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# Thermal conductivities of Ti–SiC and Ti–TiB$_2$ particulate composites

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Composites of commercial-purity titanium reinforced with 10 and 20 vol% of SiC and TiB$_2$ particulates were produced by powder blending and extrusion. Heat treatments were conducted on each of these composites. The thermal diffusivities of the composites were measured as a function of temperature using the laser flash technique. Thermal conductivities were inferred from these measurements, using a rule-of-mixtures assumption for the specific heats. It has been shown that, while an enhancement of the thermal conductivity is expected to arise from the presence of both types of reinforcement, this behaviour is in fact observed only with the Ti–TiB$_2$ composites. The thermal conductivity of Ti–TiB$_2$ composites is significantly greater than that of the unreinforced matrix and rises with increasing volume fraction of reinforcement. In contrast, the conductivities of the Ti–SiC composites were considerably lower than that of the unreinforced titanium and decreased with increasing volume fraction of SiC reinforcement. These results have been interpreted in terms of the thermal resistance of the reaction layers which exist between the matrix and two types of particulate reinforcements. The faster reaction kinetics between SiC and Ti gives rise to a thicker reaction layer for a given heat treatment than that between Ti and TiB$_2$ and is also accompanied by a much larger volume change ( − 4.6%). It is proposed that this volume decrease, giving rise to interfacial damage and a network of microcracks, is at least partly responsible for a high interfacial thermal resistance, reducing the conductivity of the Ti–SiC composite. These results indicate that TiB$_2$ would be preferable to SiC as a reinforcement in Ti for situations where a high thermal conductivity would be beneficial.

## 1. Introduction

It is being increasingly recognised [1, 2] that transport properties such as thermal and electrical conductivity may be of considerable significance for advanced composites in various potential applications. For example, thermal conduction plays an important role in determining the resistance to thermal shock [3–6]. Furthermore, combinations of high conductivity and good mechanical strength obtainable by the reinforcement of pure metals with ceramic may be more attractive than those obtainable by alloying, which often sharply reduces both electrical and thermal conductivities.

Thermal properties of Ti reinforced with ceramics such as SiC and TiB$_2$ are of particular interest, from both practical and theoretical viewpoints. Handbook data indicate both SiC and TiB$_2$ to have higher thermal conductivities than Ti and its alloys. However, heat is transported in these ceramics only via phonons, which are readily scattered by defects, so that the thermal conductivity is highly sensitive to microstructural features such as grain size and porosity. Furthermore, enhancement (or even retention) of the matrix thermal conductivity in a composite requires the transport of heat across the matrix–reinforcement interface. There is very little published information about how efficiently this can occur in metal–matrix composites (MMCs), where transport is in general predominantly via electrons in the matrix and phonons in the reinforcement. Moreover, Ti-based composites are particularly prone to interfacial chemical reaction during processing and high-temperature service. It has been established that in Ti–SiC this reaction can both impair mechanical properties [7, 8] and reduce the thermal conductivity [9]. Less information is available about the Ti–TiB$_2$ system, although there is evidence that the chemical reaction [10] which occurs (to form TiB) is slower and creates less damage in the vicinity of the interface [11] than is the case for Ti–SiC.

Considerable effort has been directed towards the modelling [12–18] of thermal conduction in composites. (Electrical conduction is mathematically identical.) Many of these models are oriented towards continuous fibre or particulate composites, but the Eshelby method [19–21] can be used for short and misoriented fibres. There is particular interest in simulation of the effect of an interfacial thermal resistance. The Hasselman and Johnson model [17] (for spheres) and the Eshelby method [22] (for ellipsoids) are particularly well suited to the incorporation of the effect of interfacial resistance.

Experimental studies have been made of both thermal [9, 21, 23, 24] and electrical [21, 25–27] conduction in MMCs. These have in general confirmed the validity of theoretical models, but there has been little information about interfacial heat transport. (Of course, in many systems the reinforcement is effectively a thermal insulator, so that heat transfer across the interface is negligible in any event.) The data of Gordon and Clyne [21] indicate that titanium reinforced with SiC short fibres has a conductivity below that expected with a perfect interface, assuming a handbook value for the conductivity of the SiC, but above that expected in the absence of any interfacial heat transfer. Reeves *et al.* [11], studying Ti reinforced with SiC particulate, also found lower conductivities than expected if the interfaces were perfect, with the discrepancy increasing after heat treatment. Very thick reaction layers led to conductivities below those expected with insulating reinforcements, which was attributed to the effect of a network of cracks within and around the reaction zone.

In the present paper, experimental data are presented for Ti–SiC and Ti–$TiB_2$ particulate composites, with and without substantial heat treatments. The main objective is to clarify the role of interfacial heat transfer during heat conduction in these materials and to establish correlations with microstructural and mechanical information.

