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Thermal Conductivity in Hot-Pressed Silicon Carbide

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Abstract: Silicon carbide ceramics with equiaxed grain structure were obtained by hot-pressing at 2000°C in a vacuum with the aid of sufficient amounts of Al_2O_3 and Y_2O_3 as sintering additives. The thermal conductivity of the SiC ceramics was investigated using a laser-flash technique and a pure polycrystalline 6H-SiC prepared by Lely's method for bulk crystal growth for comparison purposes. Experimental results revealed that the thermal conductivity of the SiC ceramics, consisting primarily of 6H, 4H and 3C polytypes with various concentrations, appeared to exhibit a strong dependence upon the content as well as the polytype of the SiC at lower temperatures, the dependence, however, became less pronounced at elevated temperatures. Solid-solution and inhomogeneity are assumed to play an important role in further reducing phonon mean free path of the ceramics. A considerable deviation in thermal resistivity between the SiC ceramics and the pure 6H-SiC was observed. A difference in the temperature dependence of phonon mean free path compared with the theoretical expectation was discussed. © 1996 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

Silicon carbide (SiC) has been known as a promising candidate material for ceramic engine components because of its excellent oxidation resistance and strength retention to high temperature, and has been widely used for refractories, wear-resistance tools, and electronic devices by utilizing its high thermochemical stability, hardness, and large energy gap characteristic, respectively. Its high stability with respect to neutron radiation, associated with sufficient thermal conductivity, gives scope for a potential application in nuclear industries.[1,2] Recently, SiC has become a prime candidate material for high-temperature heat exchanger applications, primarily because of its excellent thermo-stability and corrosive resistance in severe environments, and sufficient thermal conductivity.[3] Further, one of the attractive features in the use of SiC ceramics as a heat exchanger is that it allows electro-discharge machining for delicate design, and components with complex geometry.[4] This is of great interest and a number of vigorous investigations have been conducted.[4,5]

To obtain a near full-dense SiC compact has been a major topic for many researchers over the last three decades[6] and one of the most successful methods to obtain SiC compacts with density in excess of 98% theoretical density is by employing liquid phase sintering with the aid of sintering additives such as Al_2O_3 and Y_2O_3, etc.[6–8] However, high-temperature treatments often lead to a change in the crystal structure, i.e. $\beta \rightarrow \alpha$ transformation. This often accompanies the formation of large hexagonal grains, which promote stress concentration, and consequently is detrimental to mechanical properties of the compact. However, this is avoided by either employing hot-pressed sintering (as discussed in this article) or by employing pressureless sintering under a nitrogen atmosphere[9] and higher content of sintering aids.[8]

In considering the use of SiC as materials for thermal-conversion, as well as for heat exchanger systems, the thermal conduction properties have become one of the crucial considerations for design purposes. Although thermal conduction properties of SiC ceramics have been well-documented,[3] they appeared to be a function of processing. The influence of phase distribution on these properties was barely found. Sakai and Aikawa[10] have recently published an article which, to the authors' knowledge, might be the latest opened literature concerning the thermal conductivity in relation to the phase distribution of the SiC aggregate. They found that the thermal conductivity of the SiC aggregate decreased appreciably as the content of 4H polytype increased, which might result from the influence of a solid solution effect of Al_2O_3 into β-SiC (3C-SiC) during hot-pressing. In this study, emphasis will be focused on investigating the thermal conduction properties of SiC ceramics. An attempt to explore the influence of phase distribution of SiC polytypes on thermal conduction of the SiC ceramics is also tentatively presented.

