An effective strategy for preparing transparent ceramics using nanorod powders based on pressure-assisted particle fracture and rearrangement

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Abstract: Achieving full densification of some ceramic materials, such as Y\textsubscript{2}O\textsubscript{3}, without sintering aids by spark plasma sintering (SPS) is a great challenge when plastic deformation contributes limitedly to the densification as the yield stress of the material at an elevated temperature is higher than the applied sintering pressure. Herein, we demonstrate that particle fracture and rearrangement is an effective strategy to promote the densification during the pressure-assisted sintering process. Specifically, Y\textsubscript{2}O\textsubscript{3} nanocrystalline powders composed of nanorod and near-spherical particles were synthesized and sintered at various temperatures by the SPS. The results show that the relative density of the ceramics prepared by the nanorod powders is higher than the density of the ceramics from the near-spherical powders after 600 °C due to the fracture and rearrangement of the nanorods at low temperatures, which leads to the decrease of particle size and the increase of density and homogeneity. Based on this novel densification mechanism, ultrafine-grained Y\textsubscript{2}O\textsubscript{3} transparent ceramics with good optical and mechanical properties were fabricated successfully from the nanorod powders.

Keywords: transparent ceramics; nanorods; fracture and rearrangement; Y\textsubscript{2}O\textsubscript{3}; spark plasma sintering (SPS)
1 Introduction

Transparent ceramics can find a wide range of applications, such as transparent armors, infrared (IR) domes/windows, host materials for fluorescence application, and laser hosts, on account of their processing flexibility in fabricating large-sized or complex-shaped products [1–8]. In most cases, the preparation process of any ceramics is sintering consolidated/packed powders at high temperatures [9,10]. Therefore, the characteristics of the powders, including chemical purity, particle size, and particle morphology, significantly influence the microstructures and performances of the transparent ceramics [9].

As highly transparent ceramics require full density, well-reputed wisdom believes that the initial powders with high sintering activity for obtaining the highly transparent ceramics have some important characteristics including small particle size, narrow size distribution, spherical/near-spherical shape, and low-degree agglomeration [9]. References [11–13] have shown that the ceramic powders with small-sized particles have large specific surface areas and high sintering activity. Flat surfaces from non-spherical powders will have a lower driving force for sintering due to the lack of curvature [14]. Besides, mono-dispersed powders can avoid large pores between agglomerates and form crack-like voids in the final ceramics [15]. Thus, many types of research for preparing the nano ceramic powders with mono-dispersed particle size distribution and spherical particle morphology have been carried out [15–18]. However, there are still many difficulties with these aforementioned desirable characteristics [9,15].

Recently, large ceramic particles can be fractured and rearranged during high-pressure-assisted sintering, which provides a viable route for fabricating fully dense ceramics even using anisotropic-shaped powders [19,20]. For example, with the aid of a high pressure (5 GPa), Y2O3 nanorod powder compact can achieve near full density through particle fracture, rearrangement, deformation, and interface sliding at only 500 °C without atomic diffusion [20]. Furthermore, the grain size of the sintered Y2O3 ceramics is smaller than the initial particle size. However, most sintering methods, such as spark plasma sintering (SPS), vacuum sintering, and hot isostatic pressure (HIP) sintering cannot provide such a high pressure [1,9]. In addition, it is still an unrevealed controversy whether the anisotropic-shaped nanorod powders can obtain the fully dense ceramics using these conventional sintering methods with dominated densification mechanisms of the atomic diffusion. If they can prepare the transparent ceramics, is there a sintering mechanism different from that of spherical/near-spherical powders?

To address these questions, we have tried to use a solvothermal method to synthesize the Y2O3 nanocrystalline powders with singularly-shaped nanoparticles. As a comparison for checking sintering efficiency, near-spherical Y2O3 nanocrystalline powders were also synthesized by a co-precipitation method. Then, the SPS was employed to sinter ceramics at various temperatures from 200 to 1500 °C. The microstructures of these powders, as well as the relative densities, microstructures, and grain sizes of the sintered ceramics, were analyzed. The sintering efficiency and densification mechanism of the nanorod powders were investigated. Furthermore, the optical and mechanical properties of the Y2O3 transparent ceramics obtained at the optimized sintering temperature were analyzed.

