

Manufacture of Spherical Ceria and Its Polishing Properties for Silica Films

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Abstract

Ceria is used in polishing processes for the manufacture of many devices, for example, in the planarization of semiconductor SiO₂ film or mirror polishing of smartphone cover glasses. However, cerium is a rare-earth element, supplies of which are at risk due to restrictions on mining as a result of environmental pressures. Most commercial ceria is prepared by comminution, with subsequent classification to control the particle-size distribution; consequently, the resource efficiency of ceria declines as increasingly finer grades are produced. In an attempt to overcome this problem, focused on a resource-efficient process for preparing ceria, the practicality of the resulting ceria, and the possibility to reuse ceria after use in polishing. Ceria prepared by our new process was found to combine a high degree of monodispersity with a high resource efficiency. When a sample of the novel spherical ceria with a particle diameter of around 200 nm was used to process semiconductor SiO₂ film, the removal rate was improved to about 130% of that of commercial ceria of a similar size prepared by comminution; moreover, the surface roughness was reduced to about 56% (equivalent to that produced by colloidal silica), and a good flatness was achieved. Furthermore, analysis of the spherical ceria after polishing confirmed that there was no physical change in morphology and no chemical change in the particle surface. The experimental results and analysis showed that spherical ceria prepared by our novel process can potentially be used in planarization of semiconductor SiO₂ films and might be capable of being reused after polishing.

1 Introduction

As a result of the recent acceleration in the uptake of 5G cellular networks and the growth in demand for data centers there has been a significant increase in the production of high-performance multifunction devices. Ceria (CeO₂) is used in several polishing processes during the manufacture of such devices, for example in the planarization of semiconductor SiO₂ films and in mirror polishing of smartphone cover glasses. However, cerium is a rare-earth element, and the supply of ceria is at risk because cerium mining has been restricted in response to environmental pressures. In 2011, in an attempt to find a solution to the problem of limitations on the supply of ceria, we developed and put to practical use a previously reported technology for recycling ceria [1]. In 2014, we also reported on this ceria-recycling technology and on the mechanism of polishing of glass substrates with ceria [2,3]. This technology to reuse ceria has also been patented [4–6].

While we were elucidating the mechanism of polishing of glass substrates with ceria, we confirmed by scanning electron microscopy (SEM) that the ceria abrasives did not change shape after polishing. We also confirmed that in the polishing of glass substrates with ceria, the glass component derived from the object to be polished was present in the slurry in a dissolved or gel-like state [2,3]. Therefore, spherical ceria, which does not undergo physical or chemical changes, might be capable of being reused after use in polishing.

Ceria is typically prepared by a liquid-phase method from an aqueous solution obtained by treating cerium with a mineral acid. There are two methods for producing fine ceria particles: a building-up method, in which atoms and molecules are amalgamated together, or a breaking-down method, in which a powder or the like is comminuted. Commercially,

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ceria is produced by a breaking-down method in which an aqueous solution, prepared by treating cerium with a mineral acid, is treated with an alkali to precipitate ceria, which is then dried, calcined at a high temperature, and comminuted to produce fine particles. There are both dry and wet forms of the breaking-down method, however as shown in Fig. 1, the limits of comminution are claimed to be 1–3 μm and 0.1–0.3 μm (100–300 nm), respectively, and it is not possible to obtain particles with a size of several hundreds of nanometers that are well shaped and highly monodisperse.

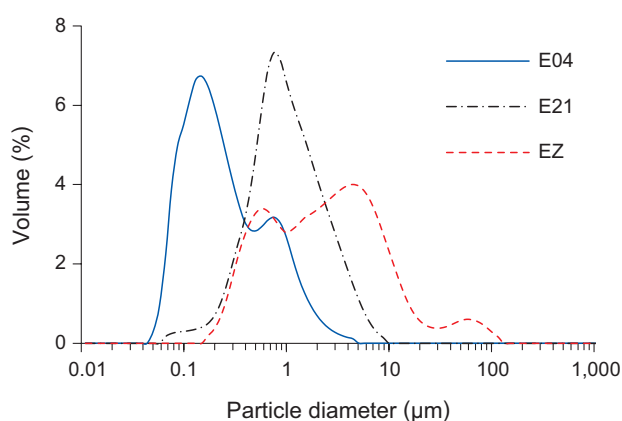


Fig. 1 Particle-size distributions of commercial ceria samples E04, E21, and EZ (Mitsui Mining & Smelting Co., Ltd.).

Furthermore, in the polishing of aluminosilicate substrates with samples of commercial ceria with various particle sizes prepared by the breaking-down method, as shown in Fig. 2, the removal rate (RR) tend to increase linearly with decreasing particle size. In this polishing process, in addition to its abrasive action, the ceria acts as a solid catalyst for the dissolution and erosion of the silicate surface; consequently, the contact area has a marked effect on the RR.