2 THEORY FOR THERMAL CONDUCTION

For most dielectric solids with perfect lattice, the thermal conduction behaviour is primarily controlled by the transfer of thermal-elastic waves (called phonons by Peierls[11]). The transport of phonons will be scattered easily by other phonons upon increasing the temperature, thereby resulting in a decreased length of the phonon mean free path, consequently decreasing the thermal conductivity. Thus the phonon mean free path (L) can be related to the inverse of absolute temperature (T) by:[11]

$$L \propto \frac{1}{T} \tag{1}$$

However, in real dielectric solids, imperfections such as pore, impurity, crystal boundary and dislocation, etc., are unavoidably present during preparation. These imperfections interrupt with phonons causing phonon scattering, thereby further reducing the mean free path by:[12]

$$\frac{1}{L} = \frac{1}{L_1} + \frac{1}{L_2} + \dots \tag{2}$$

where L_1, L_2,... represent the phonon mean free paths caused by various scattering effects. Considering the thermal conduction as a result of transportation of thermal elastic waves, thermal conductivity (K) of a dielectric solid can be expressed by:[13]

$$K = \frac{1}{3} \rho \, L \, C_p \, \nu \tag{3}$$

where ρ is the bulk density of the solid (in g/cm^3), C_p is the heat capacity of the solid (in J/g·K) and ν the elastic wave velocity in the solid (in cm/s). Alternatively, the thermal conductivity can simply be determined by direct measurement of the density (ρ) and thermal diffusivity (α, in cm^2/s) of the solid:

$$K = \rho \, C_p \, \alpha \tag{4}$$

The thermal conductivity (K), as well as the diffusivity (α), is inversely proportional to the temperature if C_p and ν of the solids are approximately constant in a specific range of temperatures.

3 EXPERIMENTAL PROCEDURES

Four chemically pure SiC powder mixtures (Showa Denko, purity of α ~98·6%, with a mean grain size of 0·46 μm; and Mitsui Touatsu, MSE 20, purity of β ~98·7%, mean grain size of 0·15 μm) with α/β ratios of 100/0, 50/50, 20/80, and 0/100 in weight percent, designated A-, B-, C- and D-SiC, respectively, were prepared. The composition (in volume percentage) of α- and β-SiC in terms of phase distribution for the above mixtures are given in Table 1 and designated as **Raw**. The mixtures were placed in a plastic bottle containing SiC balls and ethanol, mixed for 24 h. To promote sinterability, 5 wt% of Al_2O_3 and 2 wt% of Y_2O_3 were added prior to ball-milling. After drying, the powders were pressed uniaxially under 100 MPa

Table 1. Comparison of the concentration (in volume percent) of SiC polytypes for A-, B-, C- and D-SiC starting materials (Raw) and after hot-pressed sintering (HP)

Polytypes	3C		6H		4H	
Materials	Raw	HP	Raw	HP	Raw	HP
A-SiC	13.5	5.0	86.5	87.3	0.0	7.7
B-SiC	42.4	29.2	57.6	52.5	0.0	18.3
C-SiC	67.2	7.0	37.3	88.3	0.0	4.7
D-SiC	100	76.0	0.0	0.0	0.0	24.0