2 Materials and methods

Herein, the Y2O3 nanorod powders were synthesized by the solvothermal method. Y(NO3)3·6H2O (> 99.99%, Yutai Qixin Chemical Co., Ltd., China) was dissolved in deionized water to a concentration of 0.1 mol/L. Then, the solution was added drop by drop into a diluted ammonia solution (0.5 mol/L). The mixed solution was stirred for 6 h and aged for 20 h to get a gel-like precipitate. After that, the precipitate was washed with deionized water and ethanol in turn, several times by a centrifugal machine. Near-spherical powders were fabricated by directly drying the washed precipitate, and the precursor powders were sieved, and calcined at 800 °C for 4 h. To prepare singularly-shaped nanocrystalline powders, the washed precipitate was subsequently diluted by ethanol and placed into a Teflon reactor to react at 200 °C for 24 h. The reactants were washed centrifugally with ethanol, and then dried at 60 °C. Finally, the as-obtained precursor powders were sieved by a 200-mesh screen and calcined at 800 °C for 4 h to remove the adsorbed water and bound water.

The as-prepared powders were placed into a graphite die with an inner diameter of 10 mm. The densification processes of the samples were carried out in an SPS system (LABOX-325, SINTER LAND, Japan) with an
ambient pressure of 6 Pa. A thin carbon foil with a thickness of 0.2 mm was inserted between the punches and the inner die wall to maintain good thermal and electric contacts. The samples were sintered at a temperature range of 200–1500 °C with a ramping and cooling rate of 20 °C/min and held for 5 min at the final sintering temperatures. The temperature range during the sintering processes from ~20 to 600 °C was monitored by a thermocouple. Further temperature range (600–1500 °C) was regulated and controlled by an IR thermometer focused on a small hole located at the graphite die surface. A uniaxial pressure of 50 MPa was applied to the Y$_2$O$_3$ samples before sintering and released gradually to 0.1 MPa to reduce thermomechanical stress after the dwelling process as the desired sintering was finished. As the high sintering temperatures ($\geq 1200$ °C) lead to black-colored Y$_2$O$_3$ ceramics caused by the carbon contamination from the carbon foil, post-annealing of the ceramic samples SPSed at $\geq 1200$ °C was conducted at 1100 °C for 8 h in air using a muffle furnace to eliminate the carbon contamination. The as-obtained samples were mirror polished on both two sides for in-line transmittance and Vickers hardness tests. The thermally etched surfaces were obtained by annealing the polished ceramics at a temperature of 50 °C lower than the sintering temperature for 30 min.

The crystal structures of the as-synthesized powders were investigated by X-ray diffraction (XRD) using an X-ray diffractometer (D8 Advance, Bruker, Germany). The XRD data were recorded over the 2θ range of 10°–70° with a step size of 0.02°. The high-magnified microstructural images of the powders and ceramics were checked by a transmission electron microscope (TEM; F200, JOEL, Japan). The ceramic sample for the TEM test was prepared by a “lift-out” method using focused ion beam (FIB). The thermally etched surfaces of the ceramics sintered at different temperatures were investigated by a scanning electron microscope (SEM; JIB-4700F, JOEL, Japan). The in-line transmittance test was performed on an ultraviolet–visible–near infrared (UV–Vis–NIR) spectrophotometer (Lambda 750, PerkinElmer, USA). The Vickers hardness tests were operated by a dynamic ultra-microhardness tester (DUC-21ls, Shimadzu, Japan) at an applied load of 0.5 N for 15 s. At least ten measurements were carried out on each sample.

3 Results and discussion

3.1 Powder characterization

Figure 1(a) shows the XRD pattern of the synthesized Y$_2$O$_3$ powders by the solvothermal method after calcined at 800 °C. The pattern is well-indexed to the Y$_2$O$_3$ phase (PDF Card No. 43-1036). Broadening diffraction peaks indicate that the crystal size of the powders may be in the nanoscale. After the whole pattern fitting on the XRD data by Jade software (fitting error $< 10\%$), the average crystallite sizes of the calcined powders are calculated from the peak broadening of the XRD pattern by Scherrer’s equation [21]:

