



Chemical mechanical glass polishing with cerium oxide: Effect of selected physico-chemical characteristics on polishing efficiency

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ABSTRACT

Cerium oxide with an excellent glass polishing efficiency was prepared by annealing carbonate or oxalate precursors. The temperature of calcination was identified as a critical parameter that governs the key properties, including the polishing efficiency, of the polishing powders; conversely, the time of calcination appears to be less important. Only the cerium oxides prepared at temperatures above 700 °C exhibited good glass polishing capabilities in terms of both the material removal rate and the quality of the polished surface; the maximum polishing efficiency was produced by the samples annealed at 1050 °C. Polishing powders were characterized using X-ray diffraction (XRD), advanced microscopic techniques (SEM, TEM), Brunauer–Emmett–Teller (BET) surface area, X-ray photoelectron spectroscopy (XPS) and other techniques. Detailed XRD and microscopic investigations revealed a strong correlation between the crystallinity of cerium oxide and its polishing efficiency, which is consistent with the mechanical effect of the polishing mechanism. However, XPS measurements suggest that the chemical characteristics, namely the presence of the Ce^{3+} ions, also play an important role in glass polishing and planarization. Both mechanical and chemical contributions to the polishing process are influenced by the calcination temperature.

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1. Introduction

The process of glass polishing and planarization is of fundamental importance in many industries, including jewelry, precise optics, laser techniques and electronics [1–4]. Cerium oxide is one of the most effective glass polishing agents, providing high-quality polished surfaces [2,5,6]. The high polishing efficiency of cerium oxide stems from its chemical properties because the polishing of glass and certain other surfaces is not only a purely mechanical process, but it also involves a complex set of chemical interactions between the polishing agent, the polished surface and the polishing medium [7]. Although mechanical factors, such as pressure or relative motion of the polishing tool with respect to the polished surface, dominate in the material removal process, chemical interactions cause surface smoothing and planarization. This process is commonly described as chemical mechanical polishing (or planarization) (CMP) [1,4,8–10], and the factors affecting its

efficiency have been increasingly investigated recently in relation to the growing demand for high-quality surfaces in the production of modern electronic devices.

Ceria-based polishing powders are typically prepared via the thermal decomposition of suitable precursors, which are typically cerium oxalates, hydroxides, acetates, or carbonates [5,11–14]. The contents of cerium oxide in the polishing powders vary over a relatively broad range from ca. 50 to 100% [15], and the presence of other rare earth metals in the polishing powders is not considered a critical factor because cerium oxide forms solid solutions with the oxides of other lanthanide elements [16] and incorporates them into its crystal lattice without changing its crystalline structure. However, the content of cerium oxide in high-grade, commercially available polishing powders typically approaches 100% [15]. Based on the theory of CMP, the polishing agent should have suitable mechanical characteristics, such as mechanical resistance, particle size distribution and crystallinity [13,14,17–20]; concurrently, it should retain sufficient chemical reactivity to interact with the polished surface under given conditions [21–23]. In some branches, such as in shallow trench isolation CMP, the

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polishing selectivity is required, i.e. an ability to remove silicon dioxide and not silicon nitride [24].

It is generally accepted that the properties of cerium oxide are governed primarily by the conditions that it experiences during thermal treatment (annealing), primarily by the calcination temperature [25]. It was found for the polishing agents prepared from carbonate precursors that the polishing efficiency (expressed in terms of the removal rate) increases with the calcination temperature [24,26,27], with the most dramatic changes occurring in the temperature range of ca. 300–700 °C. According to Praveen et al. [24], the efficiency of polishing agents did not increase with further increasing the calcination temperature up to 1000 °C, whereas we observed in our earlier works a certain increase for the polishing agents annealed at temperatures slightly above 1000 °C [26,27]. In the temperature range of ca. 300–700 °C, not only the main physical characteristics of cerium oxide change dramatically, but also its sorption ability and reactivity [28]. It is therefore unclear, which characteristics may be related to the polishing efficiency. Controversies exist, for example, around an effect of the particle size and morphology of the polishing agents, or concentration of the polishing slurry [29]. Great advances were achieved in recent time in understanding the chemical aspects of the polishing process, especially in the shallow trench isolation CMP (as reviewed by Srinivasan et al. [30]), where pH control as well as chemical and other additives are frequently used to enhance the polishing selectivity [24,31]. Experiments with oxidizing agents, such as hydrogen peroxide, emphasized the role of the cerium speciation (the Ce(III)/Ce(IV) ratio) in CMP [32,33]. As the polishing efficiency decreased in the presence of hydrogen peroxide, it was deduced that Ce^{3+} rather than Ce^{4+} is effective in the polishing of silicate substrates. This conclusion seems to be correct despite the fact that hydrogen peroxide may exhibit a dual (both oxidizing and reducing) effect on cerium ions [34], as the presence of the Ce^{3+} ions in the polishing slurry (filtrate) was confirmed by UV–vis spectroscopy [24].

