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Thermophysical properties of an amorphous polymer-derived Si/B/N/C ceramic

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ABSTRACT

The thermophysical properties of an amorphous, multinary precursor derived Si/B/N/C ceramic with the approximate composition of $SiBN_3C$ were determined experimentally. Thermal diffusivity $a(T)$ was determined from 77 to 300 K with an alternative laser-flash setup using the resistance change of a thin (circa 100 nm) gold strip as fast temperature probe. From 300 to 873 K a standard laser-flash setup was used. Specific heat capacity $c_p(T)$ was determined by temperature modulated differential scanning calorimetry (MDSC) in the temperature range between 190 and 773 K. Using the relation $\lambda(T) = a(T) \cdot c_p(T) \cdot \rho$ with the specimen density of $\rho = (1561 \pm 47)\,kg\,m^{-3}$, which was considered to be temperature-independent, the thermal conductivity $\lambda(T)$ was calculated. The analysis yielded values for the thermal conductivity at room temperature of $\lambda(296\,K) = (0.490 \pm 0.042)\,W\,m^{-1}\,K^{-1}$, and at the highest investigated temperature of $\lambda(773\,K) = (0.706 \pm 0.063)\,W\,m^{-1}\,K^{-1}$. Furthermore, space-resolved laser-flash measurements with a lateral resolution of about 1.5 mm were performed at 300 K to examine local inhomogeneities on the specimen. Within the given measurement uncertainties, no inhomogeneities could be found.

1. Introduction

Newly developed amorphous high-performance Si/B/N/C ceramics synthesized from molecular or polymer precursors are highly promising candidates for high temperature thermal barrier layers or for fiber-reinforced ceramic composites used for example in turbines to enable higher operating temperatures and therefore higher efficiencies [1]. These amorphous ceramics show extraordinary features: $SiBN_3C$, for example, is the non-oxide ceramic which is most stable regarding oxidation as it stays amorphous up to 1900 K in air and up to 2200 K in inert atmosphere [2]. Some other outstanding properties are a very low density, a small thermal expansion coefficient, a high thermal shock resistivity and a good mechanical performance such as creep resistance even at high temperatures. Thus, there is a wide spectrum for potential applications for these materials. However, the fabrication of Si/B/N/C fibers, for example, is the hitherto most advanced project [2,3].

Amorphous Si/B/N/C ceramics are based on single-source precursors, for example trichlorosilylaminodichloroborane (TADB). TADB is frequently used as it can be produced easily using low cost raw materials, which can be reused or recycled, and is easy to synthesize [1]. To produce an amorphous Si/B/N/C ceramic as the one examined in this work, the preceramic polymer N-methylpolyborosilazane is synthesized out of TADB and methylamine CH_3NH_2. This polymer is then pyrolysed under argon atmosphere at 1400 °C forming a ceramic with the approximate stoichiometric composition $SiBN_{2.5}C_{1.2}$, which is labeled $SiBN_3C$ [3].

For amorphous Si/B/N/C ceramics there are only a few values for thermophysical properties, like thermal diffusivity, specific heat capacity and thermal conductivity, available in literature. For example, the thermal conductivity of a TADB-derived ceramic fiber manufactured by Bayer AG was given in Ref. [2] as $0.4\,W\,m^{-1}\,K^{-1}$ at 1800 K, in Ref. [4] as $(1.1 \pm 0.3)\,W\,m^{-1}\,K^{-1}$ at room temperature and in Ref. [1] as $3\,W\,m^{-1}\,K^{-1}$ at 1500 K. For Si/B/N/C ceramics based on a different precursor, some thermophysical values can be found in Ref. [5] where the thermal conductivity was determined to be in the range of 0.7–1.1 $W\,m^{-1}\,K^{-1}$ in the temperature range between 500 and 1400 K. This is in the same order of magnitude as the literature values for the TADB-based Si/B/N/C ceramic. Nevertheless, these data cannot be simply transferred to Si/B/N/C ceramics based on other precursors, as for example TADB, since the chemical composition is quite dissimilar. Therefore it is the objective of this work to experimentally determine reliable values for thermal diffusivity, specific heat capacity and thermal conductivity of synthesized Si/B/N/C ceramic specimens over a wide temperature range from 80 to 900 K. Although Si/B/N/C ceramics are predestined for high temperature applications, low temperature measurements of thermophysical properties are quite interesting as they might give further information about the structure or density of states of the ceramic.