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

where $D$ is the crystallite size, $\lambda$ (= 1.5406 Å) is the wavelength of Cu Kα radiation, $\theta$ is the angle of the peak maximum (in 2θ), and $\beta$ is the full width at half maximum (FWHM, in 2θ). From the calculation, the average crystallite size is 19.8±0.6 nm. Figure 1(b) displays the microstructure of the as-prepared powders after calcined at 800 °C. The Y$_2$O$_3$ powders after the solvothermal reaction and calcination present a rod-like morphology and well-dispersed characteristics (Fig. 1(b)). The length of the nanorods is about 500 nm. The formation mechanism of anisotropically shaped nanorods rather than spherical nanoparticles may be induced by the competition between crystallographic anisotropy and crystallite growth kinetics in the solvothermal reaction process [22]. The inset in Fig. 1(b) reveals that the nanorods tend to agglomerate along their long axis. Figure 1(c) shows the microstructure of a monodispersed nanorod. The diameter of the nanorod is about 20 nm, which is consistent with the average crystallite size calculated from the XRD data. Figures 1(d) and 1(e) present the high-resolution TEM (HRTEM) images taken from different areas in Fig. 1(c). We can observe that a nanorod is composed of randomly oriented polycrystalline. Besides, the Y$_2$O$_3$ nanoparticles synthesized by the chemical precipitation method also show a single phase (Fig. 2(a)). Its calculated crystallite size by the Scherrer’s equation is 17.8±0.4 nm, which is close to the calculated crystallite size of the nanorod particles. The TEM images (Figs. 2(b) and 2(c)) confirm that the powders were composed of agglomerated near-spherical nanoparticles, and the size of the primary nanoparticle was consistent with the calculated result from the XRD data.
3.2 Densification and grain growth

Figure 3 compares the microstructures of the Y$_2$O$_3$ green bodies compacted under 50 MPa at room temperature using the nanorod powders and near-spherical nanopowders. The green body from the nanorod powders compacted at 50 MPa is composed of randomly and loosely distributed nanorods. The microstructure morphology shows that these nanorod particles are non-uniformly packed, and the pore size and distribution are non-uniform. This microstructural heterogeneity in the packed green bodies may lead to the formation of large pores, and crack-like voids in the final ceramics as different regions may have different densification behaviors [9,23]. On the other hand, the distribution of the particles and pores is uniform in the green body pressed by the near-spherical particles. The measured relative density of the nanorod Y$_2$O$_3$ green body is ~36.7%, which is lower than that of the near-spherical Y$_2$O$_3$ green body (45.2%). It is well believed that the powders consisting of the near-spherical/spherical particles are beneficial to having a uniform packing [24]. The results in Fig. 3 confirm this point of view.

Figure 4 displays the relative densities of the ceramics...
sintered from the nanorod and near-spherical powders at various temperatures. It can be observed that, when the temperature is below 400 °C, the density of the ceramics from the near-spherical powders is higher than that of the ceramics from the nanorod powders, resulting from the lower density of the green body made by the nanorod powders before sintering. However, after the temperature reaches 600 °C, with the increase of the sintering temperature, the relative density of the ceramics prepared by the nanorod powders increases significantly, which is higher than the density of the ceramics from the near-spherical powders.

Notice that 600 °C is a breakthrough point for the increase in the density of the ceramics using the nanorod powders, there may be a special mechanism in the sintering process. In pressure-assisted sintering, when an external pressure is applied to a powder compact, stress concentrates at particle contacts. Initially, the contact stress is much larger than the applied pressure due to the small contacts [25].

Generally, residual lattice stress/strain can be found in the sintered body after pressure-assisted sintering due to the uncoordinated deformations under the applied pressure [26,27]. Microscopic deviatoric strain will lead to the X-ray line broadening in addition to the reduced grain size [28,29]. It can be seen in Fig. 5(a) that, as the sintering temperature increases, the FWHM of the ceramic diffraction peaks gradually decreases, indicating a significant change in the grain size or residual lattice strain ($\varepsilon$). We can calculate the residual strain through the XRD data after the whole spectrum fitting. Williamson–Hall method [30,31] is a well-reputed method that considers the line broadening arising from small grain size and microstrain. The total broadening of the diffraction peaks can be expressed as

$$\beta_{2\theta} = \beta_{\text{size}} + \beta_{\text{strain}}$$  \hspace{1cm} (2)

where $\beta_{2\theta}$ is the FWHM for different diffraction planes, and $\beta_{\text{size}}$ and $\beta_{\text{strain}}$ are the broadening of diffraction peaks due to the grain size and microstrain, respectively. The detailed expression of Eq. (2) is defined as

$$\beta_{2\theta} = \frac{k\lambda}{D\cos\theta} + 4\varepsilon \tan\theta$$  \hspace{1cm} (3)

