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Thermophysical properties of SmTaO₄, Sm₃TaO₇ and SmTa₃O₉ ceramics

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Abstract

SmTaO₄, Sm₃TaO₇ and SmTa₃O₉ ceramics were prepared via a high temperature solid-state reaction. The results show that the phase structures of the SmTaO₄ ceramics are a monoclinic phase (space group: I2 (5)), the Sm₃TaO₇ ceramics are an ordered orthorhombic phase (space group: Ccmm), and the SmTa₃O₉ ceramics are an orthorhombic phase (space group: Cmmm). Macromolecular and crystal structures of Sm₃TaO₇ and SmTa₃O₉ are more complex than those of SmTaO₄, which can enhance phonon scattering and reduce thermal conductivity. Compared with those of SmTaO₄ and SmTa₃O₉, Sm₃TaO₇ has the smallest grain size, indicating that a small grain size has an effect on strength and ensures material stability. Because of a distorted TaO₆ octahedron and a high concentration of cation vacancy in the lattice of SmTa₃O₉, its thermal conductivity increases with increasing temperature. Compared with that of 7–8 YSZ, Sm₃TaO₇ has a low thermal conductivity (1.3–1.7 W·m⁻¹·K⁻¹ at 100 °C–900 °C), and its minimum thermal conductivity is nearly 30% lower than that of YSZ. The Sm₃TaO₇ ceramics have the highest TECs at approximately 9.82 × 10⁻⁶ K⁻¹ at 1200 °C, which is close to that of 7–8 YSZ (approximately 10 × 10⁻⁶). This indicates that Sm₃TaO₇ has the potential to be employed as a thermal barrier coating.

1. Introduction

Rare earth tantalate has attracted more attention in computed radiography, tomography and fluoroscopy because it is chemically stable, highly dense, and highly transparent in the UV, visible, and near infrared (NIR) regions [1–4]. The rare earth tantalate (YTaO₄) was first proposed by Clarke [5] as a potential thermal barrier coated ceramic material. The good performance of TBCs (Thermal Barrier Coatings) materials is due to a high melting point, excellent high-temperature phase stability, low thermal conductivity, corrosion resistance, good thermal-expansion match with the bond coat, high fracture toughness, and so on. Yttria-stabilised zirconia (7–8YSZ) has been considered the most widely used materials for high-temperature TBCs. To further improve the thermophysical properties and service temperature of YSZ, a large number of researchers have studied its modification by various methods in the past decades, but it is still difficult to improve the application temperature of YSZ. Many refractory materials have been investigated as promising TBCs, such as RE₂Zr₂O₇, RE₂Sn₃O₇, REPO₄, La₃Ce₂O₇, RE₂SiO₃, RE₃Si₂O₇, RE₃(5SiO₄)₃·O₂, ABO₃, LaMgAl₁₁O₁₉ (LHA), and Y₃Al₂Fe₅₋₄O₁₂ [6–11].

The rare earth tantalate ceramics have a high melting point and extremely low thermal conductivity (∼1.0 W·m⁻¹·K⁻¹), high thermal expansion coefficients (TECs, ∼11.0 × 10⁻⁶ K⁻¹), outstanding high-temperature phase stability and excellent mechanical properties; thus, they are widely researched as promising thermal barrier coatings (TBCs). It is believed that their application temperature is as high as 1600 °C. According to a binary phase diagram, the families of rare earth tantalate mainly consist of three kinds of compounds: RETaO₄, RE₃TaO₇ and RETa₃O₉ [12, 13]. RETaO₄ shows great potential in the application of structural materials, especially for thermal barrier coatings, because of its ferroelastic transformation and low thermal conductivity.
The SmTaO₄, Sm₃TaO₇ and SmTa₃O₉ ceramics were synthesised by a solid-state reaction at 1600 °C, and their phase structures were identified by x-ray diffraction (XRD) with a monochromator. The green bodies were compacted under 300 MPa for 5 min by cold isostatic pressing, and then sintered at 1600 °C. The microstructures and thermal properties of these ceramics were evaluated using scanning electron microscopy (SEM) and thermal analysis techniques. The results indicate that these ceramics show potential for use as thermal barrier coatings (TBCs) in high-temperature applications.
where \( \rho \) is the bulk density and \( C_p \) is the heat capacity calculated by the Neumann–Kopp rule. As the fabricated ceramics contain pores, the thermal conductivity of the full-density solid was normalised as follows:

\[
\frac{k'}{k} = 1 - \frac{3 \varphi}{2}
\]

where \( \varphi \) is the porosity determined by the Archimedes technique.