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For the determination of the thermal conductivity of small ceramic specimens the laser flash method and the temperature modulated differential scanning calorimetry are used. The first yields the thermal diffusivity a(T) while the latter yields the specific heat capacity $c_p(T)$. The temperature dependent thermal conductivity is calculated according to:

$$\lambda(T) = a(T) \cdot \rho(T) \cdot c_p(T) \quad (1)$$

with the density $\rho(T)$.

2. Experimentals

2.1. The laser flash method

The flash method is a common method for the determination of the thermal diffusivity a(T) of solids using a short heat pulse which is generated by a laser or a flash lamp [6]. The advantages of this method are a relatively simple sample preparation, a wide temperature range and short measurement times in the order of seconds. Furthermore, only relatively small quantities of the sample material are necessary.

The basic setup of a laser-flash experiment is shown in Fig. 1: the front side of a thin, disk-shaped specimen is heated with a laser pulse and the corresponding temperature rise on the rear side of the specimen is measured as a function of time. From the time-dependent temperature increase ΔT the thermal diffusivity of the investigated specimen is derived by fitting an analytical solution of the corresponding heat conduction problem to the measurement data. In this work an analytical model is used which considers parasitic heat losses [7], finite pulse lengths [8,9] and heat transport by radiation in diathermic media [10].

Usually, the temperature rise is measured contact-free by an infrared (IR) detector. To improve the absorption of the laser pulse energy and the emission of heat radiation, both sides of the sample are coated with a thin layer of graphite [6].

For the laser flash experiments a Nd:YAG laser with a wavelength of 1064 nm, possible pulse lengths between 0.3 and 20 ms and adjustable pulse energies up to 20 J is used. For measurements between 290 and 873 K, the temperature rise at the backside of the specimen is measured by an HgCdTe IR detector (MCT-detector) and the specimen is placed in a tube furnace.

Measurements at temperatures below 290 K are performed in a cryostat surrounded by liquid nitrogen in which the temperature of the specimen can be controlled between 77 and 300 K.

The MCT-detector cannot be used for measurements below 220 K due to its limited spectral responsivity. Thus, for measurements performed in the cryostat, a different temperature detection system introduced by Kogure et al. [11] was adopted. A thin gold strip is sputtered on the back side of the specimen with a Polaron SC 7640 sputter coater and the change of its electrical resistance, which is directly proportional to the temperature change at the sample backside, is measured as a function of time using a Wheatstone bridge. Since only the relative temperature rise has to be measured in laser flash experiments for determination of the thermal diffusivity, neither the absolute resistance nor the temperature coefficient of the gold strip have to be known. A comparison of this modified detection method with the standard infrared detection system shows that for a BK 7 glass specimen at 300 K an excellent agreement within a range of 3% for the derived thermal diffusivity values can be achieved.

A detailed discussion of this detection method and the description of the low temperature setup is given in Ref. [12].

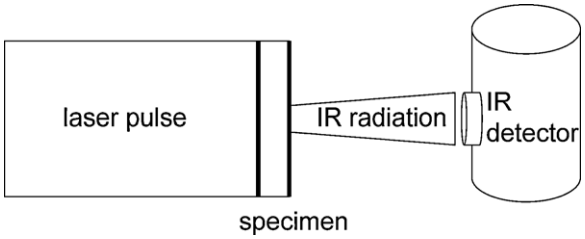

Fig. 1. Schematic depiction of the laser flash instrument.

2.2. Differential scanning calorimetry

A well-known method for the determination of the specific heat capacity c_p(T) of solids is the differential scanning calorimetry (DSC). During this procedure, the specimen and a reference specimen with known heat capacity are successively placed in a crucible and exposed to a temperature regime with constant heating and cooling rates. The temperature difference between this crucible and an empty identical crucible exposed to the same temperature regime is measured as a function of time. The specific heat capacity of the examined specimen can be obtained by calibration with the reference specimen as the temperature difference can be correlated directly to a heat flow. Besides the specific heat capacity of the specimen, some other attributes as the melting temperature or enthalpy of phase transitions can be determined [13–15].