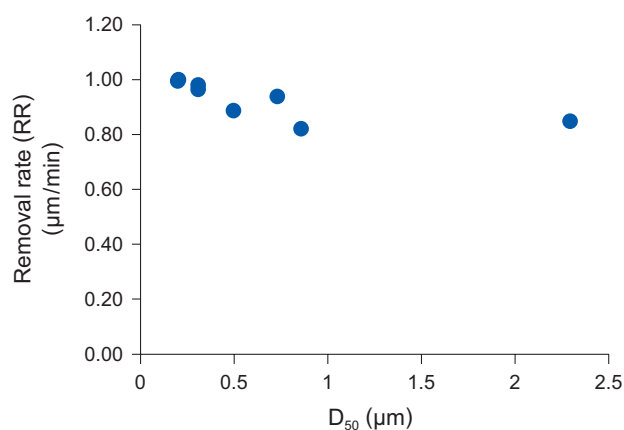


Fig. 2 Comparison of the removal rates for polishing of an aluminosilicate substrate with samples of commercial ceria of various particle sizes, manufactured by the breaking-down method.

Because the particle-size distribution of ceria prepared by the breaking-down method is controlled through classification after comminution, the resource efficiency of the ceria decreases as the fineness of the resultant ceria particles increases. In particular, the ceria used for planarization of semiconductor SiO_2 films has a particularly low resource efficiency because of the need for careful control of the particle-size distribution.

Therefore, in this study, we focused on a resource-efficient process for the preparation of ceria, the practicality of the prepared ceria, and the possibility to reuse ceria after use in polishing. By applying the technology used to manufacture silver halide particles, we developed a unique process for preparing ceria that permits control of the size, shape, and size distribution of the resulting particles. This novel ceria was used to polish a SiO_2 film, and the polishing performance was compared with those of commercial abrasives, and the morphology and surface of the novel ceria after polishing was analyzed.

2 Experimental

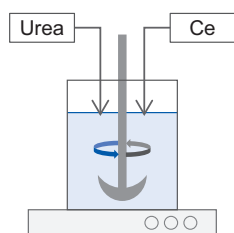
2.1 The novel ceria and its preparation process

We established a process for preparing ceria by generating uniform nuclei from a supersaturated solution and growing them as seeds in a building-up method to give particles of a size that can be controlled over a wide range with a high level of monodispersity. We prepared following ceria with controlled particle size, shape, and size distribution using established ceria preparation process:

Spherical ceria: ϕ 293 nm, 194 nm, 102 nm, 60 nm
 Square-shaped ceria: ϕ 90 nm, 30 nm

Fig. 3 shows a schematic representation of the representative process for preparing spherical ceria. Urea [$\text{CO}(\text{NH}_2)_2$; 0.20 M] was added to 10 L of a 0.01 M aqueous solution of cerium(III) nitrate, and the resulting solution was stirred and heated at 98 $^\circ\text{C}$ for one hour.

The spherical cerium(III) hydroxycarbonate that precipitated from the aqueous solution was collected on a membrane filter, dried, and calcined at 600 $^\circ\text{C}$ to give spherical ceria. Urea, used as the precipitant, generates carbonic acid by thermal decomposition and buffers the pH near the neutral point. This method for preparing the spherical ceria has been patented [7,8].



1. Urea decomposition in aqueous solution
 $\text{CO}(\text{NH}_2)_2 \rightarrow \text{NH}_3 + \text{HNCO} \leftrightarrow \text{NH}_4^+ + \text{NCO}^-$
 • In acid solution:
 $\text{NCO}^- + 2\text{H}^+ + \text{H}_2\text{O} \leftrightarrow \text{NH}_4^+ + \text{CO}_2$
 • In neutral or alkaline solution:
 $\text{NCO}^- + \text{OH}^- + \text{H}_2\text{O} \leftrightarrow \text{NH}_3 + \text{CO}_3^{2-}$
2. Formation of cerium(III) hydroxycarbonate
 $\text{Ce}^{3+} + \text{OH}^- + \text{CO}_3^{2-} \leftrightarrow \text{Ce}(\text{OH})\text{CO}_3$
3. Calcination and oxidization of cerium(III) hydroxycarbonate
 $2\text{Ce}(\text{OH})\text{CO}_3 \rightarrow \text{Ce}_2\text{O}_3 + 2\text{CO}_2 + \text{H}_2\text{O}$

Fig. 3 Outline of the process for preparing spherical ceria.