## 2. Experimental procedure

### 2.1. Composite production and microstructure

Composites containing 10 and 20% by volume of reinforcement were made by dry blending of Ti powder (purity 99.8%, 1400 p.p.m. oxygen, 50–150 μm particle size) and either SiC or $TiB_2$ particulate ($\sim$10–30 μm diameter in both cases). The powder mixture was cold-pressed into a copper can, which was evacuated, heated and sealed by electron beam welding prior to extrusion. The can was soaked for 2 h in a furnace at 900 °C and then extruded in a circular-section die of diameter 20 mm, with a die semi-angle of 45° and an extrusion ratio of about 17. The extrusion chamber was held at about 450 °C and the die at about 250 °C. An unreinforced Ti matrix extrudate was produced in exactly the same way as the composites. In addition, specimens of SiC and $TiB_2$ were manufactured for thermal measurements, using the same ceramic powders as those employed for the composites. The SiC specimens were produced by pressureless sintering at 2150 °C, while for $TiB_2$ hot isostatic pressing was employed, at 1900 °C and 50 bar. While the SiC was about 99% dense, the measured porosity level (see below) in the $TiB_2$ was about 27%; account was taken of these porosity levels in evaluating the true thermal conductivity of the two ceramics.

The extruded composites were homogeneous and contained well-dispersed particulate (Fig. 1). No interfacial reaction layer could be seen in optical or SEM micrographs with either type of reinforcement. However, a layer about 0.2 μm or so in thickness is expected [9, 28, 29] with SiC, and a somewhat thinner layer with $TiB_2$. Certain composites were heat-treated at 950 °C for 80 and 300 min. These heat treatments were carried out on short cylindrical specimens sealed in ampoules under argon.

### 2.2. Density and stiffness measurements

Porosity levels were established by precision densitometry, using a microbalance (± 10 μg). These levels were obtained on the basis of known constituent densities and volume fractions, so that they are less accurate as the thickness of the interfacial reaction layer becomes significant. For the $TiB_2$ specimen, which had a high porosity level, coating with a lacquer of known density was necessary to eliminate penetration of the suspension liquid into the specimen. Stiffness measurements were made in order to monitor the mechanical effects of interfacial damage. These also become less easy to interpret as the volume fraction of reaction products increases, but should nevertheless give a measure of the general integrity of the interfacial region. It is essential that a low-strain technique is used, since any plastic strain can itself cause damage, at least for Ti–SiC composites [11]. An ultrasonic resonance technique [30], using a mechanical impulse to excite the selected vibration mode, was therefore employed. The method was applied to specimens in the form of cylinders.

### 2.3. Thermal conductivity measurements

Thermal diffusivity measurements were made using the laser flash technique. The technique is based on the use of a small disc-shaped specimen, the front face of which is subjected to an instantaneous, uniform energy pulse. From the recorded temperature history of the opposite face the thermal diffusivity may be calculated [31].

In the apparatus used [32], the heat pulse is supplied by a 100 J Nd–glass laser with a wavelength of 1.067 μm and a pulse dissipation time of 0.6 ms. The specimen, in a pyrolytic graphite sample holder, is heated to the measurement temperature inside a graphite susceptor located within an induction coil. This assembly is located within a vessel which can contain either vacuum or inert gas. Radiation from the specimen rear face is collected by a calcium fluoride lens and mirror system and focused on to an InSb infrared detector. The biased output from this detector is amplified and fed into a microcomputer via an analogue-to-digital convertor. The computer is programmed to calculate the thermal diffusivity, $\alpha$, using