In this work the temperature modulated differential scanning calorimetry (MDSC) is used. Here, the heating rate is composed of a linear and a sinusoidally oscillating portion so that the resulting heat flow can be parted into its reversible and irreversible parts. Advantages of the MDSC method over DSC measurements are for example a higher resolution and the possibility to make quasi-isothermal measurements [13].

The apparatus used for the MDSC measurements in the examined temperature range is a TA Instruments Q2000, which can be used for determination of specific heat capacities between 190 and 800 K.

2.3. Specimen

A disk-shaped TADB-derived Si/B/N/C specimen with a diameter of 9.6 mm and a thickness 0.830 mm was prepared. The mass of the specimen was determined by weighting to (0.0938 ± 0.0002) g. The density was calculated to $\rho = (1561 \pm 47)\,\mathrm{kg\,m^{-3}}$. Since the specimen was assumed to be porous to some amount, the specimen density was additionally measured by pycnometry which yields a bulk density value of $\rho_{\mathrm{Bulk}} = (1830 \pm 92)\,\mathrm{kg\,m^{-3}}$ and therefore a porosity of the specimen of 15%. Fig. 2 shows a picture of the specimen taken by scanning electron microscopy (SEM). Some cracks

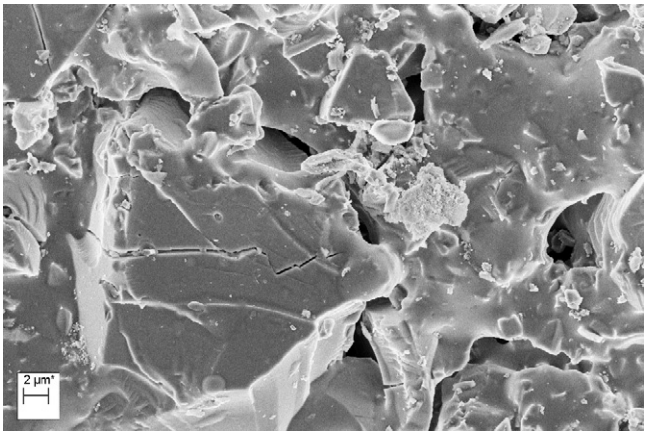

Fig. 2. Picture of the specimen derived by scanning electron microscopy.

which are approximately several 100 nm wide and some pores with sizes of several 10 μm can be identified. It can be clearly seen that the pore distribution is quite irregular.

For laser flash measurements in the temperature range between 290 and 873 K, both surfaces were coated with a thin layer of graphite. After these measurements, the specimen was heated to 1300 K in air to remove the graphite coating. For measurements in the cryostat between 80 and 300 K the back side of the specimen was coated with an approximately 300 nm thin layer of crystalline silicon dioxide by dip coating to fill the pores on the surface and thus enable the sputtering of the gold strip onto the smooth backside surface. The gold strip that was applied is 300 μm wide, 5 mm long, circa 100 nm thick and has an electrical resistance of 175 Ω.

For specific heat capacity measurements by MDSC, a small specimen with a mass of (41.530 ± 0.004) mg was used.

3. Results

3.1. Thermal diffusivity

The thermal diffusivity of the TADB-derived Si/B/N/C specimen was determined in the temperature range between 80 and 873 K using the laser flash method. Fig. 3 shows the experimentally determined values as function of the temperature. Measurements above 295 K were performed using a laser pulse with a length of 2 ms and the MCT-detector. The measurements below 295 K were performed in the LN_2-cryostat under nitrogen atmosphere using a laser pulse length of 0.3 ms and detection of the temperature rise via gold strip.