2.2 Evaluation method of the SiO₂-film-polishing performance of the novel ceria

The SiO₂-film-polishing performance of ceria prepared by our novel process was evaluated in comparison with commercial abrasives prepared by the breaking-down method. A 4-inch-diameter Si wafer on which a SiO₂ film was formed by plasma chemical vapor deposition (CVD) of tetraethyl orthosilicate (TEOS) was used as an evaluation sample. The experiment was conducted by using the novel spherical ceria [particle size (ϕ), 293, 194, 102, or 60 nm], the novel square-shaped ceria (ϕ 90 or 30 nm), commercial ceria (ϕ 220 nm: HS-8005; Hitachi Chemical Co., Ltd.), and commercial colloidal silica (ϕ 20 nm: COMPOL20; Fujimi Inc. Co., Ltd.) which is commonly used in high quality planarization. SEM micrographs of the commercial ceria and of colloidal silica are shown in Fig. 4. Table 1 lists the experimental processing conditions.

Table 1 Experimental processing conditions.

Evaluation sample	Si wafer with TEOS-SiO ₂ film Diameter: 4 in
Abrasive	1) Novel spherical ceria (ϕ 293 nm, 102 nm, 60 nm) 2) Novel square-shaped ceria (ϕ 90 nm, 30 nm) 3) Commercial ceria (ϕ 220 nm: HS-8005, Hitachi Chemical Co., Ltd.) 4) Commercial colloidal silica (ϕ 20 nm: COMPOL20, Fujimi Inc. Co., Ltd.)
Slurry concentration	0.1 wt%
Polishing pad	Ceria: IC1000 (Nitta Haas Co., Ltd.) Silica: BNP-4300-02 (BN Technology Corp.)
CMP tool	Bni52 (BN Technology Corp.)
Time	60 s
Flow rate	40~100 mL/min
Pressure	0.04 kg/cm ²

To examine the effect of the polishing pressure on the polishing performance of the novel spherical ceria and of the commercial ceria, the polishing pressure was changed from 0.01 kg/cm² to 0.12 kg/cm² under the experimental treatment conditions shown in Table 1, and the resulting polishing performance was evaluated. Furthermore, to confirm the practicality of using the spherical ceria, detailed evaluations were carried out on a 12-inch-diameter Si wafer of a size used in semiconductor manufacturing, on which a SiO₂ film was formed by plasma CVD with TEOS. The experiment was conducted by using a sample of the novel spherical ceria (ϕ 194 nm) and a commercial ceria with almost the same particle size (ϕ 220 nm: HS-8005). Table 2 shows the experimental processing conditions.

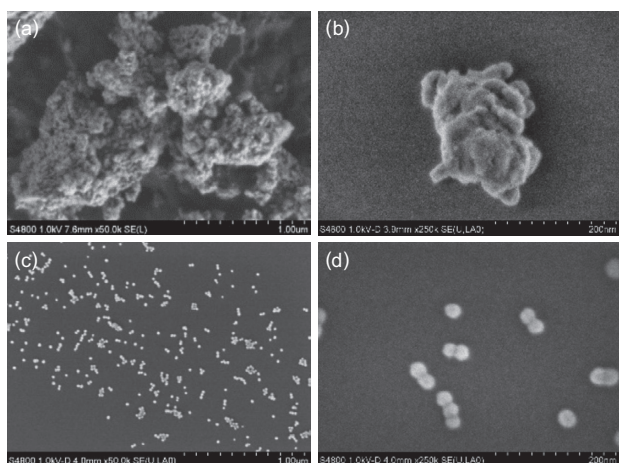


Fig. 4 SEM micrographs at two magnifications of (a) and (b) commercial ceria (ϕ 220 nm: HS-8005; Hitachi Chemical Co., Ltd.). (c) (d) Commercial colloidal silica (ϕ 20 nm: COMPOL20; Fujimi Inc. Co., Ltd.).

Table 2 Experimental processing conditions.

Evaluation sample	Si wafer with TEOS-SiO ₂ film Diameter: 12 in
Abrasive	1) Novel spherical ceria (ϕ 194 nm) 2) Commercial ceria (ϕ 220 nm: HS-8005, Hitachi Chemical Co., Ltd.)
Slurry concentration	0.1 wt%
Polishing pad	Polyurethane pad (IC1570, Nitta Haas Co., Ltd)
CMP tool	F-REX300E, Ebara Corp.
Time	60 s
Flow rate	250 mL/min
Pressure	100-200 hPa

2.3 Evaluation method of reusability of the novel ceria

To verify the possibility to reuse the novel spherical ceria after use in polishing, we observed the physical changes in the morphology after polishing under the experimental processing conditions shown in Table 1, and for the chemical changes, we focused on the chemical reaction between the spherical ceria and the SiO₂ film. To examine the chemical reaction in detail, we investigated the ¹⁸O distribution in spherical ceria adhering to a polished sample of ¹⁸O-enriched SiO₂ film by using time-of-flight secondary-ion mass spectrometry (TOF-SIMS).

The ¹⁸O-enriched SiO₂ film was produced on a 6-inch-diameter Si wafer as follows (Fig. 5). First, the native oxide film on the wafer was removed by treatment with aqueous HF. The wafer was then annealed under ¹⁸O₂ in a vacuum chamber at 15 Torr and 800 °C for 10 hours so that an ¹⁸O-enriched SiO₂ film formed on its surface.