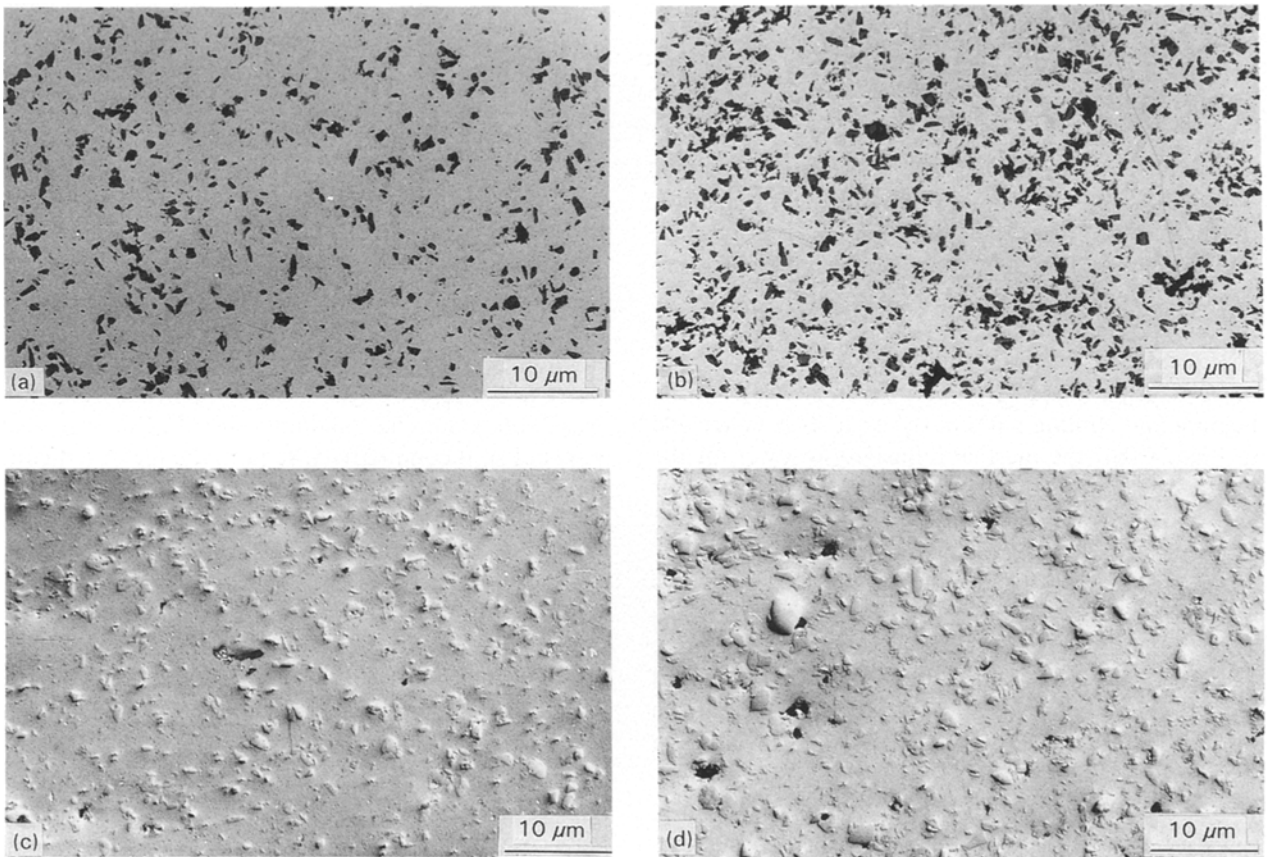

*Figure 1* Optical micrographs of composites in the as-extruded state: (a) Ti–10% SiC, (b) Ti–20% SiC, (c) Ti–10% $TiB_2$ and (d) Ti–20% $TiB_2$.

TABLE I Specific heat data [35] for constituents of the composites

| Material | Coefficient[a] | | | | |
|---|---|---|---|---|---|
| | *a* | *b* | *c* | *d* | *e* |
| Ti | 0.444 556 | 0.000 227 | 0 | 0 | 0 |
| SiC | – 0.440 83 | 0.005 706 | $-8.1 \times 10^{-6}$ | $5.34 \times 10^{-9}$ | $-1.3 \times 10^{-12}$ |
| $TiB_2$ | – 0.233 21 | 0.004 152 | $-4.7 \times 10^{-6}$ | $1.86 \times 10^{-9}$ | 0 |

[a] The specific heat, in $J g^{-1} K^{-1}$, is given by the equation $C_p = a + bT + cT^2 + dT^3 + eT^4$ where $T$ is in Kelvin.

the equation

$$\alpha = \frac{\omega L^2}{\pi^2 t_{1/2}} \quad (1)$$

where $\omega$ is a dimensionless heat term [32], $L$ is the sample length and $t_{1/2}$ is the time period required for the rear face of the specimen to reach half of the maximum value. The boundary conditions for the flash method assume a homogeneous sample uniformly irradiated, negligible pulse duration time and no heat losses. Corrections for finite pulse time are made using the method due to Clark and Taylor [33] and the $\omega$ term is corrected for heat losses using the analysis due to Cowan [34]. Corrections for sample length change during heating were made using the thermal expansion coefficient of titanium.

The thermal conductivity, $K$, is the product of thermal diffusivity $\alpha$, density $\rho$ and specific heat $C_p$:

$$K = \alpha \rho C_p \quad (2)$$

The conversion of diffusivity data to conductivity values was carried out using handbook [35] specific heat values for the titanium and SiC. The specific heat values were fitted to a fourth-order polynomial over the measurement temperature range. The specific heats of the composites were taken as a weighted mean between the values of the constituents. The data used are shown in Table I. This apparently rather crude approach should be acceptable in view of the insensitivity of specific heat values to microstructural features, although it must be noted that the heavily reacted composites contained significant amounts of other phases.

