–SiC blocks therefore magnitude of A is in very narrow band (3.8×10^{-5} to 4.3×10^{-5}). In the direction through-thickness thermal diffusivity of carbon fabric in thickness direction and matrix constituents controls the thermal diffusivity of the C–SiC composites blocks. The matrix composition is similar in the four C–SiC blocks, therefore magnitude of A are in very narrow band (9.3×10^{-5} to 9.7×10^{-5}). However, values of A for 2D C–C composites in in-plane and through-thickness direction are 3.243×10^{-5} and 14.37×10^{-5} are different but nearer to the C–SiC composites. Magnitude of A of silicon is very close to the slope of C–SiC composite blocks of through-thickness direction. Magnitude of A of SiC is higher than that of the value of C–SiC composite blocks in in-plane directions but lower than the through-thickness directions. Though on the basis of magnitude of A effect of siliconization conditions cannot be generalized but these values indicate that the higher SiC fraction and lower porosity in the block, higher is the magnitude of A. As it is evident that slopes in direction through-thickness are twice to that of in-plane direction slopes this is due the different natures of reinforcement and matrix.

Values of B indicate the impurity and microstructure of the individual C–SiC block. Values of B are different in different testing directions and vary with siliconization conditions. This gives clear indication that thermal diffusivity varies with microstructure which is bound to be affected with siliconization conditions. Variation of thermal diffusivity with microstructure has been reported by other researchers also.[17] Change in microstructure is not expected due to testing, as testing temperatures are within the siliconization temperatures. Hence the microstructure which gets evolved during siliconization must be responsible for varying thermal-diffusivities of specimens prepared at different siliconization conditions. It is evident from Fig. 4 that at lower temperature values for thermal-diffusivities are different at each siliconization conditions. While at higher temperatures values are almost same for each condition.

It may be noted that siliconization was carried out at 1450–1650 °C. The composite blocks were cooled to room-temperature. Due to matrix and reinforcement thermal mismatch interfacial de-bonding may occur which might have lead to different thermal diffusivities of the blocks. At lower temperatures, the matrix cracks (get developed during cooling after siliconization) or porosity (Fig. 5) of C–SiC composites play an important role in the overall thermal diffusivity,[6] while at higher temperatures expansion of the matrix heals all the matrix cracks and reduces their effect[18] in addition to this, at higher temperatures effect of radiations is predominant. Hence at higher temperature the effect of siliconization conditions (composition) is negligible, because thermal-diffusivities of silicon and SiC are close (at 1127 °C its value is 11.82 mm^2/s for silicon while 12.2 mm^2/s for SiC)[14,15] and interfacial gap closure above 727 °C due to expansion of matrix and reinforcement. It is well known fact that carrying out siliconization experiments at different conditions and measurement of thermal-diffusivity at each temperature which is of interest is not feasible due to high temperature and

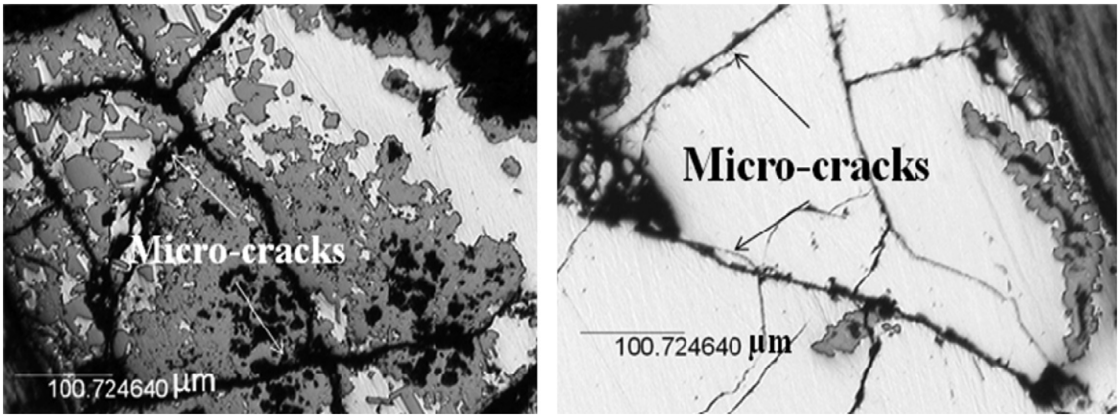

Fig. 5. Development of micro-cracks in C–SiC composites during cooling after siliconization.

vacuum involvement. Therefore it is desirable to develop a suitable model and estimation of thermal diffusivity as a function of composition in the entire temperature range of the application.

