



# Thermophysical Properties of NbAlO<sub>4</sub> and TaAlO<sub>4</sub>

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## Abstract

Thermophysical properties of the ternary oxides NbAlO<sub>4</sub> and TaAlO<sub>4</sub> are experimentally determined. For NbAlO<sub>4</sub>, the molar heat capacity is 98.9 J (mol·K)<sup>-1</sup> at 0 °C up to 155.6 J (mol·K)<sup>-1</sup> at 950 °C and for TaAlO<sub>4</sub> 97.1 J (mol·K)<sup>-1</sup> at 0 °C up to 154.2 J (mol·K)<sup>-1</sup> at 950 °C, respectively. Maier-Kelley polynomials are provided for the molar heat capacities. Thermal diffusivities in the range from 20 °C to 700 °C ( $\alpha_{\text{NbAlO}_4}$ : from 0.009 to 0.004 cm<sup>2</sup>·s<sup>-1</sup> and  $\alpha_{\text{TaAlO}_4}$ : from 0.017 to 0.005 cm<sup>2</sup>·s<sup>-1</sup>), bulk densities at 25 °C ( $\rho_{\text{NbAlO}_4}$  = 3.94 g·cm<sup>-3</sup> and  $\rho_{\text{TaAlO}_4}$  = 6.07 g·cm<sup>-3</sup>) and melting points of the oxides are measured, and the thermal conductivities are calculated from these properties. The thermal conductivity from 20 °C to 700 °C of NbAlO<sub>4</sub>  $\lambda_{\text{NbAlO}_4}$  is in the range from 0.020 to 0.013 W·cm<sup>-1</sup>·K<sup>-1</sup> and of TaAlO<sub>4</sub>  $\lambda_{\text{TaAlO}_4}$  in the range from 0.039 to 0.015 W·cm<sup>-1</sup>·K<sup>-1</sup>, respectively. A porosity correction for thermal conductivities is applied, and with that, data for perfectly dense material are provided.

**Keywords** Calorimetry · Refractories · Specific heat capacity · Thermal conductivity

## 1 Introduction

The mixed oxides niobium aluminate NbAlO<sub>4</sub> and tantalum aluminate TaAlO<sub>4</sub> are of interest for new thermal barrier coatings (TBCs) and environmental barrier coatings (EBCs) [1]. Both have similar thermal expansion coefficients (3.0–5.7·10<sup>-6</sup> K<sup>-1</sup> at 100 °C to 1200 °C [1]) as SiC ceramics (3.2–5.1·10<sup>-6</sup> K<sup>-1</sup> at 0 °C to 1000 °C [2]) and have excellent thermodynamic stability in contact with the surface oxide SiO<sub>2</sub>, which grows on such ceramics in usage [1]. State of the art yttria-stabilized zirconia (YSZ) TBCs have excellent thermophysical properties but are limited to operating temperatures below 1200 °C because of a solid-solid phase transition [1,

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3]. The temperature limits of  $\text{NbAlO}_4$  and  $\text{TaAlO}_4$  seem promising to exceed the limit of YSZ [4–6]. Furthermore, the possible occurrence in refractory metal-alumina composites lead to the investigation of the thermodynamic stability in metal-ceramic composites [5, 7, 8]. Both ternary oxides show no thermodynamic stable phase transition until melting [5]. Metastable phase formation of the tetragonal polymorph of  $\text{TaAlO}_4$  from the melt and solid-solid transitions of the metastable  $\text{TaAlO}_4$  polymorphs (tetragonal and orthorhombic) into the thermodynamically stable crystal structure are reported by Feng et al. [6]. Beside the thermal expansion and thermodynamic stability as first selection criteria, more thermophysical properties of the materials in question are needed to estimate the advantages and disadvantages of a new material. Thermal diffusivity and thermal conductivity are the most important properties to describe the heat transfer in a material or manufactured component.

A set of thermophysical data (measurement of thermal diffusivity and the following calculation of thermal conductivity) was published by Chen et al. [1], but calculated with heat capacity obtained by the Kopp-Neumann rule. This work aims to provide a consistent set of experimentally determined data with  $2\sigma$  uncertainties for thermophysical properties, which are essential for the selection of materials in the development process of new coatings and new use cases in general.