The evaluation sample was polished with the spherical ceria, and the amounts of ¹⁸O present on the surface of the spherical ceria before and after polishing were measured to examine for the presence of a chemical reaction between CeO₂ and SiO₂ during polishing with spherical ceria.

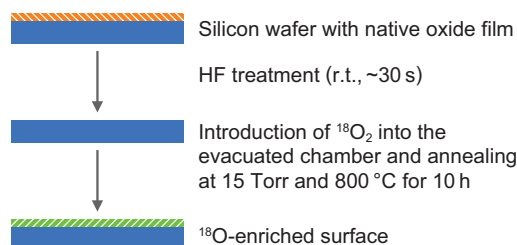


Fig. 5 Process for the introduction of ¹⁸O into the SiO₂ film. 1. Aqueous HF treatment to remove the native oxide film. 2. Introduction of ¹⁸O₂ into an evacuated chamber and annealing at 15 Torr and 800 °C for 10 h.

3 Experimental results and discussion

The experimental results of the novel ceria are shown below.

Fig. 6 shows a comparison of the particle-size distribution of the novel spherical ceria prepared by the process shown in Fig. 3 and that of commercial ceria prepared by the breaking-down method. The spherical ceria had a narrow particle-size distribution and a high monodispersity. Moreover, its preparation does not require the removal of large quantity of classified material, unlike that of commercial ceria prepared by the breaking-down method. Spherical ceria is therefore more resource efficient.

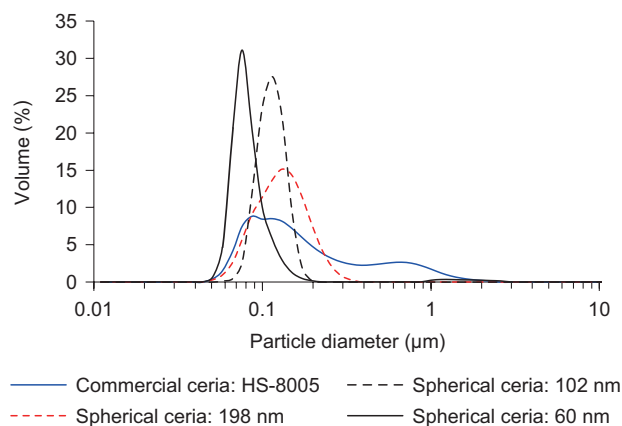


Fig. 6 Comparison of the particle-size distributions of the novel spherical ceria and a commercial ceria prepared by the breaking-down method.

Fig. 7 shows examples of images obtained by transmission electron microscopy (TEM) of spherical ceria prepared by the processing method described above, before and after calcination. By calcination at 600 °C in atmospheric air, the synthesized precursor spherical cerium(III) hydroxycarbonate particles were converted into cerium(IV) oxide particles, while their shape was retained. The particle size of the precursor particles could be controlled in the range 50–1500 nm by changing the conditions of temperature and concentration for the urea-precipitation process.

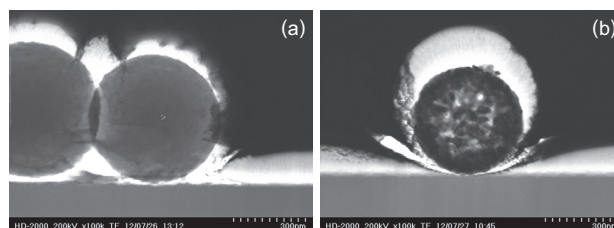


Fig. 7 TEM micrographs of the novel spherical ceria before and after calcination. (a) Before calcination. (b) After calcination.

Fig. 8 is an example of an SEM image of a sample of spherical ceria produced by the process shown in Fig. 3, demonstrating that that particles were all highly spherical in shape (200 nm). The particle diameter is 20–30% less than that of the precursor particles, because the spherical hydroxycarbonate precursor particles are converted into oxide particles while their shape is maintained. Therefore, to produce ceria particles of a given size, an allowance must be made for the decrease in particle size as a result of the calcination process. As can be seen in Fig. 8, the novel spherical ceria is uniformly spherical and has a very narrow particle-size distribution. The coefficient of variation (CV), an indication of the monodispersity of the particles, is less than 7%. Because the shape of

the particles is similar to that of so-called colloidal silica, it would be expected to produce polished surfaces with few defects, such as scratches.

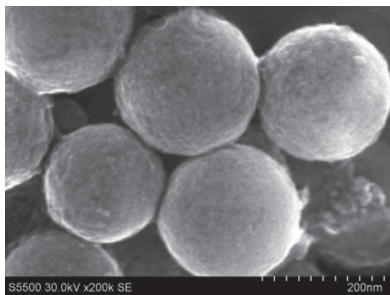


Fig. 8 SEM micrographs of the novel spherical ceria.