4. Thermal-diffusivity estimation

The value of thermal conductivity (λ) for composite materials depends on the individual values of the fiber (λ_f), matrix (λ_m) and interface layer (λ_i) together with the processing parameters, morphology and porosity.[9,10,19] Several approaches have been taken towards predicting the value of λ for composite materials with the simplest methods being the parallel and series models as follows:

$$\lambda = V_f\lambda_f + (1 - V_f)\lambda_m \tag{2}$$

$$\frac{1}{\lambda} = \frac{V_f}{\lambda_f} + \frac{1 - V_f}{\lambda_m} \tag{3}$$

where V_f is the volume fraction of fibers. It is known that the value of λ in SiC composites is strongly dependent on the level of porosity.[20] A relationship that has been used to take into account porosity factor is given in the following equation[21]:

$$\lambda_{\text{exptl}} = \lambda_{\text{ideal}}\left(\frac{1 - \varepsilon}{1 + 11\varepsilon^2}\right) \tag{4}$$

where λ_{exptl} is the value of thermal conductivity for the composite containing porosity, λ_{ideal} is the value of thermal conductivity for a composite when there is no porosity and ε is the fractional porosity. The models in the above form would be handy to estimate approximate values for unidirectional or randomly distributed fiber reinforced composites. In case of multidirectional non-isotropic fiber reinforced composites like 3D-stitched C–SiC composites, these models may not estimate as close as it would be for unidirectional composites. However thermal diffusivity is widely used as design input for transient heat flow conditions like aerospace/propulsion for wide temperature range. Therefore an attempt is made to estimate thermal diffusivity for 3D-stitched SiC composites. The above models are further simplified in terms of thermal diffusivity of individual constituents.

Thermal conductivity (λ) is converted into thermal-diffusivity (a) as

$$\alpha = \frac{\lambda}{\rho C_v} \tag{5}$$

where ρ is the density and C_v is the heat capacity at constant volume.

It can be remembered that C–SiC composites contain SiC, residual silicon, un-reacted carbon matrix apart from carbon fibers. For simplification following assumptions are made:

1. Un-reacted carbon matrix and carbon fibers are represented together as 2D C–C composites made from PAN-8H satin fabric and coal–tar pitch using graphitization and HIPIC technique.
2. To estimate thermal diffusivity of 3D-stitched C–SiC in in-plane direction and through-thickness direction, respective direction thermal diffusivities of 2D C–C composites are taken into computation.
3. Thermal diffusivity of SiC and silicon is direction independent.

The model equations in terms of thermal diffusivity are written as:

$$\alpha_{\text{C-SiC}} = V_{\text{C-C}}\alpha_{\text{C-C}} + V_{\text{Si}}\alpha_{\text{Si}} + V_{\text{SiC}}\alpha_{\text{SiC}} \tag{6}$$

$$\frac{1}{\alpha_{\text{C-SiC}}} = \frac{V_{\text{C-C}}}{\alpha_{\text{C-C}}} + \frac{V_{\text{Si}}}{\alpha_{\text{Si}}} + \frac{V_{\text{SiC}}}{\alpha_{\text{SiC}}} \tag{7}$$

$$\alpha_{\text{exptl}} = \alpha_{\text{ideal}}\left(\frac{1 - \varepsilon}{1 + 11\varepsilon^2}\right) \tag{8}$$

where $\alpha_{\text{C-SiC}}$ is the thermal diffusivity of C–SiC composites, $V_{\text{C-C}}$ is the volume fraction of total carbon in the C–SiC composites, $\alpha_{\text{C-C}}$ is the thermal diffusivity of 2D C–C composites in respective measurement direction, V_{Si} is the volume fraction of residual silicon, α_{Si} is the thermal diffusivity of silicon, V_{SiC} is the volume fraction of SiC and α_{SiC} is the thermal diffusivity of SiC. α_{exptl} is the value of thermal diffusivity for the composite containing porosity, and α_{ideal} is the value of thermal diffusivity for a composite when there is no porosity.