Chemical additives may exhibit a multiple effect on the polishing process, as they affect not only the interactions on the polished surface, but also complex interactions of the polishing agents with other components of the polishing slurry and with the polishing pad [35], which make difficult to discriminate between the polishing agent-related effects and other (side) effects. There is still relatively few studies which demonstrate clear relationships between the preparation conditions, the measurable and/or controllable characteristics of the polishing powder and its polishing efficiency [12,14,36,37]. To establish the relations between the preparation route of the ceria-based polishing agent, its physico-chemical characteristics and polishing efficiency, we prepared a series of cerium oxides by calcination of the cerium carbonate precursor at different temperatures ranging from 200 to 1200 °C. The glass polishing efficiency was measured in a simple arrangement without a further treatment (e.g. milling) of the polishing agents and without a chemical modification of the polishing slurry. A set of well-established methods together with some more advanced ones, such as electron microscopy (SEM, TEM/HRTEM) or X-ray photoelectron spectroscopy (XPS), were used for a detailed characterization of the polishing agents. It was proven that the calcination temperature affects both mechanical processes during CMP (mainly by governing the crystallinity of cerium oxide) as well as chemical interactions (mainly by governing the Ce(III)/Ce(IV) ratio).

2. Material and methods

2.1. Preparation of polishing powders

If not stated otherwise, cerous nitrate ($Ce(NO_3)_3 \cdot 6H_2O$) of reagent-grade purity (99.9%, trace metal basis) and ammonium

bicarbonate (99.5%) were used to prepare the ceria-based polishing powders used in this study using a carbonate precursor, and oxalic acid dihydrate (> 99%) was used to prepare the polishing powders using an oxalate precursor. The chemicals were obtained from Sigma-Aldrich (Steinheim, Germany). The carbonate precursor was prepared via the precipitation of an aqueous solution of cerous nitrate (0.2 mol/L) with an excess of ammonium bicarbonate (0.5 mol/L) during stirring; the completeness of the precipitation was checked via reaction with oxalic acid. After adding the final amount of ammonium bicarbonate, agitation continued for one additional hour, and the precipitate was left until the following day. Next, the precipitate was separated by filtration, washed with water and then dried overnight at 110 °C. Certain polishing powders were prepared from the rare earths concentrate originating from Kola apatite [38]; this concentrate was dissolved in nitric acid and further treated in a similar way to the cerium nitrate solution. Cerium oxalate was prepared from the cerium nitrate solution via precipitation with saturated oxalic in a similar way as cerium carbonate. Polishing powders were prepared from these precursors via calcination at pre-determined temperatures between 200 and 1200 °C for two hours with an exception of the experiments, in which an effect calcination time was examined.

2.2. Methods of characterization

A FEI Nova NanoSEM 450 scanning electron microscope (SEM) was used to examine the polishing powders in two modes: secondary electrons (SE) and back scattered electrons (BSE). Typically, a powdered sample was dispersed in ethanol and treated in an ultrasonic bath for 10 min before deposition onto a silicon wafer chip.

High-resolution transmission electron microscope (HRTEM) analysis was performed with an FEI Talos F200X. A regular, 300-mesh copper grid coated with silicon dioxide (SiO_2)/monoxide (SiO) was used during sample preparation.

X-ray diffraction (XRD) measurements were performed using an MPD 1880 diffractometer (Philips). The crystallite sizes were calculated from the diffraction line broadening using the Scherrer formula [39]:

$$a = \frac{K \lambda}{\beta \cos \theta} \quad (1)$$

where K is the shape factor, λ is the wavelength of the applied radiation, β is the broadening of the diffraction line, and θ is the diffraction angle. The Scherrer calculator from the X-Pert High-Score Plus SW package was used for these calculations.

The specific surface area of the sorbents was measured using the BET method (N_2 adsorption) with a Sorptomatic 1900 Carlo Erba instrument.