Fig. 9 is an example of an SEM micrograph of samples of spherical ceria of two different particle sizes, produced by the method described above. Because the spherical ceria has high monodispersity, the number of active particles contributing to polishing is large, suggesting that highly efficient polishing might be expected.

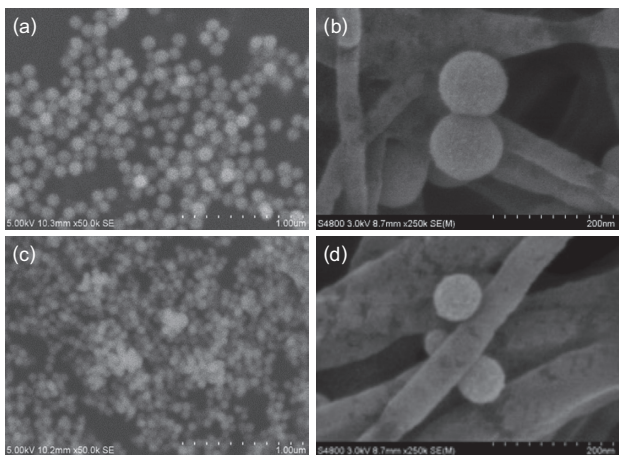


Fig. 9 SEM micrographs of novel spherical ceria of different particle diameters at two magnifications. (a) and (b) 102 nm; (c) and (d) 60 nm.

Furthermore, by changing the concentration conditions during synthesis in the range 0.001 to 0.1 M for the aqueous solution of cerium(III) nitrate and 0.02 to 2 M for urea, square-shaped ceria particles were obtained. Fig. 10 shows examples of SEM micrographs of square-shaped ceria particles of various sizes.

Fig. 11 shows a comparison of the SiO₂-film-polishing performances of various samples of the novel ceria with those of two commercial abrasives (colloidal silica and ceria prepared by the breaking-down method) under the experimental conditions shown in Table 1. The RRs were compared with that of the commercial ceria, with the relative RR of commercial ceria (Fig. 11(a)) set at a value of 1.

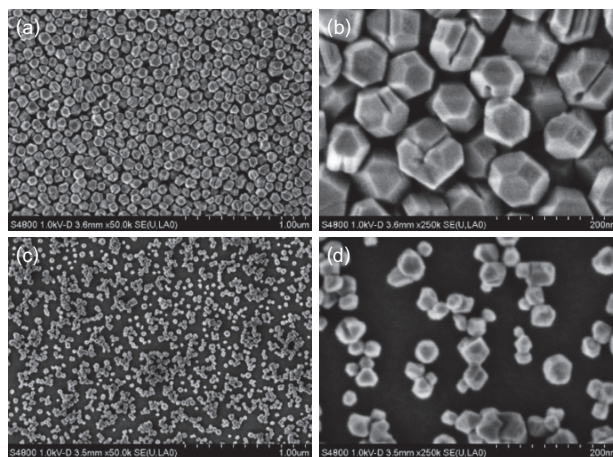
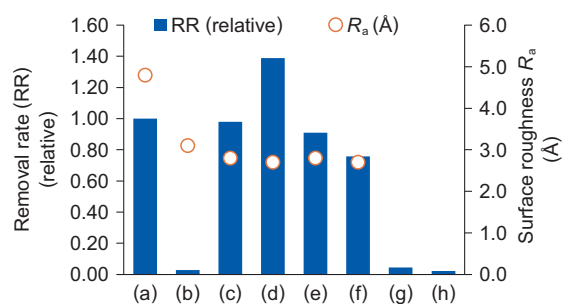


Fig. 10 SEM micrographs of novel square-shaped ceria particles of various sizes at two magnifications. (a) and (b) 90 nm; (c) and (d) 30 nm.



(a) Commercial ceria: 220 nm (e) Spherical ceria: 102 nm
 (b) Commercial colloidal silica: 20 nm (f) Spherical ceria: 60 nm
 (c) Spherical ceria: 293 nm (g) Square-shaped ceria: 60 nm
 (d) Spherical ceria: 194 nm (h) Square-shaped ceria: 30 nm

Fig. 11 Comparison of the relative RR and surface roughness R_a when polishing a Si wafer with a TEOS-SiO₂ film.

A comparison between the novel spherical ceria (Fig. 11(d)) and the commercial ceria with a similar particle size of about 200 nm (Fig. 11(a)) showed that the RR increased to 137% for the spherical ceria. The surface roughness of the sample polished by the spherical ceria, $R_a = 2.8 \text{ \AA}$, was reduced by about 56% compared with the that produced by the commercial ceria (4.8 \AA); the corresponding value for the colloidal silica was 3.1 \AA . We believe that the reason for this improvement is that the spherical ceria had a sharper particle-size distribution and showed superior dispersibility compared with the commercial ceria produced by comminution, and that it contained larger number of active particles that contributed to processing.