Specimens were in the form of circular discs, 3 mm thick and 8 mm in diameter. These were machined from both composite and unreinforced extrudates so that the axis of the disc was parallel to the extrusion axis. Measurements were made during heating and cooling between 100 and 700 °C with temperature intervals of 30 to 40 °C. The heating and cooling periods extended over a total of 2 h.

## 3. Results and discussion

### 3.1. As-extruded composites

Measured thermal diffusivity data are shown in Fig. 2 for the as-extruded Ti–SiC and Ti–$TiB_2$ composites, as a function of temperature. Measurements were made during both the heating and cooling periods. The results indicate that, within experimental error, the measured values (for a given temperature) were the same before and after heating. This confirms that no significant microstructural changes occurred during the measurements. This was the case for all of the specimens, so the following figures are presented with no distinction made between measurements from the heating and cooling parts of the cycle. It is noticeable in Fig. 2a that the thermal diffusivity is lower for the higher SiC content, whereas the reverse is the case for $TiB_2$. In addition, a clear trend is apparent for the diffusivity to decrease with increasing temperature. However, this does not necessarily reflect the changes in conductivities, since the specific heats of the constituents are also expected to change with temperature. The remaining data are therefore presented as thermal conductivities, since this property is expected to be more closely related to microstructural features of the materials.

A comparison is shown in Fig. 3a between the thermal conductivities of the unreinforced matrix, and of both types of composite, in the as-extruded state. These results confirm that raising the content of SiC particulate causes a small progressive reduction in conductivity, whereas an increase is produced when $TiB_2$ particles are incorporated. In Fig. 3b, experimental conductivity data are shown for the SiC and $TiB_2$ specimens, together with the Ti results. The data have been corrected for the presence of the porosity (1% for SiC and 27% for $TiB_2$), using the Eshelby method [21], so that the values shown should correspond to dense material. These corrections, which are based on assuming randomly-distributed, spherical pores of zero conductivity, require the measured conductivities to be increased by about 1.5% for SiC and 56% for $TiB_2$. It can be seen that both of the ceramic constituents, particularly the SiC, have appreciably higher conductivities than Ti. This is in agreement with most published handbook data. It is evident that some marked microstructural difference between the two types of composite must be responsible for the observed characteristics. A pronounced difference in the nature of the interface is the most obvious explanation. When exploring this behaviour, account

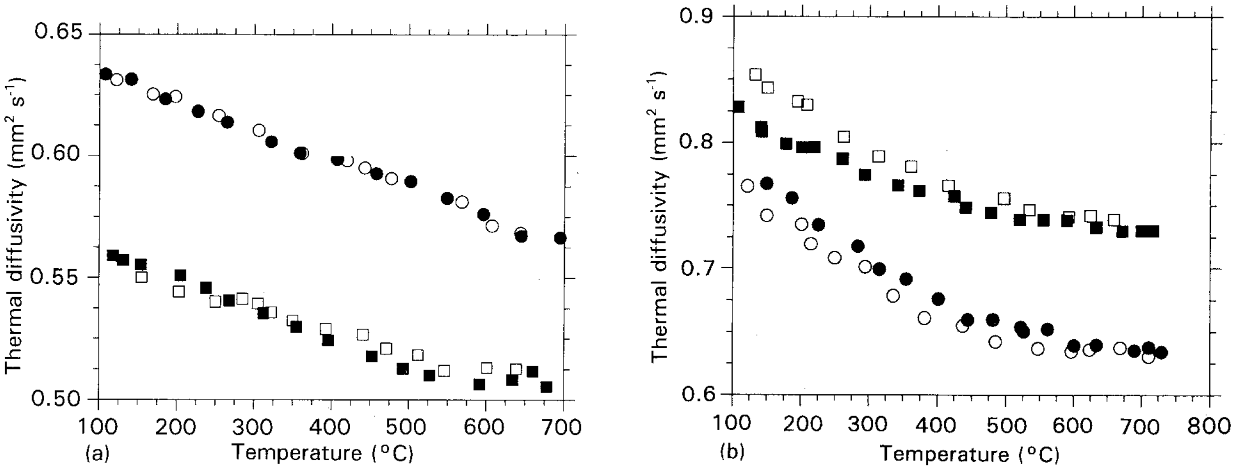

*Figure 2* Measured thermal diffusivity data over a range of temperature for (a) Ti–SiC and (b) Ti–$TiB_2$ composites, all in the as-extruded state. (●) 10% SiC or $TiB_2$, heating; (○) 10%, cooling; (■) 20%, heating; (□) 20%, cooling.