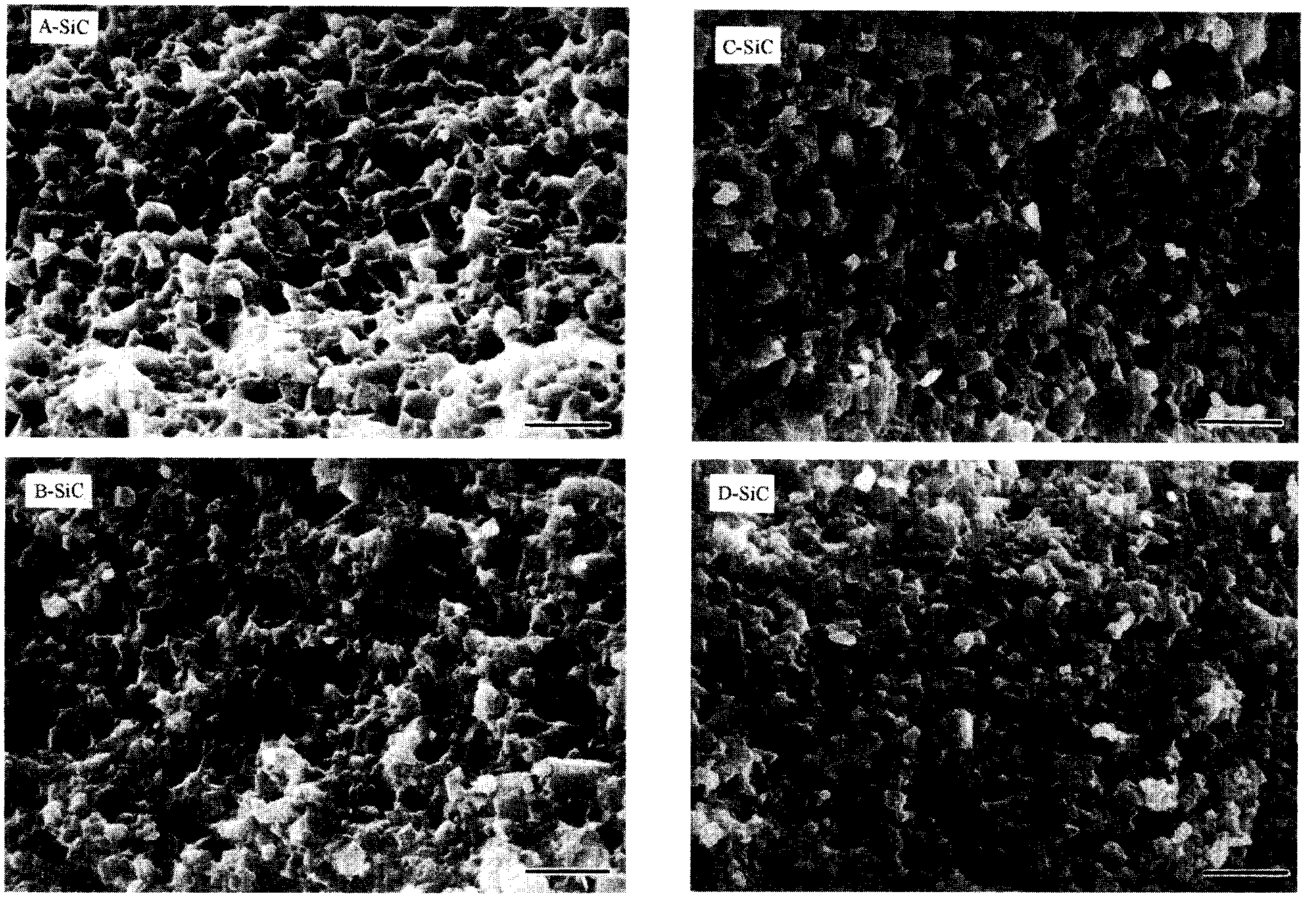

Fig. 1. Microstructure of the four kinds of SiC ceramics.

into a cake-shape of 6 cm diameter and 1 cm thickness. The preform was placed into a graphite die with both top and bottom faces coated with boron nitride powder to prevent the occurrence of undesired chemical reactions during heating. The graphite die was heated by an inductive-heating furnace to 2000°C for 2 h in vacuum under a uniaxial pressure of 30 MPa. A pure 6H-SiC ceramic, provided from Opto-Electric Materials Research Laboratories at the Industrial Technology Research Institute was obtained under N_2 atmosphere by an improved Lely's method for bulk crystal growth and the manufacturing procedures are described in a previous publication by Chuang and Hsu.[14]

The specimens used for thermal diffusivity (α) and specific heat (C_p) measurements were prepared with dimensions of 10 mm in diameter and 2·0 mm in thickness. The experimental procedures for the measurement of α and C_p have been described previously.[15]

The microstructures of the as-sintered SiC aggregates were examined using a scanning electron microscope (Cambridge Instruments, SEM-360). The crystallographic structures of the ceramics were determined by means of an X-ray diffractometer (Philips, APD-1700) equipped with a microcomputer to facilitate phase identification and analysis.