For the system  $\text{Al-Nb-O}$ , a phase diagram of the quasi-binary section  $\text{Al}_2\text{O}_3\text{-Nb}_2\text{O}_5$  was published by Fedorov et al. [4] which was taken as experimental input in the thermodynamic assessment of Gebauer et al. [5]. However, other works give different melting points and in one publication a congruent melting of  $\text{NbAlO}_4$  is proposed [9–11]. A determination with modern equipment to confirm the invariant temperature published by Fedorov et al. [4] is pending in the literature.

For the system  $\text{Al-O-Ta}$ , a fusion diagram was published by King et al. [12]. Detailed information could not be obtained, such as the type of reaction of melting (congruent or incongruent) of the ternary oxide  $\text{TaAlO}_4$ . A preliminary assessment by Gebauer et al. [5] used the fusion data of King et al. as congruent melting of  $\text{TaAlO}_4$ . Later, Feng et al. [6] provided a new assessment based on levitation melt experiments with extreme rapid cooling rates. As undercooling is an issue in oxide systems, the determination of the melting with differential thermal analysis (DTA) is still pending in the literature. The congruent melting modeled by Gebauer et al. [5] has been confirmed by Feng et al. [6].

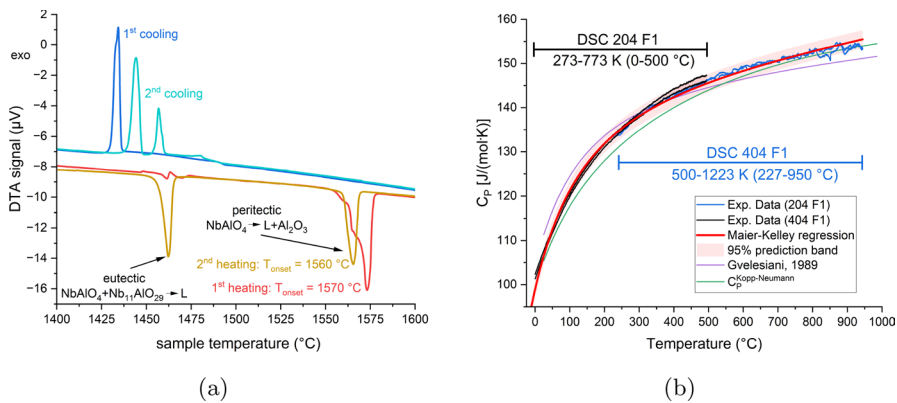
## 2 Materials and Methods

**Materials** Mixed oxide powders were used to prepare the samples by solid-state reactions. The used powders are  $\text{Al}_2\text{O}_3$  TM-DAR from TAIMICRON (99.99 %),  $\text{Ta}_2\text{O}_5$  puratronic® from Alfa Aesar (99.995 %) and  $\text{Nb}_2\text{O}_5$  puratronic® from Thermo Fisher (99.995 %). The starting powders were weighted-in in a molar ratio of 1:1 and mixed in dry state in a DAC 150 speedmixer (Hauschild GmbH) for 10 min and then pressed cold-isostatically (in silicone molds, without any binder, wet-bag method) at 500 MPa into pellets. The pellets for the laser flash analysis (LFA) ( $\approx 12$  mm diameter,  $\approx 2$  mm height) were fired for 72 h at 1350 °C ( $\text{NbAlO}_4$ ) and 1600 °C ( $\text{TaAlO}_4$ ), respectively, in a muffle furnace. Samples for

differential scanning calorimetry (DSC) and DTA were reground and pressed isostatically after the initial synthesis (same as for the LFA samples), and annealed with the same heat treatment again. All samples were analyzed by XRD to verify that they are single phase samples.  $\text{NbAlO}_4$  and  $\text{TaAlO}_4$  were determined to be in the thermodynamically stable monoclinic crystal structure  $\text{C}^2\cdot\text{m}^{-1}$  (space group # 12) after the initial synthesis as well as after the thermophysical measurements.