Rearranging Eq. (3), we can get

$$\beta_{2\theta} \cos\theta = \frac{k\lambda}{D} + 4\varepsilon \sin\theta$$  \hspace{1cm} (4)

Equation (4) is known as the UDM of the modified Williamson–Hall equation [31] considering the isotropic nature of the grains. Figure 5(b) shows the fitted linear plotting of the $\beta_{2\theta} \cos\theta$ as a function of $4\sin\theta$ for the diffraction patterns of the ceramics sintered at 200–1000 °C. The calculated results show that the residual micro-strain increases with the increase in the temperature and reaches the maximum value at 400 °C, as shown in Fig. 6. As the temperature continues to increase, the residual stress/strain decreases gradually until it reaches the minimum value at 600 °C.
increase, the $\varepsilon$ decreases rapidly and tends to be a stable value after 800 °C (Fig. 6). To elucidate the mechanism of $\varepsilon$ variations, the TEM images of the ceramic samples sintered at 200–900 °C were recorded. The results show that when the sintering temperature is ≤ 400 °C, the sample is mainly composed of the nanorod particles. As the temperature reaches 600 °C, most nanorods disappear, and the length of the remaining nanorods is greatly reduced. Equiaxed particles, which have a particle size of ~20 nm, like spherical powders, can be observed. As we know, the materials lose strength when being heated, meaning that the strength decreases with the temperature increasing. Thus, we can conclude that the Y$_2$O$_3$ nanorods begin to fracture to be the nano-equiaxed particles at 400 °C under the applied pressure of 50 MPa. Meanwhile, the deviatoric stress in the compact is released, and the $\varepsilon$ decreases fast. Significantly, after the fracture and rearrangement of the nanorods, the contacts between the particles enlarge (Fig. 6), which results in a higher density at 600 °C. When the temperature reaches 800 °C, the nanorods completely disappear and transform into the near-spherical particles, indicating that the densification mechanism should be the same at higher temperatures for these two types of powders with different morphologies.

In a temperature range of about 600–1100 °C, the sintering of the Y$_2$O$_3$ ceramics prepared by the nanocrystalline powders with different morphologies proceeded into the intermediate stage, evidenced by the increase in the relative density from ~60% to ~90% with the temperature increasing, as shown in Fig. 4. According to the sintering theory, the proportion of continuous pore channels gradually decreases at this stage, and closed pores begin to form. It is worth noting that the density of the ceramics prepared from the nanorod powders is consistently slightly higher than that of the ceramics prepared from the spherical powders at different temperatures. When the sintering temperature increases to 1200 °C, the relative densities of the ceramics prepared by the nanorods and rounded powders both reached 95%, indicating that the sintering process entered the final stage. Figure 7 displays the SEM images of thermally etched surfaces of the ceramics sintered from the nanorod powders and near-spherical powders at 1200–1500 °C. It can be seen in Fig. 7 that many closed pores are located at the triangular grain boundary in the ceramics sintered from the spherical powders at 1200 °C (Fig. 7(e)). As the
sintering temperature increases, the number of the closed pores gradually decreases. At 1400 °C, the relative density of the ceramics reaches a maximum. On the other hand, fewer and smaller residual pores inside the ceramics sintered at 1200 °C with the nanorod powders can be found (Fig. 7(a)). At 1300 and 1400 °C, the pores almost disappeared, and the ceramics with a relative density higher than 99% exhibited a dense structure. However, as the temperature continued to increase to 1500 °C, the densities of the ceramics sintered from different powders start to decrease accompanied by obvious grain growth. This phenomenon that the density decreases with the elevation of the temperature during the high-temperature range is common in the pressure-assisted sintering process [11,32]. In the case of the SPSed Y2O3 ceramics, it is more likely that the competition between the grain growth and the densification induces a decrease in the density after 1300–1400 °C. At a high-temperature range beyond 1300 °C, the migration rate of thermally-activated grain boundary mobility is higher than that of the pores, resulting in the detachment of the pores from the grain boundary and residual small pores within the grains [33]. Overall, two types of powders with different morphologies exhibited the same densification tendency during sintering at ≥ 800 °C, and the nanorod powders can achieve a higher degree of the densification at the same sintering temperature.