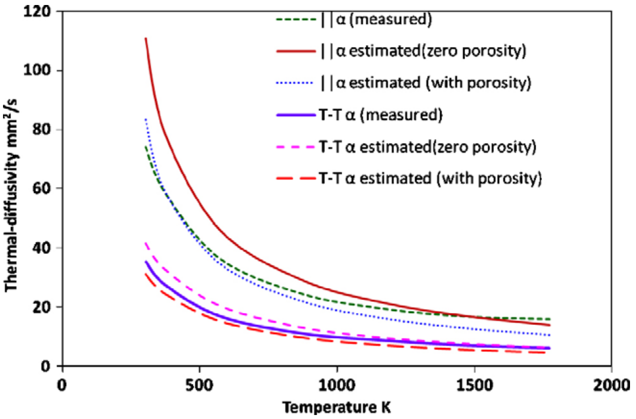

Fig. 6. Comparison of estimated and measured thermal diffusivity of blocks siliconized at 1650 °C for 120 min.

Composition given in Table 1 is converted into volume fraction based on porosity free to calculate α_{ideal} for C–SiC blocks. With the help of empirical constants (Table 2) and volume fraction of constituents (Table 1), thermal diffusivity for in-plane direction is estimated using Eq. (6) and for through-thickness direction using Eq. (7). α_{exptl} is estimated by normalizing α_{ideal} with porosity given in Table 1 and Eq. (8).

Estimated values (α_{ideal} and α_{exptl}) for C–SiC composite blocks siliconized at 1650 °C for 120 min in directions in-plane and through-thickness were plotted for entire temperature range of interest and compared with the measured values in both the directions (Fig. 6). Below 727 °C values of α_{exptl} are very close to the measured values in both the testing directions which indicate that effect of porosity must be taken into account below 727 °C. Above 927 °C α_{ideal} are much closer to the measured values which indicate that effect of porosity is negligible. This phenomenon is due to the effect of radiation heat transfer. Above 727 °C radiation heat transfer is predominant where porosity does not have effect on heat transfer, while below 727 °C contribution of radiation heat transfer is low compared to conduction heat transfer. Effect of porosity in conducting heat transfer is very high which acts as a thermal resistance. Similarly thermal diffusivities for other C–SiC blocks which were siliconized at different conditions are also estimated. Like blocks which were siliconized at 1650 °C, 120 min, estimated values for other blocks were very close to the measured values.

5. Conclusions

3D-stitched C–SiC composites are successfully fabricated using coal–tar pitch impregnation, carbonization, graphitization, HIPIC and LSI method. Effect of siliconization conditions on thermal diffusivity of 3D-stitched fiber architecture-based C–SiC composites was investigated using laser-flash equipment in in-plane and through-thickness directions up to 1500 °C. The in-plane thermal-diffusivities of the C–SiC composite blocks are two-three times higher than those in the through-thickness direction. Thermal-diffusivity decreases with increase in testing temperature for all the C–SiC composite blocks and in both the testing directions. It varies from 77 at room-temperature to 14.7 mm^2/s at 1500 °C in in-plane and 36–6.1 mm^2/s in through-thickness direction. Effect of siliconization on thermal-diffusivity is higher at lower testing temperatures. Beyond 927 °C difference in thermal-diffusivities of C–SiC composite blocks is negligible in both the testing directions. Thermal-diffusivity data could be well correlated with an empirical relation. Using volume fraction of the constituents of C–SiC composites thermal-diffusivity was estimated for both the testing directions and compared with experimental. The estimated thermal diffusivities at any siliconization condition show reasonably good correlation with the measured values. The information was used as design input for designing of the jet-vanes.

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