**Methods** For differential thermal analysis, a Setaram SetSys Evolution was used with a high-temperature graphite furnace, tungsten sample holders (type C thermocouple) and tungsten crucibles. The heating and cooling rates were  $10 \text{ K}\cdot\text{min}^{-1}$ , if not stated otherwise. The shift of crystallization peaks to lower temperatures due to undercooling effects is heavily dependent on the cooling rate, and therefore cooling curves were not evaluated for the determination of melting temperatures. As shown for  $\text{TaAlO}_4$  in this work, the extrapolated onset temperatures of the peaks in the heating signals are independent of the heating rate. As flowing gas, Argon (99.9999 mass% purity) was used. Due to the lack of calibration possibilities with ITS-90 metals (incompatibility with tungsten crucibles), the correction described below was considered as the most valuable and trustworthy step to correlate the measured temperature close to the real temperature. The sample temperature signal of the DTA was corrected with the following equation:  $T_{\text{corr}} = T_{\text{exp}} + f(T_{\text{exp}})$  with  $T_{\text{corr}}$  being the corrected temperature,  $T_{\text{exp}}$  being the uncorrected value measured by the DTA, and  $f(T_{\text{exp}})$  being the linear correction function.  $f(T_{\text{exp}})$  was determined with the measured melting points of  $\text{Al}_2\text{O}_3$ ,  $\text{Ta}_2\text{O}_5$  and  $\text{Nb}_2\text{O}_5$  in comparison to literature data [13–15]. All determined melting temperatures were evaluated as extrapolated peak onset temperatures according to Della Gatta et al. [16].

For differential scanning calorimetry, a Netzsch DSC 204 F1 (up to  $500^\circ\text{C}$ , Al crucibles) and a Netzsch DSC 404 F1 (up to  $950^\circ\text{C}$ , Pt crucibles) were used with heating rates of  $10 \text{ K}\cdot\text{min}^{-1}$ . A  $\tau$ -sensor (type E thermocouple, Netzsch GmbH) was used in the DSC 204 F1 and was temperature calibrated with several reference materials (adamantane, In, Sn, Bi, Zn, CsCl) provided by Netzsch GmbH. A Netzsch CC300 liquid nitrogen cooling unit was used with the DSC 204 F1 to start measurements at  $-20^\circ\text{C}$  to provide reliable data above  $0^\circ\text{C}$ . The DSC 404 F1 was equipped with a type S sensor, which was operated under argon and argon-oxygen (80:20) gas, but no influence of the atmosphere on the heat flow signals could be detected. The temperature calibration was done using Ag and Au measured under argon (with alumina liner inside the Pt crucibles). The samples were sintered pellets to reach the best possible heat conduction. With polished sample surfaces a good contact between sample and crucible was achieved. Each  $C_p$  measurement was done in the three-step method (baseline, reference, sample) according to ASTM E1269-24 [17]. Sapphire standard reference material disks (NIST SRM 720 provided by Netzsch GmbH) were used. All  $C_p$  values given in this work are in Joule per Kelvin and per mole of compound. The uncertainties given with the heat capacity data (Eqs. 1 and 2) and in the according plots (Figs. 1b and 3b) are expanded uncertainties with 95 % confidence (coverage factor  $k = 2$ ).



**Fig. 1** DTA and DSC results for  $\text{NbAlO}_4$ : (a) DTA signals for the incongruent melting of  $\text{NbAlO}_4$ ; (b) experimentally determined heat capacity of  $\text{NbAlO}_4$  compared to Kopp-Neumann and Gvelesiani et al. [22]

LFA measurements were conducted in a Netzsch LFA 427 from 25 °C up to 700 °C. The samples were coated with carbon spray for good laser pulse absorption. At each temperature step, the thermal diffusivity measurement was repeated at least 5 times with two samples. The measured temperature rise signals were evaluated with the Dusza combined model [18] to obtain the thermal diffusivity data. The density of the LFA samples was determined by the Archimedes method, with pure ethanol. The procedure was repeated with three samples of each phase two times. Both ternary oxides crystallize thermodynamically stable in a monoclinic structure, and therefore have a different thermal diffusivity and thermal conductivity for each crystallographic orientation due to the anisotropy. The used samples are polycrystals derived from fine powders without significant texture after firing, and therefore the samples provide an average over all crystallographic orientations. All values for thermophysical properties derived in this work are such average values.