4 RESULTS AND DISCUSSION

4.1 Phase distribution and microstructure of the SiC aggregates

Table 1 lists the distribution of SiC polytypes of the starting powders (**Raw**) and hot-pressed compacts (**HP**). The concentrations of the polytypes in the SiC aggregates are determined according to the method proposed by Hase *et al.*,[16] who measured the phase distribution of the polytype through the use of X-ray diffraction peak intensity. The starting powders, except for the D-SiC which is composed entirely of 3C-SiC (β phase), are primarily of a mixture of 3C- and 6H-SiC (α phase) polytype, and no 4H polytype (α phase) can be detected under the exposure of X-ray diffraction. The $\beta \rightarrow \alpha$ transformation of SiC is a common phenomenon in the preparation of SiC aggregates,[6] and is usually accompanied by a microstructure evolution from equiaxed (cubic form) to plate-like or tabular[7] grains (hexagonal form). After hot-pressing, the

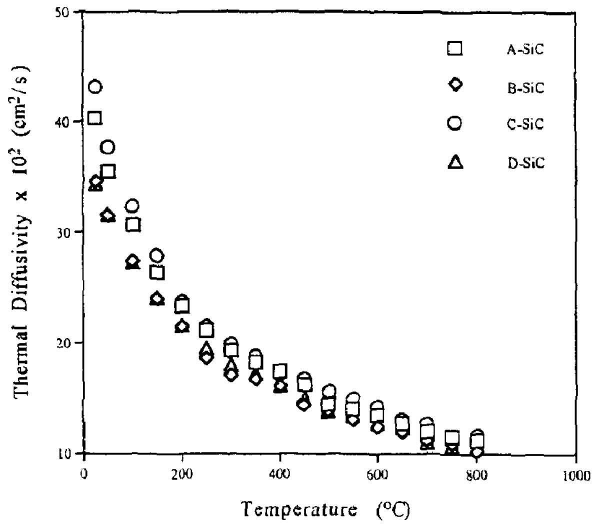

Fig. 2. Temperature dependence of thermal diffusivity of the SiC ceramics.

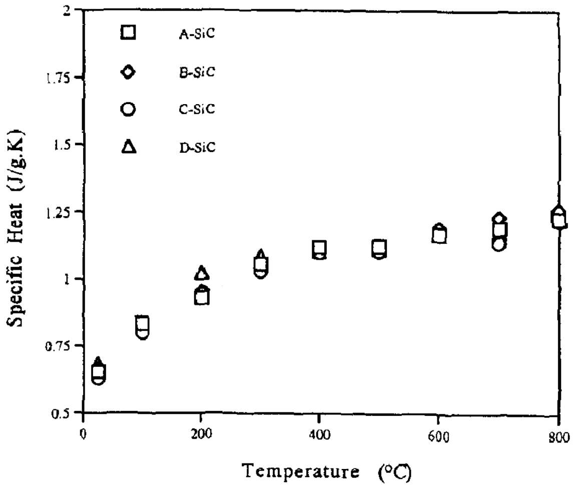

Fig. 3. Specific heat of the SiC aggregates for various temperatures.

XRD analyses revealed that phase transformation has occurred with considerable amounts of 3C polytype being transformed into 4H and 6H polytypes, except that of D-SiC aggregate, where 3C transformed to 4H only.

The microstructures were determined by examining the fractured surface of the aggregates, shown in Fig. 1. Their microstructures are roughly similar, consisting of equiaxed grains with dimensions over the range of ~1–3 μm. However, grain size distribution is apparently different, with at least 50% of grain size, determined topographically, in the range of 2–3 μm for A- and C-SiC, and approximately 30% for the B-SiC, while the rest of the grain sizes are roughly 1 μm or less. Most of the grains are ~1 μm in dimension for the D-SiC. However, no "plate-like" or "tabular" grains arising from hexagonal structure have been found. Since the addition of sufficient amounts of alumina and yttria in the hot-pressing of SiC, liquid phase sintering is expected to occur according to the phase diagram by Noguchi and Mizuno,[17] who indicated that liquid phase formed at temperatures as low as 1800°C in the Al_2O_3–Y_2O_3 system. Accordingly, this appears to be consistent with the concept of liquid phase sintering, which states that grains are separated from each other by the presence of large amounts of the liquid phase during densification. Grain growth was thus inhibited, resulting in a small-grained microstructure, as observed experimentally. Reports on the role of alumina together with yttria in phase transformation of SiC are limited,[18] and will be reported in detail separately.