XPS measurements were performed with a PHI VersaProbe II XPS system (by Physical Electronics) with a monochromatic Al-K α source (15 kV, 50 W) and a photon energy of 1486.7 eV. All spectra were measured in a vacuum of 1.3×10^{-8} Pa at room temperature (i.e., 20 °C). The powder samples of cerium oxide were mounted with double-sided tape in a sealed glove box to minimize possible redox reactions. The analyzed area on each sample was 200 μm in diameter, and the survey scan spectra were measured with a pass energy of 187.850 eV, a binding energy range of 0–1300 eV and an electron volt step of 0.8 eV. For the high-resolution spectra, a pass energy of 23.500 eV and an electron volt step of 0.2 eV were used. Dual-beam compensation was used for all measurements. The obtained spectra were evaluated with MultiPak software (version 9.4.0.7.; Ulvac – PHI, Inc.), and all binding energies were referenced to the carbon peak C 1 s at 284.80 eV.

The particle size distribution was measured using the photo-sedimentation method using a centrifugal particle-size analyzer

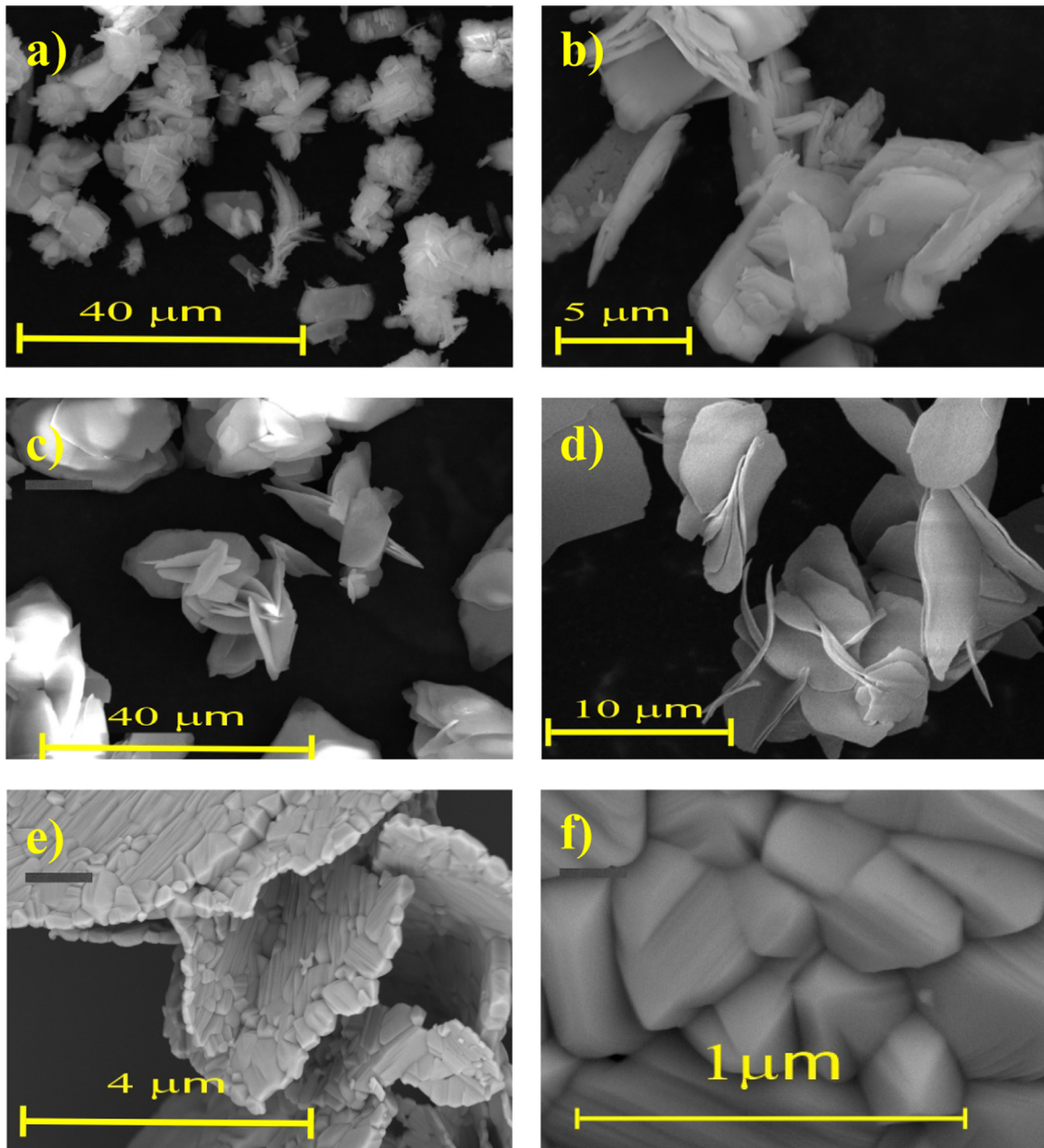


Fig. 1. SEM images of polishing powders prepared from oxalate (a & b) and carbonate (c & d) precursors by calcination at 800 °C. Sub-micron structure of cerium oxide prepared by calcination of cerium carbonate at 1200 °C shown in e) and f).