We could not accurately measure the surface roughness R_a produced by processing with the novel square-shaped ceria (Fig. 11(g), (h)), because it had a low RR and a large in-plane variation. This is probably because the rotational torque fluctuates greatly and polishing resistance changes significantly during processing with square-shaped ceria.

Fig. 12 and Fig. 13 shows the effect of polishing pressure on the polishing performance of the novel spherical ceria and that of commercial ceria, prepared by the breaking-down method, on changing the polishing pressure from 0.01 kg/cm² to 0.12 kg/cm² under the experimental treatment conditions shown in Table 1.

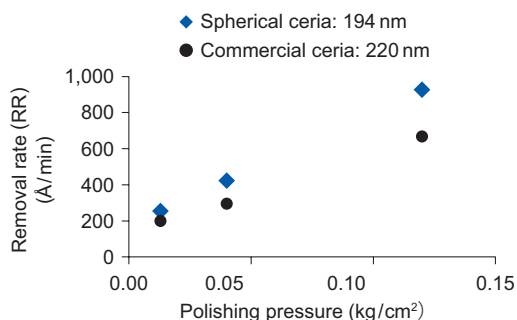


Fig. 12 Comparison of the RR at two polishing pressures for polishing of a TEOS-SiO₂ film on a 4-inch-diameter Si wafer with the novel spherical ceria and commercial ceria prepared by the breaking-down method.

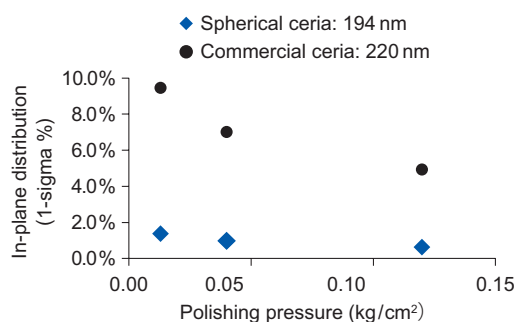


Fig. 13 Comparison of the in-plane distribution of different polishing pressures when polishing a TEOS-SiO₂ film on a 4-inch-diameter Si wafer with the novel spherical ceria and commercial ceria prepared by the breaking-down method.

On polishing at a higher polishing pressure, the novel spherical ceria showed a greater increase in the RR and an improved in-plane distribution compared with the commercial ceria prepared by the breaking-down method. As the spherical ceria had a narrow particle-size distribution and a high monodispersity, the polishing pressure was uniformly generated and it is therefore likely that the number of active

particles contributing to the processing was large and that the activity of each particles increased when the polishing pressure was increased.

To evaluate the novel ceria in more detail, we used as a substrate a 12-inch-diameter Si wafer on which a SiO₂ film was formed by plasma CVD using TEOS, and we compared the action of a sample of the spherical ceria that showed a good RR and surface roughness in the processing of a TEOS-SiO₂ film on the smaller Si wafer, as described above, with that of commercial ceria used in planarization of semiconductor SiO₂ films. Table 3 shows the RR and in-plane distribution of the 12-inch-diameter Si wafer evaluation sample processed for one minute.

The RR of the novel spherical ceria increased to 230 nm/min, which was about 130% of the value of 175 nm/min for the commercial ceria prepared by the breaking-down method. In addition, the novel spherical ceria produced a surface roughness *R*_a of 2.6 Å, a reduction of about 56% compared with that of commercial ceria (4.6 Å). Furthermore, the in-plane distribution was 5.01% for commercial ceria, whereas that of the novel spherical ceria was 2.22%, a reduction to about 44%. Again, because the spherical ceria had a sharp particle-size distribution and high monodispersity, polishing pressure was uniformly generated and, therefore, the number of active particles contributing to processing was large. It can be said that the smaller and more monodisperse the particles are, the greater the number of active particles contributing to the processing on increasing the polishing pressure.

Further, there is a limit to the polishing pressure achievable with a chemical mechanical polishing (CMP) tool, and the larger the processing area, the more difficult it is to produce a uniform polishing pressure. In the case of the 4-inch evaluation sample, the in-plane distribution improved with increasing polishing pressure (Fig. 11), but in the case of the 12-inch sample, the in-plane distribution did not change on increasing the polishing pressure (Table 3).

Table 3 The primary particle size of abrasive and the average removal rates and in-plane distributions for a 12-inch-diameter Si wafer surface.

	Original primary particle size (nm)	Polishing pressure (hPa)	Average removal rate (RR) (nm/min)	Surface roughness (<i>R</i> _a) (Å)	In-plane distribution (1-sigma %)
Spherical ceria	194	100	156	2.8	2.20
		150	198	2.7	2.00
		200	230	2.6	2.22
Commercial ceria (HS-8005)	220	200	175	4.6	5.01

This might be because the polishing pressure of the CMP tool was not uniformly applied to the polishing surface when the processing area of the evaluation sample was larger.