4.2 Conduction properties

Figure 2 shows the temperature-dependence of thermal diffusivity of different SiC aggregates, the roughly T^{-1}-dependence diffusivity for all the SiC aggregates, suggesting a dominant phonon conduction behaviour, which resembles most polycrystalline materials.[19] A considerable difference, i.e. about 25%, in thermal diffusivity was seen at room temperature with 0·44 cm^2/s for C-SiC and 0·34 cm^2/s for B-SiC; however, the difference became smaller at elevated temperatures, e.g. ~12% at 800°C. This difference in thermal diffusivity may be the result of a number of factors such as impurity, grain size, crystal boundary, porosity, etc., of the aggregates. However, their grain structures, e.g. size and shape, are roughly similar. The pore content is approximately 1–1·3 % in volume determined using a water displacement method. Therefore, the effect of microstructure on the difference in thermal diffusivity for the SiC aggregates is assumed to be negligible, regardless of the consideration of the spatial distribution of pore and pore size effect, which is partly caused by their close analogy in mechanical properties.[20]

Table 2. Impurities content (in weight percent) of starting α- and β-SiC powders

	SiC	SiO$_2$	Al	C	Fe	Cr
α-SiC	~98.6	0.52	0.019	0.84	0.051	—
β-SiC	~98.7	0.09	0.002	—	0.031	0.01

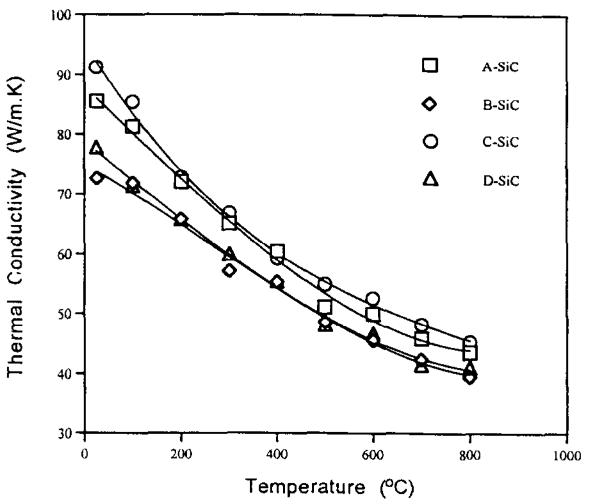

Fig. 4. Temperature dependence of thermal conductivity of the SiC aggregates.

Charvat and Kingery[21] reported that the presence of impurity causes a considerable reduction in thermal conduction properties, even when contamination is below 1%. As a matter of fact, impurities usually have a significant effect on the thermal conduction of highly pure polycrystalline, as well as single-crystal, solids.[22] For the SiC aggregates, considerable amounts of the additives, i.e. 5 wt% Al_2O_3 and 2 wt% Y_2O_3, together with impurities originating from the starting powders (see Table 2 for details), were embedded as a boundary phase within the sintered compacts. Hence, it may be difficult, or impractical, to account for the difference in diffusivity in terms of the impurity content. One of the probable effects arising from experimental findings may result from different ratios of starting α- and β-SiC powders leading to resultant SiC aggregates containing various crystallographic polytypes. This will be discussed later.

The specific heat of the SiC for various temperatures is illustrated in Fig. 3, which shows nearly the same temperature dependence for all types of SiC aggregates. In fact, they are not approximately constant with respect to temperature and do not seem to follow the theoretical expectation.[11] The values of thermal conductivity were thus obtained by multiplying the thermal diffusivity, density and specific heat (reading from the line of best-fit in Fig. 3) according to eqn (4), and are shown in Fig. 4. It is obvious that the diffusivity and the conductivity demonstrated a somewhat large difference in temperature dependence, and this is mainly because of the consideration of specific heat. The C-SiC ceramic exhibits the highest values of thermal conductivity for all temperatures in the study range, and is higher by around 25% and 12% at 25°C and 800°C, respectively, than that of the B-SiC, which exhibits a conductivity minimum between them.