Figure 4 also shows the grain growth profile of the ceramics with the elevation of the sintering temperature. We use Nano Measure software to statistically count the average grain sizes of the ceramics from their thermally etched surface images. The grains with obvious and distinguishable boundaries are selected for statistics. As the grains of the ceramics sintered at relatively low temperatures (200–1100 °C) are difficult to distinguish, the whole pattern fitting on the XRD patterns with fitting errors < 7% was conducted for calculating the average grain size. The results show that the ceramics sintered from different powders share the same grain growth trend. The average grain sizes of the ceramics sintered ≤ 1100 °C are less than 100 nm and are in the sub-micron scale when the sintering temperature is in a range of 1200–1400 °C. As the temperature rises to 1500 °C, the average grain size will increase to 4.6 and 3.7 μm for the ceramics prepared from nanorod and near-spherical powders, respectively. The larger grain size may be induced by the increased surface energy produced in the fracture process of the nanorods, providing the driving force for the grain growth. From the perspective of high-density and small grain sizes for the sintered ceramics, 1300 °C is the optimized sintering temperature for the Y2O3 nanorod powders as the sintered ceramics reached the near-full density with ultra-fine grain size. Besides, the corresponding ceramics after polishing are highly transparent. Its optical property is discussed in Section 3.3.

In Ref. [20], we employed a very high pressure (5 GPa) to densify the nanorod powders. This very high pressure not only allows particle fracture and rearrangement, but also allows the plastic deformation of the Y2O3 particles (the yield stress of Y2O3 in a temperature range of 0–1500 °C is about 100–1000 MPa [34]). These unique mechanisms enable complete densification almost without the mechanism of the atomic diffusion as the densification temperature is only 520 °C, evidenced by the final grain size of the dense transparent ceramics being the same as the crystallite size of the raw powders [20]. However, 5 GPa is a very high pressure,
which is difficult to achieve and requires high cost. Most of the common sintering methods, such as the SPS and hot-pressing methods, involve the assisted pressure with a magnitude of dozens of mega pascal, which is lower than the yield stress of $\text{Y}_2\text{O}_3$ at various temperatures [34], and thus the contribution of the plastic deformation in the densification process is negligible. Therefore, the ceramics sintered at 1200–1500 °C from the spherical particle powders still have some residual pores, which are almost opaque, indicating that it is difficult to use the SPS to obtain highly transparent $\text{Y}_2\text{O}_3$ with no sintering aid added to promote densification. However, although the sintering pressure was only 50 MPa, the contact stress at the particle contacts, when the external pressure is applied to a powder compact, is much larger than the applied pressure due to the small contacts. Besides, the flexural strength of the optimal SPSed $\text{Y}_2\text{O}_3$ ceramics is only about 122 MPa [35] and will decrease with the elevation of the temperature. Thus, the $\text{Y}_2\text{O}_3$ nanorods are possibly fractured with the elevation of the temperature. Through the particle fracture and rearrangement, the transparent fine-grained $\text{Y}_2\text{O}_3$ ceramics were finally obtained using the nanorod powders, indicating that the particle fracture and rearrangement is an effective densification mechanism. In Fig. 6, we consider that the SPS process of the nanorod powders can be divided into three stages with the sintering temperature increasing. The first stage is the deviatoric stress-accumulation stage. Then, the nanorods begin to fracture and rearrange in the second stage. The deviatoric stress begins to release, and the residual micro-strain decreases significantly. After that, the grain growth is triggered. This stage is equivalent to the intermediate sintering stage in most ceramic sintering methods as the relative density of the corresponding ceramics sintered at 800 °C is about 77.2% [9]. Based on the aforementioned results, we consider that the fracture and rearrangement of the nanorods at low temperatures leads to the decrease of the particle size and the increase of the density and homogeneity of the compact before the beginning of the atom diffusion. This process can significantly improve the morphologies of the powder compact and pore-channel, and enhance the atom diffusion efficiency.