In addition, to verify the possibility of reusing the novel spherical ceria after it is used in polishing, we observed the physical changes in the morphology after polishing under the experimental processing conditions shown in Table 1, and it was confirmed that the morphology of the spherical ceria did not change after polishing (Fig. 14).

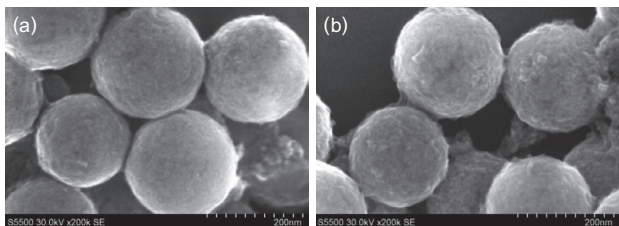


Fig. 14 SEM micrographs of the novel spherical ceria before and after polishing. (a) Before polishing. (b) After polishing.

Moreover, for the chemical changes, we focused on the chemical reaction between the spherical ceria and the SiO₂ film. We investigated the chemical reaction between the CeO₂ and the SiO₂ film by examining the ¹⁸O distribution of spherical ceria adhering to a processed ¹⁸O-enriched SiO₂ film by using TOF-SIMS (Fig. 15).

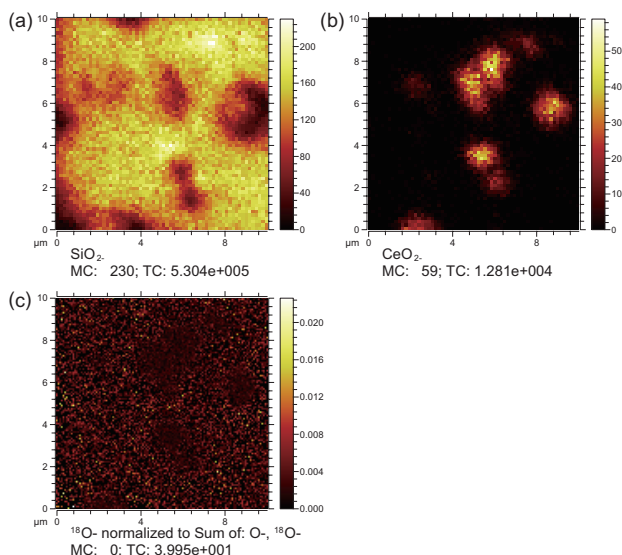


Fig. 15 TOF-SIMS images of an evaluation sample polished with the novel spherical ceria (imaging mode). (a) Secondary-ion images of SiO₂⁻ and (b) CeO₂⁻, and (c) ¹⁸O-densities of the evaluation sample.

When the secondary-ion images of SiO₂ and CeO₂ were compared, a contrast appeared, indicating that it was possible to observe the spherical ceria and the

SiO₂ film separately. Moreover, the ¹⁸O density at locations where spherical ceria were present was less than that in the SiO₂ film. Therefore, ¹⁸O from the SiO₂ film was not transferred to the surfaces of the spherical ceria. The results showed that there was no chemical reaction between the spherical ceria and the SiO₂ film.

Based on the above results, the mechanism of SiO₂ polishing by ceria is discussed as follows.

From our analysis of the state of the ceria, we consider that the mechanism of SiO₂ polishing with ceria conforms closely to Preston's law [9]. On the other hand, we confirmed by SIMS isotope analysis that no chemical reaction occurs on the surface of the polished ceria. Moreover, morphological observations by SEM showed that there was no change in the size or morphology of the particles after polishing, suggesting that the reaction that occurs during polishing might be reversible and that a so-called catalytic effect or function might occur. Therefore, the charge transfer of Ce³⁺ and Ce⁴⁺ inside the ceria at a certain polishing pressure is considered to cause Ce³⁺ on the surface of ceria and the interaction between the ceria and SiO₂ surface. With these things in mind, the mechanism of SiO₂ polishing by ceria is illustrated in Fig. 16.

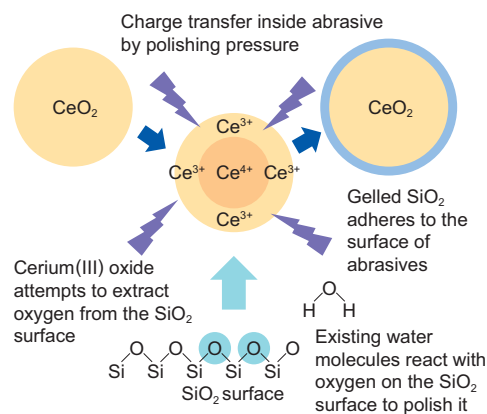


Fig. 16 Mechanism of SiO₂ polishing by ceria.