### 3 Results and Discussion

#### 3.1 Sample Characterization

$\text{NbAlO}_4$  samples had a density of  $3.94 \pm 0.02 \text{ g}\cdot\text{cm}^{-3}$  and  $\text{TaAlO}_4$  samples  $6.07 \pm 0.01 \text{ g}\cdot\text{cm}^{-3}$ , respectively. The porosity was calculated from the measured density and the theoretical density (X-ray density from the unit cell) from Chen et al. [1]. For the  $\text{NbAlO}_4$  samples, the porosity is 9.4 % and for the  $\text{TaAlO}_4$  samples 5.5 %, respectively. All samples have less porosity than the maximum recommended value for LFA samples of 10 % according to DIN 18755 [19]. Schlichting et al. [20] analyzed the porosity-dependent thermal conductivity of YSZ (also synthesized from cold-isostatically pressed and sintered powder mixtures) and concluded that the description  $\lambda_{dense} = \lambda_{porous} \cdot (1 - 1.5\phi)^{-1}$ , following theoretical considerations of Maxwell, fits the experimental findings very well.  $\lambda_{dense}$  is the

thermal conductivity of perfectly dense material,  $\lambda_{\text{porous}}$  is the thermal conductivity of the porous sample, and  $\phi$  is the volume fraction of the pores (which is taken as the measured porosity). This correction is also applied in this work and given in the figures for the thermal conductivities.

### 3.2 NbAlO<sub>4</sub>

#### 3.2.1 Melting Point

The ternary oxide NbAlO<sub>4</sub> melts incongruently:  $\text{NbAlO}_4 \leftrightarrow \text{liquid} + \text{Al}_2\text{O}_3$  [4]. The invariant temperature was determined by the extrapolated onset temperatures of the DTA heating signals, shown in Fig. 1a. The cooling curves (shifted by +2  $\mu\text{V}$  for better visualization) showed large undercooling effects, which can be explained by the following solidification path: alumina was precipitated from the liquid, then the alumina-depleted liquid was stabilized below the peritectic temperature while precipitation of NbAlO<sub>4</sub> occurs and finally the liquid solidified at around the temperature of the eutectic reaction  $\text{liquid} \leftrightarrow \text{NbAlO}_4 + \text{Nb}_{11}\text{AlO}_{29}$  (1425 °C [4]). In the second heating, the melting of the eutectic can be observed, which is not detected in the first heating's signal. The first heating segment shows a peak with two shoulders (which could be explained by a non-uniform contact between crucible and sample before first melting) and an onset temperature of the main peak of 1570 °C. The second heating signal shows a peak with an onset temperature of 1560 °C. This temperature matches very well with the experimental findings of Fedorov et al. [4].