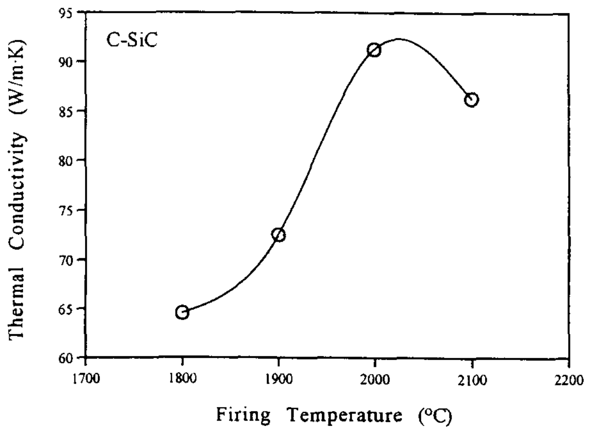

Fig. 5. Effect of firing temperature on thermal conductivity of the C-type SiC ceramic.

4.3 Effect of firing temperature

The effect of firing temperature on thermal conductivity of the SiC aggregate was investigated by selecting the C-SiC ceramic as a representative material because of its maximum value of thermal conductivity. Figure 5 shows that the conductivity increases with increasing firing temperature to a maximum value at ~2000°C, then decreases at 2100°C. The bulk density of the sintered compacts ranges from 97–99·3% of the theoretical density, depending on the firing temperatures. However, it may not cause such a large difference, i.e. ~30%, in conductivity values, reading from values at 1800°C and 2000°C, respectively. In fact, to have a completely satisfactory explanation may be difficult at present. Nevertheless, an attempt from the viewpoint of polytype distribution in the SiC aggregates discussed may provide a new understanding of thermal conduction in SiC ceramics.

4.4 Effect of phase distribution

The actual reason for the difference in thermal conductivity, as well as in diffusivity (Fig. 1), of these SiC aggregates is unclear. However, an attempt is made based on a previous postulation by considering the effect of various distributions of SiC polytypes in the aggregates. Plotting the phase distribution of SiC polytypes against the thermal conductivity of the aggregates, shown in Fig. 6, the thermal conductivity illustrates a strong dependence upon the concentration and types of SiC polytypes at lower temperatures. It is interesting to note that a conductivity minimum was attained when the SiC compact was a mixture of SiC polytypes with sufficient concentrations of

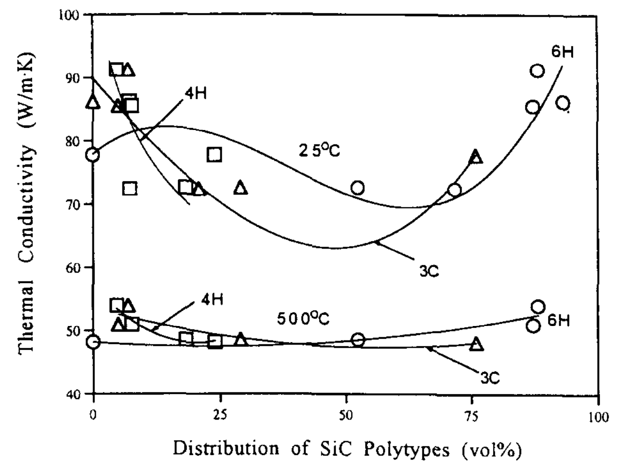

Fig. 6. Effect of phase distribution of SiC polytypes on the thermal conductivity of the SiC aggregates.

each phase. The conductivity increased as one of the polytypes became dominant, especially for SiC with a higher concentration of 6H polytype. Since the conduction properties of most dielectric solids are imperfection-sensitive, the conductivity may be dominated by the content of 4H polytype rather than that of 6H. The conductivity decreased with increasing content of 4H polytype, which is consistent with results reported by Sakai and Aikawa,[10] who assumed that 3C → 4H transformation is promoted by incorporating Al_2O_3 into the 3C-SiC lattice, leading to a solid-solution effect that reduces the thermal conductivity. Therefore, the solid-solution effect is expected to be operative, to some extent, in the aggregates.