From this mechanism of SiO₂ polishing by ceria, it can be said that higher RR can be obtained by increasing the interaction between ceria and SiO₂ film. Therefore, it is necessary to increase the number of ceria with Ce³⁺ on the surface, for which monodisperse and spherical ceria with uniform polishing pressure are suitable. As shown in Fig. 17, the spherical ceria produced by our novel process is expected to apply uniform pressure and a higher number of active particles per unit area of the SiO₂ film than the non-uniform commercial ceria produced by the breaking-down method.

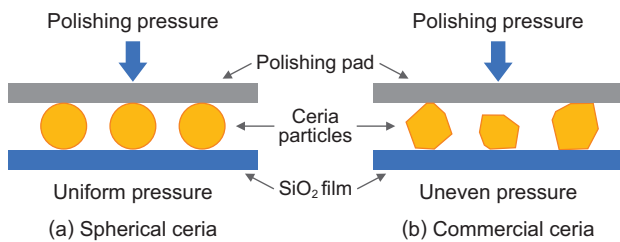


Fig. 17 Image of polishing pressure applied to each particle and SiO₂ film. (a) Spherical ceria and (b) Commercial ceria prepared by the breaking-down method.

In terms of the practicality, one consideration regarding the use of spherical ceria is the relationship between the polishing pressure and the particle size. The smaller and more monodisperse the particles are, the greater will be the number of particles per unit area, but at a constant polishing pressure the number of active particles contributing to processing will be reduced because the polishing pressure applied to each particle is lower and, consequently, the particle density per unit volume decreases. As the number of contacts with the SiO₂ film decreases, the number of active particles contributing to the processing decreases. We therefore consider that it is necessary to select an optimal particle size that will depend on the polishing pressure attainable with the CMP tool.

4 Conclusion

In this study, we focused on a resource-efficient process for the preparation of ceria, the practicality of the resulting ceria, and the possibility to reuse ceria after use in polishing. The results show that our novel process for preparing spherical ceria is resource efficient, and the spherical ceria produced by this process have potential to be used for planarization of semiconductor SiO₂ films and, moreover, might be reusable after use in polishing. In summary, the results are as follows:

- 1) The novel spherical ceria is highly resource efficient as it is not necessary to remove large quantities of material by classification during the preparation process.
- 2) As a result of comparing the polishing performance of the novel spherical ceria with that of commercial ceria produced by the breaking-down method in polishing a 12-inch-diameter Si wafer, the size of which is used in semiconductor manufacturing, novel spherical ceria was superior in terms of removal rate, surface roughness *Ra*, and in-plane distribution, and can potentially be used in planarization of semiconductor SiO₂ films.

- 3) Analysis of the novel spherical ceria after polishing confirmed that no physical change in morphology and no chemical change of the surface of the particles occurred, therefore, might capable of being reused after polishing.
- 4) Our study on the polishing mechanism suggests that the catalytic effect of the novel spherical ceria on SiO₂-film-polishing is more significant. The effect of polishing pressure on polishing performance needs to be further discussed.

References

- [1] New Energy and Industrial Technology Development Organization (NEDO). 'Application of industrial technology innovation: The practical application of rare metal substitution and reduction' grant. NEDO Activity Report Annual Report 2012, 25 (2012) (in Japanese).
- [2] Maezawa A and Nagai Y. Recycling of the used cerium oxide slurry. *Ceramics Japan* 49, 40–43 (2014) (in Japanese).
- [3] Maezawa A and Nagai Y. Recycle of cerium oxide polishing powder. *Konica Minolta Tech. Rep.*, 11, 97 (2014); available at https://www.konicaminolta.jp/about/research/technology_report/2014/pdf/11_maezawa.pdf (Accessed: Nov. 4th, 2020) (in Japanese).
- [4] Takahashi A, Nagai Y, and Maezawa A. Abrasive material regeneration method and regenerated abrasive material, US Patent 9,796,894 (B2) (2017).
- [5] Nagai Y, Maezawa A, and Takahashi A. Abrasive regeneration method, US Patent 9,802,337 (B2) (2017).
- [6] Inui C, Maezawa A, Nagai Y, and Hirayama N. Recovery method for abrasive, US Patent 10,286,522 (B2) (2019).
- [7] Mizoguchi K, Maezawa A, Ito N, Takahashi A, Wakamatsu H, Nagai Y, and Inui C. Polishing material particles. Method for producing polishing material, and polishing processing method, US Patent 9,868,885 (B2) (2018).
- [8] Mizoguchi K, Maezawa A, Takahashi A, Ito N, Hirayama N, Wakamatsu H, Nagai Y, and Inui C. Abrasive: Method for producing abrasive, and polishing method, JP 6424818 (2018).
- [9] Preston F W. The theory and design of plate glass polishing machines. *J. Soc. Glass Technol.* 11, 214–256 (1927).