#### 3.2.2 Heat Capacity

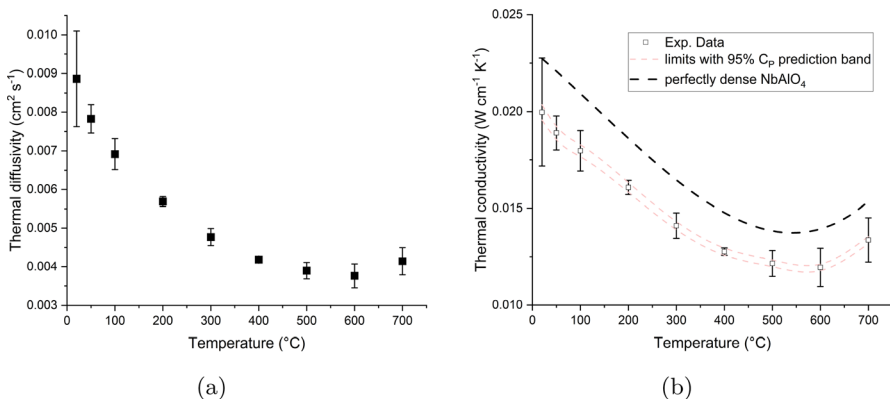
The specific heat capacity of NbAlO<sub>4</sub> was determined by using two different DSCs, operated so that the temperature ranges overlapped. Different samples were used for each DSC. The results are plotted in Fig. 1b. The two different DSC experiments show very similar results in the overlapping range, so that a continuous regression with a Maier-Kelley polynomial was done. The polynomial is given in Eq. 1. For comparison, the heat capacity calculated by the Kopp-Neumann rule based on the binary oxide data from NIST [21] is also given in Fig. 1b as well as the data (calculated from enthalpy increment measurements) from Gvelesiani et al. [22]. The measured values show a very similar behavior between 600 °C and 800 °C as the Kopp-Neumann derived data, but show significant deviation outside that range. The previously published data from Gvelesiani et al. lead to a significantly lower heat capacity at high temperatures.

$$C_p^{\text{NbAlO}_4}(T/K) = 139.87 \pm 0.04 + (0.01469 \pm 4 \cdot 10^{-5}) \cdot (T/K) + (-3354981 \pm 4098) \cdot (T/K)^{-2} \text{J}/(\text{mol} \cdot \text{K}) \quad (1)$$

### 3.2.3 Thermal Diffusivity and Conductivity

The thermal diffusivity data of the LFA measurements are given in Fig. 2a. The thermal diffusivity of  $\text{NbAlO}_4$  ranges from 0.009 to  $0.004 \text{ cm}^2 \cdot \text{s}^{-1}$ . At around  $550^\circ\text{C}$  the thermal diffusivity has a minimum. Above this temperature, the thermal diffusivity increases slightly, in contrast to the expected decrease (which is mainly due to Umklapp-phonon scattering [23, 24]). Caliarì et al. [25] detected a similar upwards trend above  $400^\circ\text{C}$  in their thermal diffusivity measurements of  $\text{Nb}_2\text{O}_5$  coatings. Almadhoni and Khan [26] found a similar trend in several oxides, but at higher temperatures, and stated that the increase is caused by the increasing influence of radiative transport mechanisms through the sample (infra-red radiation) with rising temperatures.

The thermal conductivity of  $\text{NbAlO}_4$   $\lambda_{\text{NbAlO}_4}$  is shown in Fig. 2b. The error bars indicate the errors propagated from the thermal diffusivity and density measurements. The uncertainty based on the heat capacity measurements are entered as light-red dashed lines: if the upper/lower limit of the 95 % prediction band from the  $C_p$  regression polynomial is used for the calculation of the thermal conductivity, the values would be on the light-red dashed lines in Fig. 2b. These lines were added to visualize the uncertainty from the DSC data in comparison to the uncertainty from the LFA measurement. The prediction for perfectly dense  $\text{NbAlO}_4$  is given as black dashed line, which is calculated using the porosity correction given in Sect. 2. The correction of the experimental data leads to  $\approx 16.4\%$  higher values for perfectly dense material than for the porous samples.  $\lambda_{\text{NbAlO}_4}$  is  $0.013$  to  $0.02 \text{ W} \cdot \text{cm}^{-1} \cdot \text{K}^{-1}$ , which is at  $25^\circ\text{C}$  an order of magnitude lower than the thermal conductivity of alumina ( $\approx 0.24 \text{ W} \cdot \text{cm}^{-1} \cdot \text{K}^{-1}$  [27]).  $\text{NbAlO}_4$  has a similar thermal conductivity as  $\text{Nb}_2\text{O}_5$  ( $\approx 0.025 \text{ W} \cdot \text{cm}^{-1} \cdot \text{K}^{-1}$  [25]), but is less thermally conductive than YSZ ( $0.03 \text{ W} \cdot \text{cm}^{-1} \cdot \text{K}^{-1}$  [20])