### 3.3 In-line transmittance

$\text{Y}_2\text{O}_3$ is an attractive optical material used as an IR dome and laser host [36]. As the $\text{Y}_2\text{O}_3$ transparent ceramics were successfully obtained from the nanorod powders, the in-line transmittance spectra and the optical image of the ceramics sintered at 1300 °C are presented in Fig. 8. It can be seen in the optical image that, the sample sintered under the optimized condition is highly transparent with the maximum transmittance of 52.0% and 80.8% in the visible light (the transmittance in the visible region is given at a wavelength of 550 nm) and IR wavelength ranges, respectively. The relatively lower transparency in the visible light band than that in the IR band indicates light scattering. Reference [37] shows that residual porosity can have a pronounced effect on the transparency. It was demonstrated by Mie scattering calculation that only 0.1% of residual porosity can deteriorate the transparency. The work of Zhu et al. [38] confirmed that the transmittance of the $\text{Y}_2\text{O}_3$ ceramics in the visible wavelength range can be improved by eliminating the nanopores. Thus, the main source for light scattering resulting in the decrease of the transmittance at the visible wavelength range in this work should be assigned to small pores. Figure 9 shows the TEM picture of the ceramics sintered at 1300 °C and annealed at 1000 °C. The inset is the optical image of corresponding ceramics (CDU represents Chengdu University).

![Fig. 8 In-line transmittance profile of $\text{Y}_2\text{O}_3$ transparent ceramics sintered at 1300 °C and annealed at 1000 °C. The inset is the optical image of corresponding ceramics (CDU represents Chengdu University).](image-url)
It confirms that the average grain size of the ceramics is about ~0.31 μm, and a few pores (marked by yellow circles) remain on the triangular grain boundaries. Optimizing the heating rate and adding sintering aids may improve the transmittance in the visible wavelength range [39]. We will conduct related experiments in the future. Generally, the transparent ceramics prepared by the SPS tend to appear yellow or brown colors, which is mainly caused by the absorption of the visible light by the dislocations or oxygen vacancies formed during the sintering process [40–42]. The as-obtained Y2O3 transparent ceramics are colorless after annealing at 1100 ℃ for 8 h in air, indicating near no dislocations or oxygen vacancies inside the transparent ceramic sample after annealing. Table 1 representatively lists the maximum transmittances of the Y2O3 transparent ceramics prepared by the SPS in the visible light (at 550 nm) and the IR bands. It can be seen in Table 1 that, the transmittance of the Y2O3 transparent ceramics via the SPS method in the visible light band is difficult to exceed 80%, which is generally lower than the transmittance in the IR range. The fabrication of the Y2O3 transparent ceramics with good optical properties needs high sintering temperatures or sintering aids to promote the densification [35,43–46]. However, when the nanorod powders were used in the present work, the transparent ceramics with a transmittance of more than 80% in the IR band were obtained without any sintering aids. The average grain size of the transparent ceramics in this work is only 0.31 μm, indicating that the sintering temperature for achieving full densification may be lower. As a comparison, the transmittance of the Y2O3 transparent ceramics sintered at the same temperature by the SPS, as reported in Ref. [35], is only 62.1% in the IR band. Besides, although the aforementioned sample (3.5 mm) is thicker than that in this study (1.0 mm), it has no transmittance in the visible light band. To sum up, the as-synthesized nanorod powders can be used for obtaining the Y2O3 transparent ceramics with good optical properties and small grain size without sintering aids.

3.4 Mechanical property

Furthermore, mechanical properties are very important in mechanical industrial applications of the transparent ceramics [47]. According to Hall–Petch relationship, the strength of the material is inversely proportional to the square root of the average grain size [48]. Therefore, reducing the grain size is of great significance for improving the mechanical properties of the transparent ceramics. As the average grain size of the Y2O3 transparent ceramics prepared at the optimal sintering temperature (1300 ℃) is only 0.31 μm, we have measured the mechanical properties of the sample. The main factors determining the Vickers hardness of the Y2O3 ceramics are the grain size and relative density. Moreover, due to the indentation size effect, the greater the load during the test, the lower the hardness [49]. Zhang et al. [50] calculated the theoretical hardness value of Y2O3 to be 7.71 GPa based on the first-principles study. The hardness value (7.67 GPa) of the IR-transparent Y2O3 ceramics with an average grain size of 21.5 μm is basically consistent with this theoretically calculated hardness [51]. Serivalsatit et al. [52] have prepared the Y2O3 transparent ceramics with a relative density of about 99.8%. Under a loading force of 0.5 N, the hardnesses of the samples with average grain sizes of 0.34 and 328 μm were measured to be 9.1 and 7.2 GPa, respectively. In the present work, the Y2O3 transparent ceramics sintered at 1300 ℃ show a Vickers hardness of 9.0 GPa, exceeding the theoretical value, which is mainly caused by the strengthening of ultra-fine grains (average grain size = 0.31 μm). The indentation fracture