Shimadzu SA CP-2. The Rosin–Rammler model was used to describe the particle size distribution [40]:

$$R(x) = 100 \exp(-x/x_d)^n \quad (2)$$

$$R(x_d) = 100/e \quad (3)$$

where x_d is the characteristic particle size and n is the grain size exponent.

2.3. Glass polishing measurement

The glass polishing efficiencies of the polishing powders were examined using a Körger polishing machine. The polishing powders were applied in the form of aqueous suspensions with concentrations of 50 g/L, and the polishing pad was made of synthetic Syntepol leather. For each test, three pieces of standard optical glass with dimensions 2 cm × 2 cm × 2 cm were placed in a special

holder, and the polishing test was performed with a polishing rate (i.e., the velocity of the testing glass relative to the polishing pad) of 3.7 ± 0.1 m/s and a pressure of 80 kPa. Before testing, the glass cubes used for the polishing tests were pre-treated by polishing with the highly efficient polishing agent Cerox 1670 (Rhodia, La Rochelle, France) until a perfectly polished surface was achieved. The rate of glass removal was determined via gravimetry from the mass loss during the polishing test, and the results were expressed in terms of a linear rate of glass removal in nm/s. From a series of 8 measurements, the relative standard deviation of the repeatability was estimated to be 7.8%, and the qualities of the polished surfaces were examined by visual inspection.

3. Results and discussion

Cerium oxide prepared by the thermal decomposition of a carbonate or oxalate precursor appears as a pale-yellow free-flowing