**Fig. 2** LFA results for  $\text{NbAlO}_4$ : (a) Thermal diffusivity of  $\text{NbAlO}_4$ ; (b) Thermal conductivity of  $\text{NbAlO}_4$ . Boxes: experimentally determined data with light-red dashed limits from the  $C_p$  confidence band; black dashed: thermal conductivity of perfectly dense  $\text{NbAlO}_4$

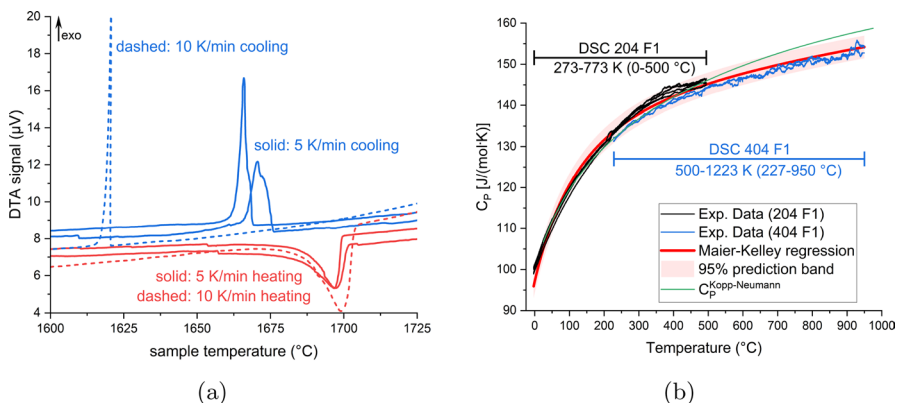
### 3.3 TaAlO<sub>4</sub>

#### 3.3.1 Melting Point

The congruent melting temperature of TaAlO<sub>4</sub> was determined with a heating and cooling rate of 5 K·min<sup>-1</sup> and 10 K·min<sup>-1</sup> to analyze the undercooling effects. No severe undercooling was observed with 5 K·min<sup>-1</sup> cooling rate, but undercooling of around 50 K was detected with a cooling rate of 10 K·min<sup>-1</sup>. As shown in Fig. 3a, the peaks in the heating curves were not shifted between the 5 K·min<sup>-1</sup> and 10 K·min<sup>-1</sup> runs. For visualization, the DTA heat flow signals were shifted by +1.5  $\mu$ V to +10  $\mu$ V without any influence on the onset temperatures. The onset temperature of the melting peaks is determined to be 1685 °C. This result matches well with the fusion diagram of King et al. [12] (1693 °C) where no indication on the type of reaction is given. The result obtained by Feng et al. [6] (1669 °C) from cooling curves is lower, but the shift to a lower temperature can be explained by undercooling effects also found in this work. In this work, no other peak in the DTA signal was found up to 2000 °C and therefore the melting behavior is determined to be congruent.

#### 3.3.2 Heat Capacity

As for NbAlO<sub>4</sub>, the heat capacity for TaAlO<sub>4</sub> was determined by both DSC machines in two different but partially overlapping temperature ranges. The results show good compatibility between both experiments. To our knowledge, no data have been published so far for the ternary oxide. The Maier-Kelley-type regression lead to a polynomial which represents the temperature range from 0 °C up to 950 °C very well. The polynomial is given in Eq. 2. The heat capacity derived by the Kopp-Neumann rule is leading to much higher values at high temperatures as the experimental



**Fig. 3** DTA and DSC results for TaAlO<sub>4</sub>: (a) DTA signals of the congruent melting of TaAlO<sub>4</sub>; (b) heat capacity of TaAlO<sub>4</sub>

results. The Maier-Kelley polynomial gives more reasonable values when extrapolating up to the melting temperature of TaAlO<sub>4</sub>.

$$C_p^{\text{TaAlO}_4}(T/K) = 142.02 \pm 0.04 + (0.01193 \pm 5 \cdot 10^{-5}) \cdot (T/K) + (-3594884 \pm 3114) \cdot (T/K)^{-2} \text{ J/(mol} \cdot \text{K)} \quad (2)$$