### Table 1 Maximum in-line transmittances of Y2O3 transparent ceramics prepared by SPS in the visible light and IR bands

| Sample  | Sintering temperature (℃) | Thickness (mm) | Sintering aid | Transmittance in the visible light band (%) | Transmittance in the IR band (%) | Ref.  |
|---------|--------------------------|----------------|---------------|------------------------------------------|---------------------------------|------|
| Dy:Y2O3 | 1550                     | 1.0            | LiF           | 75.3                                     | 80.1                            | [44] |
| Sm:Y2O3 | 1450                     | 1.7            | LiF           | 74.6                                     | 80.2                            | [43] |
| Y2O3    | 1400                     | 1.0            | No            | 43.5                                     | 75.2                            | [45] |
| Y2O3    | 1600                     | 1.0            | No            | 59.6                                     | 82.2                            | [45] |
| Y2O3    | 1400                     | 2.5            | No            | —                                         | 77.9                            | [35] |
| Y2O3    | 1500                     | 2.5            | No            | —                                         | 82.0                            | [35] |
| Y2O3    | 1300                     | 3.5            | No            | 0                                         | 62.1                            | [46] |
| Y2O3    | 1300                     | 1.0            | No            | 52.0                                     | 80.8                            | This work |
Table 2  Hardness values of Y$_2$O$_3$ ceramics

| Ceramic  | RD (%) | $H$ (GPa) | $K_{IC}$ (MPa·m$^{1/2}$) | Load (N) | GS ($\mu$m) | Ref. |
|----------|--------|-----------|--------------------------|----------|-------------|-----|
| Y$_2$O$_3$ | —      | 7.705     | —                        | —        | —           | [50]|
| Y$_2$O$_3$ | 99.40  | 7.670     | —                        | 1        | 21.5±11.1   | [51]|
| Er:Y$_2$O$_3$ | 99.73  | 7.23±0.35 | 0.81±0.07                | 0.5      | 328         | [52]|
| Er:Y$_2$O$_3$ | 99.81  | 9.09±0.41 | 1.39±0.07                | 0.5      | 0.34        | [52]|
| Y$_2$O$_3$ | 99.40  | 9.0       | 1.30                     | 0.5      | 0.31        | This work |

Note: RD is the relative density, $H$ is the hardness, and GS is the average grain size.

4 Conclusions

In summary, by using the lab-made Y$_2$O$_3$ nanorod powders as the starting materials, the ultrafine-grained Y$_2$O$_3$ transparent ceramics with good optical and mechanical properties were fabricated successfully via the SPS. An effective densification mechanism involving the particle fracture and rearrangement is revealed, which provides a viable route to fabricate the transparent ceramics using non-spherical powders. Specifically, the following results were obtained:

1) The transparent ceramics with an average grain size of 0.31 $\mu$m show the maximum transmittance of 52.0% and 80.8% in the visible light (at 550 nm) and IR wavelength ranges, respectively. The Vickers hardness and $K_{IC}$ are 9.0 GPa (the theoretical hardness value of Y$_2$O$_3$ is 7.7 GPa) and 1.30 MPa·m$^{1/2}$, respectively.

2) The unique sintering process can be divided into three stages: (i) In the primary sintering stage, the deviatoric stress accumulates with the increase of the sintering pressure. (ii) The nanorod particles can fracture and rearrange before the beginning of the atom diffusion (~600 °C), which associates with the improvement of the density and homogeneity of a powder compact and a decrease in the initial particle size. (iii) The grain grows associated with the further densification of the compact in the intermediate and the last sintering stage.

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Declaration of competing interest

The authors have no competing interests to declare that are relevant to the content of this article.

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