### 2.4. Elastic modulus and thermal expansion

The transverse acoustic velocity \((v_t)\) and longitudinal acoustic velocity \((v_l)\) were measured by a UMS Advanced Ultrasonic Modulus measurement system (TECLAB, France) at room temperature. The average acoustic velocity \((v_m)\) was calculated as follows \cite{28, 29}:

\[
v_m = \left[ \frac{1}{3} \left( \frac{1}{v_t^2} + \frac{1}{v_l^2} \right) \right]^{1/2}
\]

\[
E = \rho \frac{v_t^2 (3v_t^2 - 4v_l^2)}{(v_t^2 - v_l^2)}
\]

\[
B = \rho \left( v_t^2 - \frac{4}{3} v_l^2 \right)
\]

\[
\sigma = \frac{1}{2} - \frac{2(v_t/v_l)^2}{2 - (v_t/v_l)^2}
\]

\[
G = \frac{E}{2(2 + \sigma)}
\]

The thermal expansion rate \((dL/L_0)\) and thermal expansion coefficients (TECs) of the SmTaO4, Sm3TaO7, and SmTa3O9 ceramics were determined by a thermo-mechanical analyser (NETZSCH TMA 402 F3, Germany) from room temperature to 1200 °C. In order to check the repeatability and accuracy of thermal dilatometer, the standard material was used as a standard for calibration of dilatometer. As the equipment (NETZSCH TMA 402 F3, Germany) uses Al2O3 bracket and push rod, the standard sample is Al2O3 provided by NETZSCH. The Al2O3 standard sample has a wide temperature range \((-190 °C–1680 °C)\), high precision and low-cost. Thus, the thermal expansion rate of alumina \((Al_2O_3)\) was used as the baseline for the measurements.

### 2.5. Optical properties

The optical properties were measured using a UV–vis spectrophotometer (UV-3600 plus; Shimadzu, Tokyo, Japan), and BaSO4 powders were used as reference samples. The results of reflectance and absorption were obtained with wavelengths from 200 to 800 nm. The band gap was determined by the Kubelka-Munk method:

\[
F(R) = \frac{(1 - R)^2}{2R}
\]

where \( R \) is the reflectance and \( F(R) \) is proportional to the extinction coefficient \((\varphi)\).

### 3. Results and discussion

#### 3.1. Phase structure and microstructure

Figure 1 shows the XRD patterns of the SmTaO4, Sm3TaO7, and SmTa3O9 ceramics. Compared with that of the standard XRD spectra of defect-fluorite SmTaO4 (JCPDS data card: 24-1010), the phase structure of SmTaO4 ceramics are a monoclinic phase (space group: I2 (5)) with no evidence of impurity. The Ta atom coordinates with four O atoms to form a TaO4 tetrahedron, and the samarium ion and TaO4 tetrahedron form a structure with a coordination number less than eight \cite{19}. The XRD patterns of Sm3TaO7 ceramics are in agreement with the standard XRD spectrum (JCPDS data card: 36-0672), suggesting that the phase structure of Sm3TaO7 ceramics is an ordered orthorhombic phase (space group: Ccmm). For Sm1Ta2O7 ceramics with a magnesite type, each Ta atom coordinates with six O atoms to form a TaO6 octahedron. The crystal structure is composed of the TaO6 octahedron and the SmO7 and SmO8 polyhedrons \cite{30}. Compared with the standard XRD spectra of SmTa2O9 (JCPDS data card: 52-0566), the phase structure of SmTa2O9 is an orthorhombic phase (space group: Cmmm). The crystal structure of SmTa2O9 compounds is that the Sm3+ ionic sites consist of 2/3 Sm3+ ions and 1/3 cation vacancies, which means there is a certain extent of cation vacancies in this structure. Furthermore, Sm3+ and six oxygen ions coordinate to form asymmetrical TaO6 octahedrons \cite{31}. 

\[
\kappa = \alpha \rho C_p
\]
The XRD results demonstrate that the crystal structures of Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ are more complex than that of SmTaO$_4$, which can be explained according to the Clarke model:\[15]\:

$$\kappa_{\text{min}} = \frac{0.87 K_B N_A^{2/3} m^{1/3} \rho^{1/6} E^{1/2}}{M^{2/3}}$$  \(\text{(9)}\)

where $K_B$, $N_A$, $m$, $\rho$, $E$, and $M$ are the Boltzmann constant, the Avogadro constant, the number of atoms in the unit cell, the density of the sample, Young’s modulus, and the atomic mass of the unit cells, respectively. From equation (9), one can see that materials with low thermal conductivity should possess large molecular weights and complex crystal structures. The structures of Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ consist of high atomic masses and complex crystal structures, which means that Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ have lower thermal conductivity\[32\].