The relative uncertainty of the thermal diffusivity values determined by the laser flash method amounts to 5% according to GUM standard [16]. A significant influence on the determined thermal diffusivity due to the temperature rise of the specimen during the laser flash measurements caused by the laser pulse can be neglected as the emitted pulse energies are in the range of only 0.5–1.5 J. Only a fraction of this energy contributes to the heating of the specimen as there are energy losses due to the optical equipment, for example mirrors and windows, and radiative heat losses as the specimen reemits the absorbed energy instantly.

In order to investigate the possible influence of the additional SiO_2 layer on the laser flash measurement, the thermal diffusivity of the SiO_2-coated specimen was determined using the MCT-detector at 300 K and compared to the thermal diffusivity value of the specimen determined prior to the preparation of the SiO_2 layer. The resulting difference was less than 1% which shows that the influence of the silicone dioxide layer is negligible.

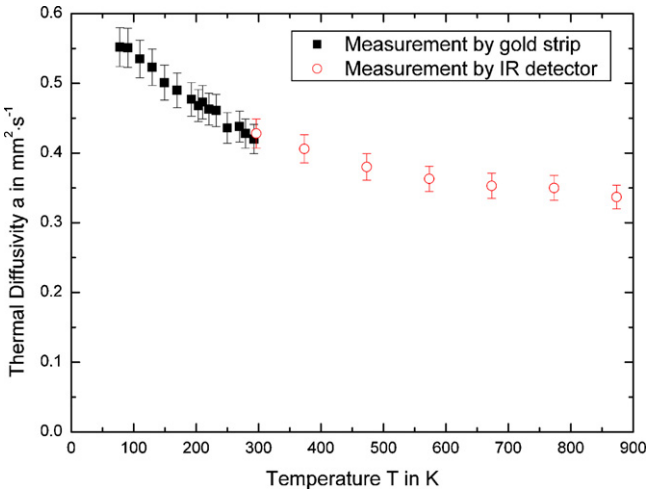

Fig. 3. Thermal diffusivity determined by laser-flash method in the temperature range between 77 and 873 K. The temperature rise at the back side of the sample was measured by a MCT-detector and by measurement of the temperature-dependant resistance change of a thin gold film, respectively.

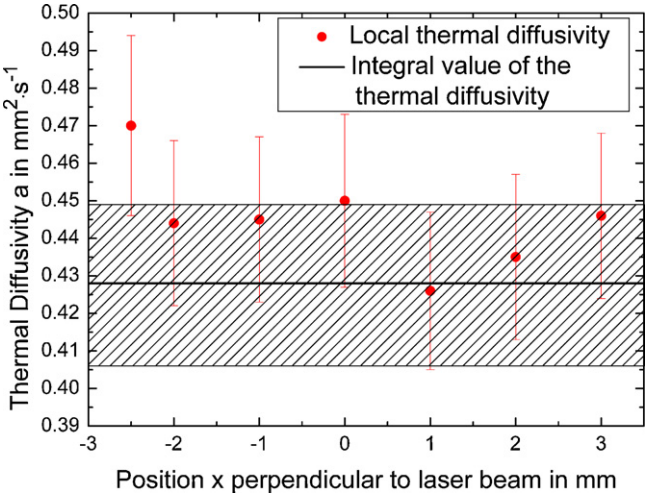

Fig. 4. Measurement of local thermal diffusivity using a space-resolved laser-flash setup. At the position "$x = 0$ mm", the specimen is centered at the optical axis and can be moved perpendicular to the laser-beam. The continuous line and the hatched box indicate the thermal diffusivity with its uncertainty determined with the standard laser-flash setup where a large part of the specimen contributes to the measurement result.

To examine the specimen for local inhomogeneities, space-resolved measurements of the thermal diffusivity were made at room temperature with an experimental setup as it was reported by Hemberger et al. [17]. A polycrystalline IR fiber consisting of AgCl (core) and AgBr (clad) was used to transfer the infrared radiation from the back of the sample to the MCT-detector. The size of the detection area is approximately 1.5 mm in diameter. At the position $x = 0$ mm, the specimen is centered on the optical axis and can be moved perpendicular to the laser beam by two stepping motors. Fig. 4 shows the determined values for the local thermal diffusivity parallel to the gold strip as well as the integral value of the thermal diffusivity that was obtained by measuring thermal diffusivity using the non space-resolved method. It can be seen that the local thermal diffusivity matches the previously determined thermal diffusivity which indicates that there are no significant macroscopic inhomogeneities or fluctuations of density within the specimen.