Theoretically, a polycrystalline material having a cubic structure is expected to have the highest thermal conductivity because of its high symmetry and order in the crystal lattice. However, the curve in Fig. 6 for 3C polytype with small content appeared to contradict theoretical expectations, i.e. conductivity decreased with increasing content of 3C polytype. This finding is likely to be explainable if the SiC aggregate with pure crystallographic structure is considered as a "homogeneous" compact (under similar considerations of microstructure), the pure structure may be inhomogenized by the incorporation of SiC with other types of structures. This gives rise to inhomogeneity scattering, in addition to the scattering effects, resulting in a decreased mean free path, which as a consequence gives reduced thermal conductivity. The grain boundary scattering should be negligible because the mean free paths for these SiC aggregates were calculated to be ~100 Å at room temperature (to be discussed in the following section), which were significantly smaller than the grain size (about 10,000–30,000 Å) of the SiC aggregates. The difference in thermal conductivity and diffusivity became smaller as temperature increased, suggesting the effect of inhomogeneity scattering was reduced, because of a considerable decrease in mean free path resulting from strong phonon–phonon and phonon–imperfection interactions. Therefore, the dependence of thermal conductivity upon the concentration of SiC polytype was less pronounced, as shown in Fig. 6 with curves of 500°C.

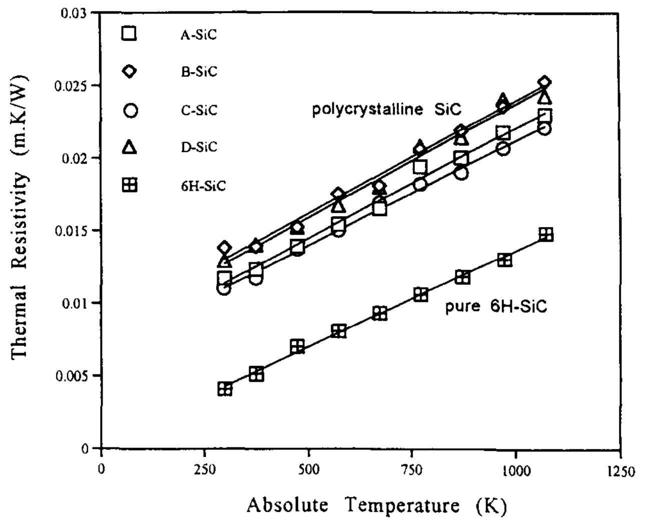

Fig. 7. Dependence of thermal resistivity of the SiC ceramics upon absolute temperature.

4.5 Characteristic of phonon transportation in SiC

Figure 7 shows the dependence of total thermal resistivity of SiC aggregates on the absolute temperature with the data of a highly pure 6H-SiC polycrystalline,[23] obtained by Lely's bulk crystal growth method, for comparison purposes. The thermal resistivity increased linearly with the absolute temperature, which is in excellent agreement with the theoretical expectation that for a dielectric solid above the Debye temperature, the total thermal resistivity (R) can be described by a linear relation:[12]

$$R = aT + b \tag{5}$$

where the slope a is determined solely by lattice conductivity and the intercept b depends on the concentration and the type of imperfections. For an ideal dielectric solid with perfect lattice, above its Debye temperature, the lattice conductivity is the only thermal conduction mechanism and eqn (5) holds with the straight line passing through the origin at absolute zero, i.e. $b = 0$. It is obvious that both the SiC aggregates and pure 6H-SiC follow the theoretical prediction with the approximate values of a, i.e. $1{\cdot}484\times10^{-5}$ m/W. For the