Furthermore, there are asymmetrical TaO$_6$ octahedrons that seriously affect the thermophysical properties of Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ compounds. The relationship between the thermophysical properties and the crystal structure of Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ is discussed in detail below.

The SEM images of SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics are shown in figure 2. During sintering, the ceramic particle size is approximately 1–20 $\mu$m, and one can see that the ceramics have a clear grain boundary and better crystallization form figures 2(a)–(f). SmTaO$_4$ has a larger grain size than those of the other two ceramics, with a grain size of approximately 10–20 $\mu$m. Figure 2(d) shows the domain structures of the SmTaO$_4$ ceramics, which may relate to an ordinary grain growth around a screw dislocation. In general, ferroelastic domain structures of YTaO$_4$ and DyTaO$_4$ ceramics can be observed in detail by transmission electron microscopy (TEM)\[22, 23\]. In Sm$_3$TaO$_7$, a small quantity of pores is observed, and the grain size is approximately 1–5 $\mu$m. A small number of pores will reduce the hardness, bending strength and creep resistance. The grain size of Sm$_3$TaO$_7$ is 1–5 $\mu$m with an irregular shape and uneven size. Compared with those of SmTaO$_4$ and Sm$_3$Ta$_3$O$_9$, Sm$_3$TaO$_7$ has the smallest grain size, indicating that a smaller grain size has an effect on the strength and ensures material stability. Thus, it is believed that a decrease in crystal size can produce excellent toughness at high temperature. Table 1 lists the density and porosity of SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics as determined by the Archimedes technique. The relative density of Sm$_3$TaO$_7$ ceramics is as high as 98.68%. A higher density can meet the thermal conductivity research requirements.
3.2. Elastic properties

The transverse acoustic velocity ($v_t$) and longitudinal acoustic velocity ($v_l$) of SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ were measured by a UMS Advanced Ultrasonic Modulus measurement system. The average acoustic velocity ($v_m$) was calculated by equation (3). Table 2 shows the elastic modulus of SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics, and the elastic modulus of Sm$_3$TaO$_7$ ceramics is as high as 205 GP. The results are consistent with those of the SEM analysis. Under normal circumstances, Young’s modulus increases with increasing bond strength, and bond strength usually increases with decreasing bond length. In particular, an ordered atomic arrangement can decrease the crystal lattice energy and increase the stability of the crystal structure. According to a phase characterization, Sm$_3$TaO$_7$ with an ordered orthorhombic phase (space group: Ccmm) has more order and is more stable than those of SmTa$_3$O$_9$ with an orthorhombic phase (space group: Cmmm). Young’s modulus increases with increasing crystal structure stability in Sm$_3$TaO$_7$, which should be the main factor affecting the variation in Young’s modulus.

3.3. Thermophysical properties

Figure 3 shows the thermophysical properties of the SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics. Specific heat capacities of the SmTaO$_4$ and SmTa$_3$O$_9$ and Sm$_3$TaO$_7$ ceramics were calculated by the Neumann-Kopp rule and are shown in figure 3(a). The specific heat increased slowly with increasing temperature, which is attributed to volumetric expansion and phonon excitation. Moreover, the specific heat of Sm$_3$TaO$_7$ is higher than that of SmTaO$_4$ and SmTa$_3$O$_9$, which contributes to a decrease in thermal conductivity.

| Sample | SmTaO$_4$ | Sm$_3$TaO$_7$ | SmTa$_3$O$_9$ |
|--------|-----------|---------------|---------------|
| Theoretical density (g cm$^{-3}$) | 8.53 | 8.03 | 7.63 |
| Measured density (g cm$^{-3}$) | 8.31 | 7.92 | 7.48 |
| Porosity (%) | 2.62 | 1.32 | 1.98 |

| Elastic modulus | SmTaO$_4$ | Sm$_3$TaO$_7$ | SmTa$_3$O$_9$ |
|-----------------|-----------|---------------|---------------|
| Young’s modulus (GPa) | 96 | 205 | 164 |
| Bulk modulus (GPa) | 111 | 174 | 107 |
| Shear modulus (GPa) | 35 | 78 | 66 |

Table 1. Experimental density and porosity of SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$.