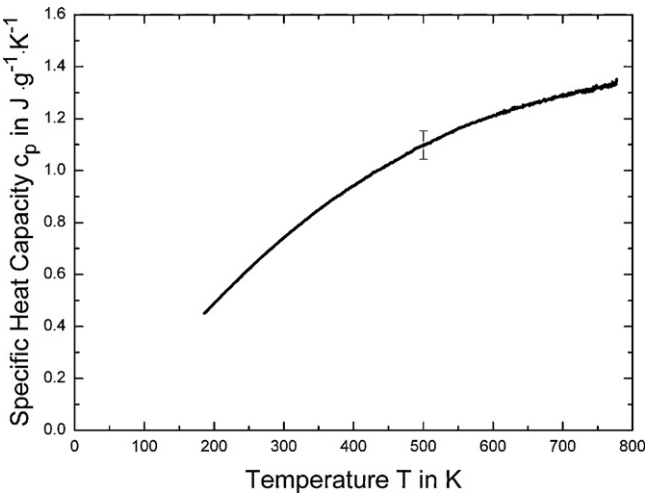

Fig. 5. Temperature dependence of the specific heat capacity c_p of an amorphous Si/B/N/C ceramic measured by MDSC in the temperature range from 190 to 773 K.

3.2. Specific heat capacity

The specific heat capacity c_p(T) of the Si/B/N/C specimen was measured by MDSC from 190 to 773 K in helium atmosphere. The amplitude of the temperature modulation was 0.5 K with a period of 80 s. The modulation was superimposed by a linear heating rate of 1 K min^{-1}. A sapphire specimen was used as reference specimen. Fig. 5 shows the measured values of the specific heat capacity. The relative measurement uncertainty is 5%.

4. Discussion

4.1. Thermal diffusivity and specific heat capacity

The thermal diffusivity decreases slightly from $a = (0.552 \pm 0.028)\,\mathrm{mm^2\,s^{-1}}$ at 80 K to $a = (0.337 \pm 0.012)\,\mathrm{mm^2\,s^{-1}}$ at 873 K, which is a relative decrease of only 64% in a range of nearly 800 K. Compared to crystalline materials, where thermal diffusivity decreases in much larger dimensions, thermal diffusivity of the Si/B/N/C specimen is only weakly temperature-dependent. A detailed description of the underlying physical theory of the thermophysical properties has to be developed in future work.

The specific heat capacity of the amorphous Si/B/N/C ceramic increases from $c_p = (0.461 \pm 0.023)\,\mathrm{J\,g^{-1}\,K^{-1}}$ at 190 K to $c_p = (1.328 \pm 0.066)\,\mathrm{J\,g^{-1}\,K^{-1}}$ at 773 K which is a relative increase of 288%.

4.2. Thermal conductivity

The thermal conductivity of the amorphous Si/B/N/C ceramic was calculated according to Eq. (1) using the experimentally determined values for the specific heat capacity, the thermal diffusivity and the density. The temperature dependence of the density was neglected as the thermal coefficient of expansion is only in the range of some $10^{-6}\,\mathrm{K^{-1}}$ according to Ref. [2] and can therefore be assumed to be already included in the given uncertainty of the density.

Fig. 6 shows the calculated thermal conductivity values in the temperature range between 190 and 773 K. The uncertainties are calculated by rules of error propagation according to GUM [16]. In the examined temperature range, the thermal conductivity increases with increasing temperature by a factor of approximately 2 from $\lambda = (0.348 \pm 0.030)\,\mathrm{W\,m^{-1}\,K^{-1}}$ at 192 K to $\lambda = (0.726 \pm 0.063)\,\mathrm{W\,m^{-1}\,K^{-1}}$ at 773 K. The thermal conductivity values were fitted as a function of temperature T with the polynomial equation of third order