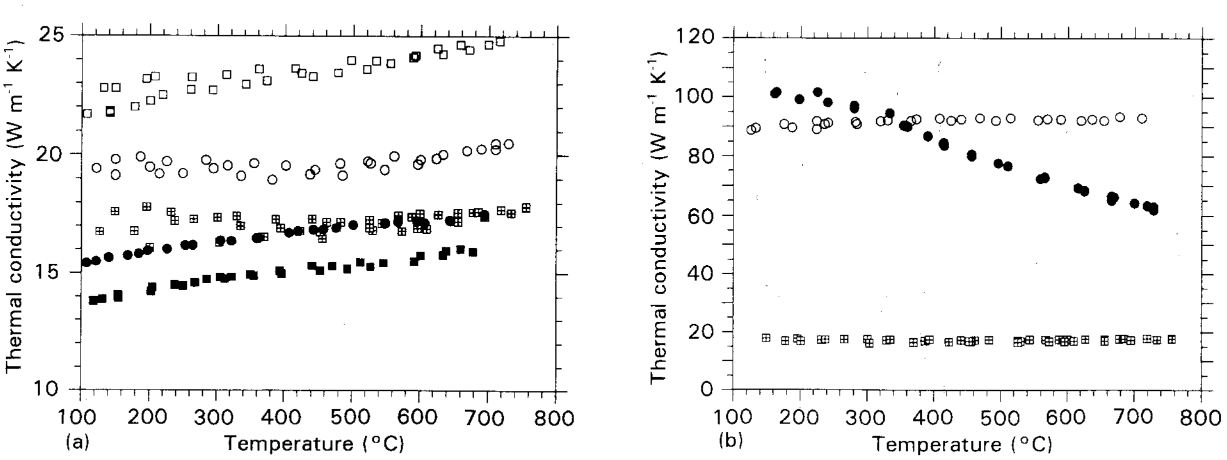

*Figure 3* Thermal conductivity data over a range of temperature. (a) (⊞) Unreinforced titanium and the four composites, all in the as-extruded state: (●) 10% SiC, (■) 20% SiC, (○) 10% $TiB_2$, (□) 20% $TiB_2$. (b) (●) SiC and (○) $TiB_2$ specimens, together with (⊞) Ti.

should be taken of the changes in particle/matrix conductivity ratio with temperature, which can be seen from Fig. 3b to differ slightly for the two systems.

In order to analyse in detail the data shown in Fig. 3, it is useful to examine models for thermal conduction in composites which incorporate an interfacial thermal resistance. Such a thermal resistance is usually characterized by an interfacial heat transfer coefficient or thermal conductance, $h$ ($\mathrm{W\,m^{-2}\,K^{-1}}$), defined as the proportionality constant between the heat flux through the boundary, $q_i$, and the temperature drop across it, $\Delta T_i$

$$q_i = h\Delta T_i \tag{3}$$

For a volume fraction f of spherical particles (having a radius $r$) in a relatively dilute composite, an analytical expression given by Hasselman and Johnson [17] can be used to predict the conductivity of the composite, $K_C$:

$$K_C = K_M \frac{\left[2f\left(\dfrac{K_P}{K_M} - \dfrac{K_P}{rh} - 1\right) + \dfrac{K_P}{K_M} + 2\dfrac{K_P}{rh} + 2\right]}{\left[f\left(1 - \dfrac{K_P}{K_M} + \dfrac{K_P}{rh}\right) + \dfrac{K_P}{K_M} + 2\dfrac{K_P}{rh} + 2\right]} \tag{4}$$

where $K_P$, $K_M$ are the conductivities of particles and matrix, respectively. This equation can be used in conjunction with the data in Fig. 3 to estimate the interfacial conductance in the composites. The results of this exercise are shown in Fig. 4. In Fig. 4a, the experimental conductivity data for the Ti–10% SiC composite have been plotted as ratios to that of the matrix, over the range of temperature. Also shown are predictions from Equation 4 corresponding to several $h$ values, using the experimental conductivity values for matrix and reinforcement at the appropriate temperatures, and a particle radius of 10 μm. It can be seen that these data are consistent with an $h$ value of around $10^6\,\mathrm{W\,m^{-2}\,K^{-1}}$, apparently rising somewhat with increasing temperature. This is a relatively small value for particle–matrix heat exchange in a system with particles of this size, and it can be seen from the figure that it represents behaviour rather closer to an insulating interface ($h = 0$) than to a perfect interface ($h = \infty$). Corresponding plots for the Ti–20% SiC composite are shown in Fig. 4b. These data suggest rather similar interfacial characteristics to the 10% composite.