Table 2. Elastic modulus of SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$.
Figure 3 depicts the thermal diffusivities of the SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics. The thermal diffusivities of SmTaO$_4$ and Sm$_3$TaO$_7$ decrease with elevated temperature and tend to flatten at high temperature, which obeys a typical $T^{-1}$ rule with temperature. This indicates that the thermal diffusivities of SmTaO$_4$ and Sm$_3$TaO$_7$ undergo nonharmonic scattering. The thermal diffusivity of Sm$_3$TaO$_7$ is lower than that of SmTaO$_4$, mainly because Sm$_3$TaO$_7$ has a large molecular mass and the TaO$_6$ octahedron can enhance phonon scattering. The thermal diffusivity of SmTa$_3$O$_9$ increases with increasing temperature, as shown in figure 3(b).

According to related reports [33], one can see that the different concentrations of cationic vacancy were introduced at the A-site, and the thermal diffusivities of SrTiO$_3$ gradually transform from $k \propto T^{-1}$ to glass-like with a change of temperature. Compared with the concentration of the A-site vacancy in SrTiO$_3$, the A-site vacancy concentration of Sm$_3$TaO$_7$ is as high as 66.7%. The high concentration of A-site vacancies can produce a low energy ‘rattling’ vibrational mode, leading to glassy thermal diffusivities in a crystalline material. Furthermore, ultraviolet light analysis shows that SmTa$_3$O$_9$ has an obvious thermal radiation effect at high temperature, which is one of the reasons why its thermal diffusivities increase with increasing temperature. When the temperature is below 600 °C, SmTa$_3$O$_9$ has the lowest thermal diffusivities compared to those of the SmTaO$_4$ and SmTa$_3$O$_9$ ceramics. The main reason is that SmTa$_3$O$_9$ ceramics have a large molecular mass and a distorted TaO$_6$ octahedron, which can enhance phonon scattering and reduce thermal diffusivities.

Thermal conductivities of the SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics were obtained by multiplying the thermal diffusivities ($\alpha$), specific heat ($C_p$) and density ($\rho$) according to equation (1). The thermal conductivity curve is obtained by equation (2), as shown in figure 3(c), and the variation of thermal conductivity and thermal diffusivity are similar. The plots of 7–8 YSZ are also included for comparison [34]. This implies that SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ may have lower thermal conductivity than that of 7–8YSZ at the current test temperature. Compared with the thermal conductivity of the industrially applied 7–8YSZ thermal barrier coating material (2.1–2.7 W·m$^{-1}$·K$^{-1}$, 25 °C–900 °C), the low thermal conductivities of Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics are approximately 30% lower. The lower thermal conductivities of the Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics are related to their complex crystal structure. The complex crystal structure and disordered atom arrangement increases the anharmonic lattice vibration, strengthens the phonon scattering and decreases the thermal conductivity.
conducted via lattice vibrations. The thermal conductivity is calculated as follows: 

\[ k_{\text{min}} = \frac{\rho C_v}{\alpha} \]

where \( \rho \) is the density, \( C_v \) is the heat capacity of the material, \( \alpha \) is the thermal diffusivity, and \( k_{\text{min}} \) is the minimum thermal conductivity. There are several theories that have been reported about the heat conduction of lattice waves in a solid, and the thermal energy is derived from the strong anharmonic vibration between phonons, which is mainly characterized by the Grüneisen constant \( \gamma \), the Debye temperature \( TD \) and the minimum thermal conductivity \( k_{\text{min}} \):

\[ \gamma = \frac{3}{2} \left( \frac{1 + \sigma}{2 - 3 \sigma} \right) \]

where \( \sigma \) is the Poisson ratio.

The variation of the phonon mean free path is consistent with the trend of thermal diffusivity and thermal conductivity. There are several ceramic calculated by the Clarke modes are as low as 0.93 W m\(^{-1}\)K\(^{-1}\) at 1200 °C, which is close to that of 7–8 YSZ (approximately 10 \times 10\(^{-6}\)). The TECs of the SmTaO\(_4\) and SmTa\(_3\)O\(_9\) ceramics are lower than that of 7–8 YSZ. In light of the low thermal conductivity, high TECs, excellent phase stability, the Sm\(_3\)TaO\(_7\) ceramics are promising candidates as thermal barrier coating materials.