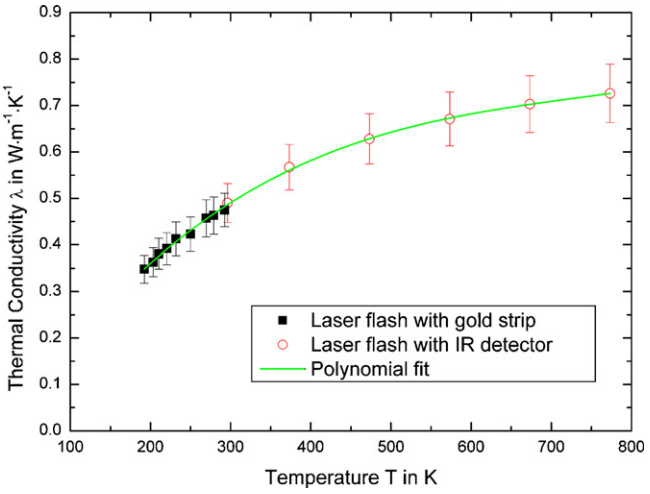

Fig. 6. Thermal conductivity calculated from experimentally determined values of specific heat and thermal diffusivity according to Eq. (1). A polynomial fit according to Eq. (2) was made.

$$\lambda = a + b \cdot T + c \cdot T^2 + d \cdot T^3 \quad (2)$$

where the coefficients are $a = -0.0587\,\mathrm{W\,m^{-1}\,K^{-1}}$, $b = 0.00265\,\mathrm{W\,m^{-1}\,K^{-2}}$, $c = -3.2626 \times 10^{-6}\,\mathrm{W\,m^{-1}\,K^{-3}}$ and $d = 1.4648 \times 10^{-9}\,\mathrm{W\,m^{-1}\,K^{-4}}$.

The increase in thermal conductivity at higher temperatures is characteristic for amorphous substances as opposed to crystalline solids [18]. The temperature dependency of the thermal conductivity is dominated by the temperature dependency of the specific heat capacity.

The experimentally determined values of the specific heat capacity and the thermal diffusivity as well as the calculated values of the thermal conductivity are compiled in Table 1. At 296 K, thermal conductivity of the Si/B/N/C ceramic is $\lambda = (0.490 \pm 0.042)\,\mathrm{W\,m^{-1}\,K^{-1}}$ which is significantly lower than the thermal conductivity of other ceramic materials like YSZ or ceramic fibers [19–21]. For example, the thermal conductivity of yttria-

Table 1
Thermophysical properties of a TADB-derived amorphous Si/B/N/C ceramic with a porosity of 15%.

Temperature, T in K	Thermal diffusivity, a in mm^2 s^{-1}	Specific heat capacity, c_p in J g^{-1} K^{-1}	Thermal conductivity, λ in W m^{-1} K^{-1}
77.8	0.552 ± 0.028	–	–
90.6	0.551 ± 0.028	–	–
110.0	0.535 ± 0.027	–	–
129.6	0.523 ± 0.026	–	–
149.3	0.501 ± 0.025	–	–
169.5	0.490 ± 0.025	–	–
192.4	0.477 ± 0.024	0.467 ± 0.023	0.348 ± 0.027
203.4	0.468 ± 0.023	0.497 ± 0.025	0.363 ± 0.028
210.3	0.473 ± 0.024	0.516 ± 0.026	0.381 ± 0.029
220.3	0.463 ± 0.023	0.543 ± 0.027	0.392 ± 0.030
231.9	0.461 ± 0.023	0.574 ± 0.029	0.413 ± 0.032
249.9	0.436 ± 0.022	0.622 ± 0.031	0.423 ± 0.033
269.5	0.438 ± 0.022	0.669 ± 0.033	0.457 ± 0.035
279.0	0.428 ± 0.021	0.693 ± 0.035	0.463 ± 0.036
292.5	0.420 ± 0.021	0.724 ± 0.036	0.475 ± 0.037
296.2	0.428 ± 0.021	0.733 ± 0.037	0.490 ± 0.038
373.2	0.406 ± 0.020	0.895 ± 0.045	0.567 ± 0.044
473.2	0.380 ± 0.019	1.058 ± 0.053	0.628 ± 0.048
573.2	0.363 ± 0.018	1.184 ± 0.059	0.671 ± 0.052
673.2	0.353 ± 0.018	1.275 ± 0.064	0.703 ± 0.054
773.2	0.350 ± 0.018	1.328 ± 0.066	0.726 ± 0.056
873.2	0.337 ± 0.017	–	–