From the data shown in Fig. 5, it is evident that the interfacial characteristics are very different in the Ti–$TiB_2$ system. These are analogous plots to those in Fig. 4 and they clearly show that the interfacial conductance is much higher in this system. In fact, the data are broadly consistent with perfect interfacial contact. The results appear to suggest, as with the Ti–SiC data, that the value of $h$ rises slightly with increasing temperature. This might be due to a change in the efficiency with which heat transported largely by electrons (in the matrix) can be transferred to phonon transport (in the ceramic) as the energy crosses the interface. An alternative explanation might lie in the changing interfacial stress state as differential thermal expansion occurs, although from this point of view a decrease in $h$ might have been expected on heating since it will reduce the compressive radial stress across the interface. However, the conclusion that $h$ increases on heating is dependent on the measured conductivities of the pure materials accurately reflecting the behaviour of the constituents in the composites. This may be unreliable, since the pure ceramics were polycrystalline and hence, even after correction for the porosity present, could have differed in conductivity from the (largely monocrystalline) reinforcement particles.

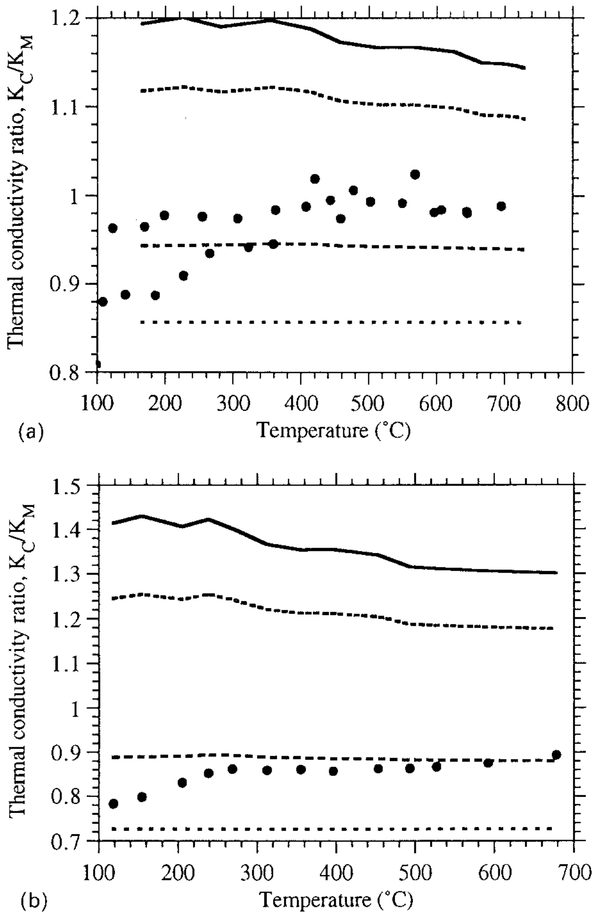

*Figure 4* Plots of the thermal conductivity ratio, $K_C/K_M$, against temperature, showing both (●) experimental data and predictions from Equation 4, based on several values for $h$ (in $\mathrm{W\,m^{-2}\,K^{-1}}$) and using measured conductivities for both matrix and reinforcement (see Fig. 3b). Plots are shown for (a) Ti–10% SiC, (b) Ti–20% SiC. (● ● ●) Experimental; (- - - -) $h = 0$, (- - -) $1 \times 10^6$, (- - -) $1 \times 10^7$, (——) ∞.

In any event, the clear conclusion from the data presented is that the thermal resistance of the interface is high in the Ti–SiC system, but low in Ti–$TiB_2$ composites. The poor thermal contact of the Ti–SiC interface is probably related to the interfacial reaction layer. The reaction between Ti and SiC, to produce TiC and $Ti_5Si_3$, has been studied in some detail [28, 29, 36–43]. It has also been reported [7, 8] that the reaction appears to have a particularly deleterious effect on the mechanical properties. In a previous study of the Ti–SiC system [9], preliminary conductivity data were reported indicating poor interfacial contact, and it was noted that the reaction is accompanied by a relatively large volume change* of

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* Such changes are simply estimated using density data.