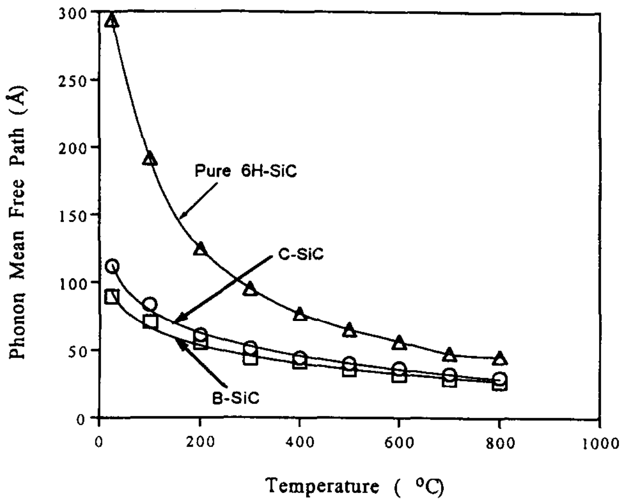

Fig. 8. Temperature dependence of phonon mean free path of the SiC aggregates.

pure 6H-SiC polycrystalline the thermal conductivity is principally dominated by lattice conductivity, with possibly a minor scattering effect by nitrogen within the lattice (so-called n-type impurity)[24] during manufacturing. However, this is not the case for the SiC aggregates, over which considerable deviation from the pure 6H-SiC is strongly related to the imperfections such as impurity, porosity and imhomogeneity effect. The approximately parallel dependence of thermal resistivity between the SiC aggregates and the pure 6H-SiC indicates that the imperfection scattering mentioned previously is temperature independent.

By combining eqns (3) and (4), the phonon mean free path is related directly to thermal diffusivity and elastic-wave velocity by:

$$L = 3\alpha/\nu \qquad (6)$$

where v is roughly constant with respect to temperature[25] and can be obtained by the modulus and density of the solid, i.e. $v=(E/r)^{1/2}$. The elastic-wave velocity in SiC was determined to be $1{\cdot}16\times10^6$ cm/s[26] and is assumed to be similar for all types of SiC employed in the present study. Therefore, the room-temperature mean free path of the pure 6H-SiC was thus determined to be 294 Å, and to be of 104·3 Å, 89·5 Å, 111·7 Å and 88·9 Å, for A-, B-, C- and D-SiC, respectively. By plotting the values of the mean free path of selected SiC solids against temperature, Fig. 8 showed an inverse temperature dependence, which, however, is beyond the theoretical prediction of eqn (1), i.e. $L \propto T^{-1}$, in this temperature range. Hence, it appears to be more general by rewriting eqn (1) as:

$$L \propto T^{-n} \qquad (7)$$

where n is a complex function of imperfection scattering factors for most dielectric solids with real lattice. The value of n increases with the degree of perfection of the solids, i.e. $n=1$ for solids with perfect lattice, and decreases considerably with the increase of the content of imperfections, e.g. $n \sim 0{\cdot}56$ for pure 6H-SiC, suggestive of an n-type impurity effect, and decreased to $\sim 0{\cdot}37$ for the SiC aggregates.

5 CONCLUSIONS

Near fully dense silicon carbide ceramics with equiaxed grain microstructure can be obtained via hot-pressing at 2000°C with sufficient amounts of Al_2O_3, together with Y_2O_3, as sintering aids. The thermal diffusivity and conductivity of the as-sintered SiC aggregates varied considerably with the concentration and type of SiC polytypes present, while the microstructure and impurity effects of the SiC aggregates were very similar. The conductivity of the aggregates decreased with an increasing content of 4H-SiC, which is a result of the solid-solution effect arising from the 3C → 4H transformation. A conductivity minimum was observed for SiC containing sufficient amounts of combined SiC polytypes, however, the conductivity increased at lower content of 4H polytype. It is essential for the improvement of the energy-transfer efficiency depending on working temperature, by controlling the content of constituent SiC polytypes during manufacturing, particularly when SiC ceramic is used as a candidate material for all-ceramic energy-conversion systems.

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