stabilized zirconia (YSZ), which is the standard material for thermal barrier coatings (TBC), has a thermal conductivity in the range of 1–3 $W m^{-1} K^{-1}$ at 300 K [20]. The thermal conductivities of gadolinium-, samarium- and neodymium-based zirconates are in the same order of magnitude [21]. SiC-fiber types used in fiber-reinforced ceramics provide thermal conductivities in the range of 5–64 $W m^{-1} K^{-1}$ at room temperature [1].

The determined thermal conductivity values do not coincide with any of the values of thermal conductivity of TADB-derived Si/B/N/C fibers given in Refs. [1,2,4]. A reason for this discrepancy could be the irregular porous structure of the examined specimen which leads to a significantly lower thermal conductivity than in nonporous solids. Pores influence the effective thermal conductivity of a sample as they are regimes of much lower thermal conductivity compared to the thermal conductivity of the surrounding solid. However, the thermal conductivity of a porous specimen is not only depending on its degree of porosity, but also on the geometry of the porous structure and the structure of the interconnections between the pores and cracks as these influence strongly the effective thermal conductivity [22]. In the literature, several models considering the influence of porosity on the thermal conductivity can be found [23]. None of these models fit to our measurement results; the thermal conductivity of our specimen is lower than would be expected from the theoretical models for a sample of this degree of porosity. This might be due to the fact that the microstructure of our investigated specimen shows a mixture between irregularly shaped pores and cracks which can hardly be adequately represented by a theoretical model.

Other factors besides the porosity that have an influence on the thermophysical properties might be small differences in chemical composition, the degree of crystallinity, impurities or the morphology of the specimen. The amount of impurities, for example, influences the thermal conductivity as phonons are scattered on impurities which leads to a reduction of thermal diffusivity. If regimes of higher crystallinity existed, the effective thermal conductivity would probably be slightly higher as a higher crystallinity leads to a higher thermal conductivity. However, the influence of these factors on the thermophysical properties is supposed to be much lower compared to the huge effect caused by the porosity, shape of the pores and interconnectivity of the pores.

5. Conclusion

The thermal diffusivity and the specific heat capacity of an amorphous, TADB-derived Si/B/N/C ceramic were determined experimentally by laser-flash and MDSC method, respectively. The obtained values were used to calculate the thermal conductivity in the temperature range between 190 and 773 K. The decrease of the thermal diffusivity with rising temperature is only weak so that a temperature dependent increase of the thermal conductivity is caused by the specific heat capacity. The thermal conductivity increases from $\lambda = (0.348 \pm 0.030)\,W m^{-1} K^{-1}$ at 192 K to $\lambda = (0.726 \pm 0.063)\,W m^{-1} K^{-1}$ at 773 K. Within this temperature range the thermal conductivity of the Si/B/N/C ceramic is lower than the thermal conductivity of TBC materials like YSZ [20] or ceramic fibers [19–21]. The results for the thermal conductivity presented in this work show the high potential for the application of porous Si/B/N/C ceramics as TBCs. Through a systematic modeling of the porosity of the specimen, even lower thermal conductivities might be achieved.

Further systematic investigations on thermophysical properties of Si/B/N/C ceramics are planned as well as measurements on Si/B/N/C ceramics based on other precursors to investigate the influence of the chemical composition on the thermophysical properties. Beyond that, measurements at higher (up to 1800 K) and even lower temperatures down to 20 K will be carried out to provide information about the thermophysical properties of the Si/B/N/C system in an extended temperature range. By using laser-flash calorimetry, the specific heat capacity will be determined down to 20 K which will make it possible to calculate the density of states which is unfortunately not possible with the first measurements on the Si/B/N/C system presented in this manuscript. Additionally, the experimental data will be compared with theoretical predictions of heat transfer in this class of amorphous ceramics.

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