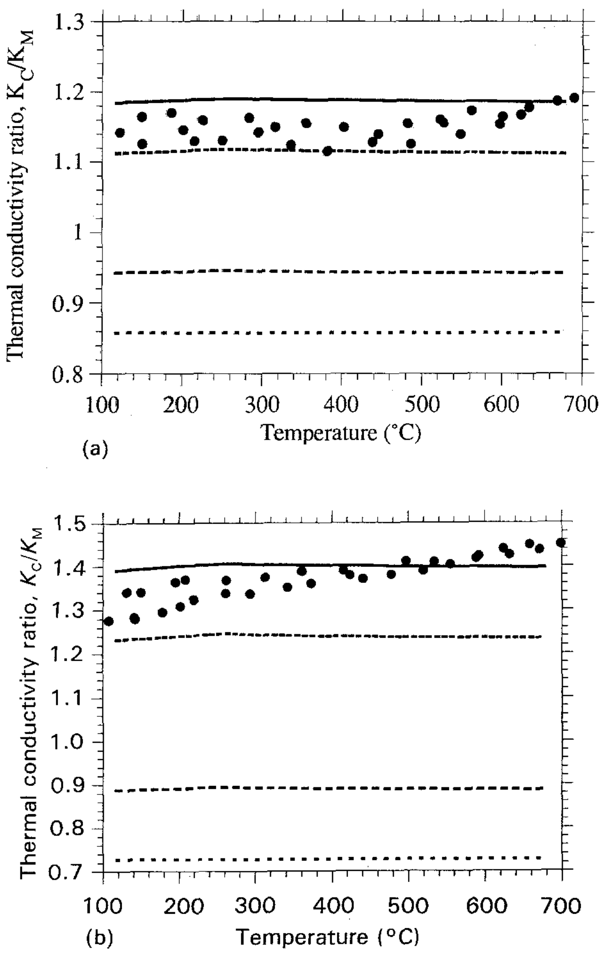

*Figure 5* Thermal conductivity ratio plots, as for Fig. 4, showing (a) Ti–10% $TiB_2$ and (b) Ti–20% $TiB_2$.

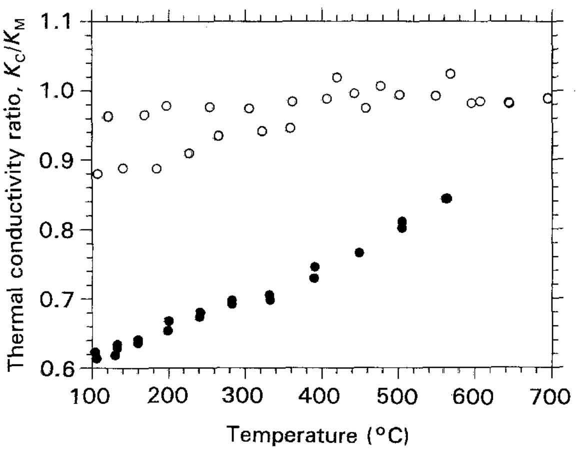

*Figure 6* Thermal conductivity ratio plots for Ti–10% SiC composites, (●) with and (○) without a heat treatment of 80 min at 950 °C.

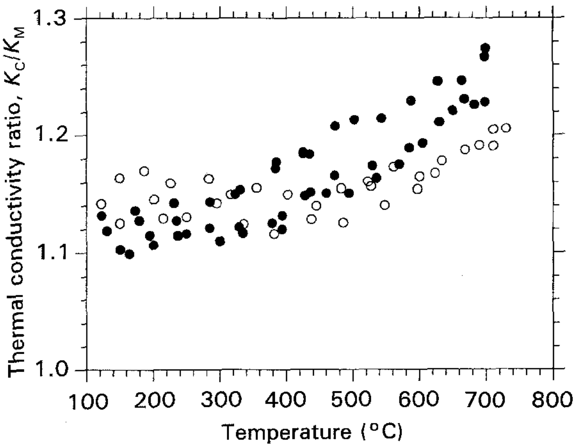

*Figure 7* Thermal conductivity ratio plots for Ti–10% $TiB_2$ composites, (●) with and (○) without a heat treatment of 300 min at 950 °C.

– 4.6%. This volume reduction will set up radial tensile stresses at the interface, opposing the differential thermal contraction stresses and tending to cause interfacial cracking. It certainly appears from the data presented here that, even with the relatively thin reaction layer formed during this processing operation (reported [9, 44] as ~0.2–0.4 μm), interfacial conduction is severely impaired.

A reaction also takes place [10, 45] in the Ti–$TiB_2$ system, to form a monoboride, TiB, although the rate of reaction is apparently slower than for Ti–SiC. It has also been reported [11, 44] that this reaction does not mechanically weaken the interface to the same degree as that in Ti–SiC. This may be at least partly a consequence of the much lower volume change accompanying the reaction, reported as – 1.4%. Certainly the data reported here confirm that the interface in these as-fabricated Ti–$TiB_2$ composites has a high thermal conductance.

## 3.2. Effect of heat treatment

Data are shown in Fig. 6 for Ti–10% SiC composites, before and after a heat treatment of 80 min at 950 °C. This treatment leads to formation of a reaction layer a few micrometres in thickness. It can be seen that the low conductivity of this composite is further depressed by the heat treatment. In fact, the conductivity is below that expected with 10% of fully insulating particles ($h = 0$). This has been explained [9] on the basis of a network of cracks being formed around the interface, effectively excluding the heat flux from a volume greater than that occupied by the particles.