### Table 3. Poisson ratio (\( \sigma \)), Grüneisen constant (\( \gamma \)), Debye temperature (\( TD \)) and minimum thermal conductivity (\( k_{\text{min}} \)) of SmTaO\(_4\), Sm\(_3\)TaO\(_7\), and SmTa\(_3\)O\(_9\).

| Elastic modulus | SmTaO\(_4\) | Sm\(_3\)TaO\(_7\) | SmTa\(_3\)O\(_9\) |
|----------------|------------|-----------------|-----------------|
| Poisson ratio (\( \sigma \)) | 0.36 | 0.30 | 0.25 |
| Grüneisen constant (\( \gamma \)) | 2.18 | 1.80 | 1.48 |
| Debye temperature (\( TD \)) | 467 | 412 | 382 |
| the minimum thermal conductivity (\( k_{\text{min}} \)) | 1.24 | 1.08 | 0.93 |
3.4. Optical properties

Figure 5 (a) shows that the reflectance of the SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics increases with wavelength. The reflectance reduces suddenly at a certain wavelength due to the $4f^5$ transition in the $4f$ shell of samarium. The shapes and positions of these peaks are consistent with those of Sm$_2$O$_3$ prepared by Jack L. Ryan. SmTa$_3$O$_9$ has the highest reflectivity, and Sm$_3$TaO$_7$ has the lowest reflectivity. Figure 5 (b) shows that the absorption of the SmTaO$_4$, Sm$_3$TaO$_7$ and SmTa$_3$O$_9$ ceramics decreases with increasing wavelength and that SmTa$_3$O$_9$ exhibits the lowest absorption. When the wavelength is greater than 500 nm, the absorption rate of
SmTa2O6 is almost zero. According to a previous analysis of the thermal diffusion coefficient of SmTa2O6, the thermal diffusion coefficient of SmTa2O6 increases with increasing temperature, which is caused by the thermal radiation effect of SmTa2O6.

Figure 5(c) shows the band gaps calculated by the Kubelka-Munk formula. The band gaps of the SmTaO4, Sm5TaO2, and SmTa3O6 ceramics are in the range of 4.1–5.0 eV and decrease with decreasing molecular mass. The band gap of each sample corresponds to the gap between the valence band formed by O 2p and the conduction band formed by Ta 5d. The band gap values of the ceramics are consistent with the results reported by Masato Machida [36], as it closely relates to the energy level of the 4f shell in the electronic structure. Thus, the SmTa2O6 ceramic sample exhibited a narrower band gap (4.1 eV) than those of the other specimens. Therefore, it is inferred that heat is conducted by phonons in ceramics, and the effect of the carrier on thermal conduction can be omitted.

4. Summary

SmTaO4, Sm5TaO2, and SmTa3O6 ceramics were prepared via a high-temperature solid-state reaction. The results show that the phase structures of the SmTaO4 ceramics are a monoclinic phase (space group: I2 (5)), the Sm5TaO2 ceramics are an ordered orthorhombic phase (space group: Ccmm), and the SmTa3O6 ceramics are an orthorhombic phase (space group: Cmmm). The crystal structures of Sm5TaO2 and SmTa3O6 are more complex than that of SmTaO4. Macromolecular mass and complex crystal structure can enhance phonon scattering and reduce thermal diffusion. Compared with those of SmTaO4 and SmTa3O6, SmTa2O has the smallest grain size, and it is believed that a small crystal size can produce excellent toughness at high temperature. SmTaO4 has a low thermal expansion coefficient (9.82 × 10−6 K−1, 1200 °C) and thermal shrinkage at low temperature, but has poor comprehensive performance and is not suitable for use as a thermal barrier coating material. SmTa2O has the smallest grain size and a high Young’s modulus and Vickers hardness, which can reduce the coating failure. SmTaO4 has a low thermal conductivity (1.3–1.7 W·m−1·K−1 at 100 °C–900 °C), a high coefficient of thermal expansion (9.82 × 10−6 K−1 at 1200 °C), and significant thermal radiation effects. Compared with that of 7–8 YSZ, the minimum thermal conductivity of SmTaO4 is nearly 30% lower. This indicates that SmTa2O has the potential to be employed as a thermal barrier coating.

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