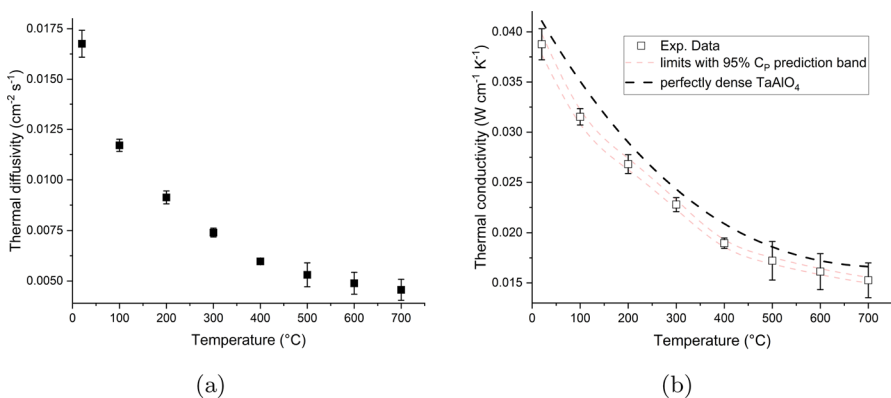
### 3.3.3 Thermal Diffusivity and Conductivity

The results of the thermal diffusivity measurements are shown in Fig. 4a. The values for TaAlO<sub>4</sub> ranges from 0.017 to 0.005 cm<sup>2</sup>·s<sup>-1</sup>. Compared to NbAlO<sub>4</sub>, TaAlO<sub>4</sub> has a higher thermal diffusivity and no increasing behavior at higher temperatures, which is in accordance with the Umklapp-phonon scattering mechanism [23, 24]. Also, the data obtained by LFA for TaAlO<sub>4</sub> have a smaller standard deviation, leading to smaller error bars (compared to Fig. 2a). This can be explained by the smaller porosity of the sample (5.5 % compared to 9.4 % for NbAlO<sub>4</sub>).

The thermal conductivity of TaAlO<sub>4</sub>  $\lambda_{\text{TaAlO}_4}$  is shown in Fig. 4b. Uncertainties and influence of the C<sub>p</sub> 95 % prediction band are included the same as for NbAlO<sub>4</sub> (see Sect. 3.2.3). As the porosity of the sample is only 5.5 %, the correction for perfectly dense TaAlO<sub>4</sub> is only  $\approx 9$  % (increase). The thermal conductivity at 25 °C is 0.039 W·cm<sup>-1</sup>·K<sup>-1</sup> and approximately by a factor of six lower than that of alumina [27], but slightly higher than that of YSZ (0.03 W·cm<sup>-1</sup>·K<sup>-1</sup> [20]).

## 4 Conclusion

The melting points of the two ternary oxides NbAlO<sub>4</sub> (1560 °C) and TaAlO<sub>4</sub> (1685 °C) and the fact that no solid-solid phase transition will occur in the oxides are promising results for the possible usage as thermal barrier coatings. Also, the



**Fig. 4** LFA results for TaAlO<sub>4</sub>: (a) Thermal diffusivity of TaAlO<sub>4</sub>; (b) Thermal conductivity of TaAlO<sub>4</sub>. Boxes: experimentally determined data with light-red dashed limits from the C<sub>p</sub> confidence band; black dashed: thermal conductivity of perfectly dense TaAlO<sub>4</sub>



low thermal diffusivity and thermal conductivity are promising for the usage of both ternary oxides as TBC. The experimental determination of the specific heat is essential, since the Kopp-Neumann prediction deviates more and more from the experimental values with increasing temperature. The given Maier-Kelley polynomials provide a good fit of the experimental data in the measured temperature range and a reliable source for extrapolation of  $C_p$  values to high temperatures.

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**Author Contributions** All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Julian Gebauer. The first draft of the manuscript was written by Julian Gebauer, and all authors commented on previous versions of the manuscript. Hans Jürgen Seifert and Peter Franke were responsible for funding acquisition and supervision. All authors read and approved the final manuscript.

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**Data Availability** No datasets were generated or analysed during the current study.

## Declarations

**Conflict of interest** All authors declare that they have no Conflict of interest.

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