The effect of a substantial heat treatment on Ti–$TiB_2$ composites is illustrated by the data in Fig. 7, which are for Ti–10% $TiB_2$ after 300 min at 950 °C. This heat treatment leads to a reaction layer similar in thickness to that for the Ti–SiC composite of Fig. 6. These results indicate that interfacial conduction is not impaired in this system as the reaction layer becomes relatively thick, and indeed appears to be slightly enhanced (particularly at elevated temperature). Evidently, interfaces in Ti–$TiB_2$ composites retain their capacity for heat conduction much more effectively than those in Ti–SiC. While the thermal properties of the reaction products in the two cases may affect the characteristics somewhat, it seems very unlikely that the compounds concerned would be sufficiently different in this regard to explain the observed behaviour. A more probable explanation lies in the degree of local microcracking, porosity and general damage in the vicinity of the interface.

TABLE II Experimental data for the density and Young's modulus of composites and unreinforced matrix, in the as-extruded state.

| Material | Density | | | Young's modulus | | |
|---|---|---|---|---|---|---|
| | Theoretical $\rho_0$ (Mg m$^{-3}$) | Experimental $\rho$ (Mg m$^{-3}$) | Change $\Delta$ (%) | Theoretical $E_0$ (GPa) | Experimental $E$ (GPa) | Change $\Delta$ (%) |
| Ti | 4.510 | 4.486 | − 0.5 | 108.5[a] | 108.5 | 0 |
| Ti–10% SiC as-fab. | 4.376[b] | 4.295 | − 1.8 | 124[c] | 112 | − 9.6 |
| Ti–20% SiC as-fab. | 4.242[b] | 4.092 | − 3.5 | 140[c] | 117 | − 16.4 |
| Ti–20% $TiB_2$ as-fab. | 4.508[b] | 4.400 | − 2.4 | 143[c] | 143 | 0 |

[a] Measured $E$ taken as $E_0$.

[b] Calculated from values for matrix and reinforcement, using the rule of mixtures.

[c] Calculated from values for matrix and reinforcement, using the Eshelby method [46].

## 3.3. Mechanical behaviour

The above conclusions are supported by the density and stiffness data shown in Table II. The density results show that the porosity levels in the composites are relatively low, although they increase with ceramic content. Interfacial reactions should not affect the apparent porosity levels significantly, since neither the mass nor the total volume occupied are expected to change; however, the transformation strains of the reactions will tend to cause internal stresses and microstructural damage. The Young's modulus data show that the Ti–SiC composites have substantially lower stiffnesses than predicted for sound material with perfect interfaces ($E_0$ values). The stiffness will be reduced by the presence of the porosity (by a proportion which is approximately twice that of the porosity content in this range), but this effect is insufficient to account for the observed reductions. This stiffness reduction is presumably caused by imperfect interfacial contact. It is clear from the stiffness of the Ti–20% $TiB_2$ composite that the interfacial integrity is much higher in this system.

## 4. Conclusions

The following conclusions can be drawn from this work.

(a) The thermal conductivity of titanium tends to be progressively raised by the presence of $TiB_2$ particles, but depressed by the presence of SiC particles.

(b) On the basis of the measured conductivities of the three constituents, and using the Hasselman and Johnson model for composite conductivity, values for the interfacial conductance (or heat transfer coefficient) can be estimated for these composites. High values ($\sim 10^8$ W m$^{-2}$ K$^{-1}$) were found appropriate for the Ti–$TiB_2$ interfaces, but low values ($< 10^6$ W m$^{-2}$ K$^{-1}$) for the Ti–SiC interfaces.

(c) In both systems, heat treatments causing matrix–particle reactions were found to decrease the interfacial conductance. The effect was markedly more pronounced for the Ti–SiC case. The observed behaviour is explained in terms of interfacial cracking and damage occurring more readily in this system. It may be that the relatively high volume contraction reported to accompany the reaction is at least partly responsible for this effect.

(d) Measurements made of specimen density and elastic modulus were consistent with the suggestion that interfacial damage, as opposed to the thermal properties of the reaction products, was primarily responsible for the observed thermal characteristics of the Ti–SiC composites.

(e) These results confirm that even very limited degrees of interfacial reaction in the Ti–SiC system can have deleterious effects on both thermal and mechanical properties. On the other hand, it has also been confirmed that the presence of $TiB_2$ in Ti can, as expected from handbook data for the constituents, raise the thermal conductivity substantially. There have been very few reports hitherto of the presence of reinforcement in MMCs improving the thermal properties of the matrix in this way.

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