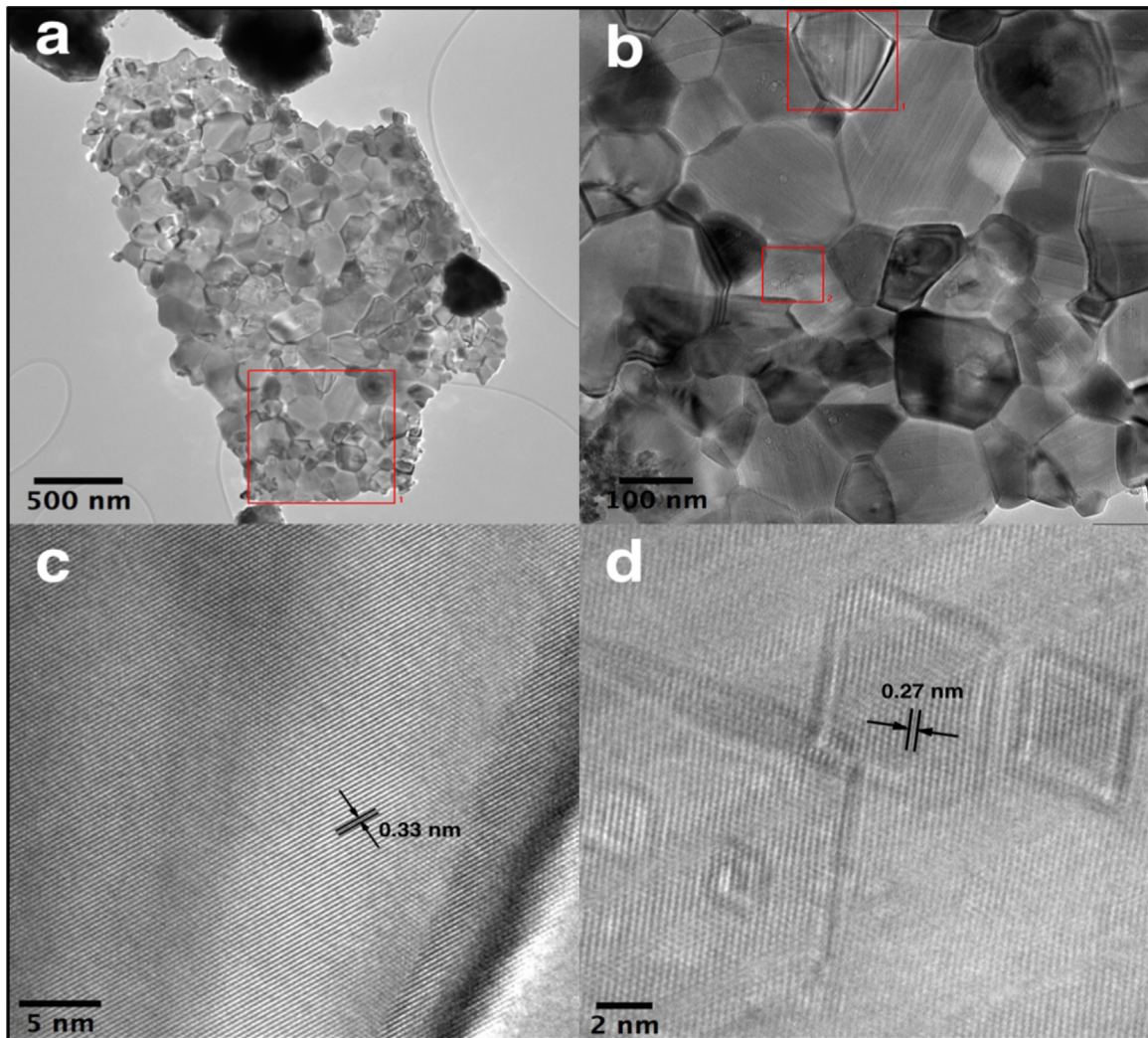


Fig. 2. TEM images of the polishing powder prepared by calcination of cerium carbonate at 1000 °C (a & b). High-resolution images (c & d) of selected areas of image (b) with calculated interspatial distances between adjacent planes (111) in c) and (200) in d).

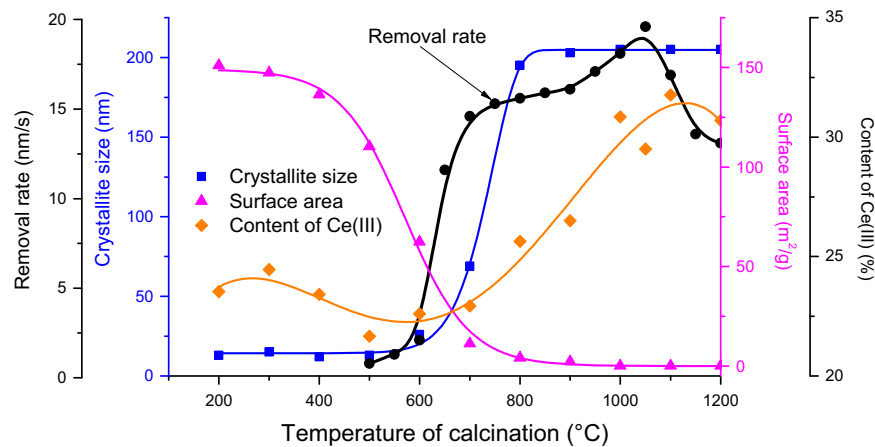


Fig. 3. Dependencies of the glass polishing efficiency (i.e., linear rate of removal), crystallite size, specific surface area and content of Ce(III) on the temperature of calcination.

powder with a relatively complex morphology. As shown in the SEM images (Fig. 1), the polishing powder consists of irregular aggregates, in which thin plates with a typical diameter of several micrometers are assembled together. Previous studies [26,41] reported that the morphology of cerium oxide does not change substantially as the

calcination temperature increases. Additionally, the particle sizes measured by the photosedimentation method varied only slightly from 7.0 to 11.4 μm as the calcination temperature increased and no distinct trend was detected (see [Supplementary material](#) for more details).

However, a more detailed examination with the aid of advanced microscopic techniques revealed a complex sub-micron structure in the polishing powders particles. As shown in Fig. 1e and f, the individual, micron-sized plates of the sample annealed at 1200 °C consist of randomly oriented sub-micron primary particles with well-developed structures that contain various crystal facets and a number of edges, tips and corners. This distinct structure is formed at temperatures above 600 °C, as shown in Fig. S1 in Supplementary material. The precursor annealed at lower temperatures (e.g., 200 and 600 °C) consisted of smooth plate-like particles. Treatment at higher temperatures (e.g., 1000 °C) led to a

Table 1

Comparison of the basic characteristics of polishing powders prepared from oxalate and carbonate precursors^a by calcination at 1100 °C.

Property	Oxalate precursor	Carbonate precursor
Bulk density (g/cm ³)	1.90	0.40
Particle size characteristic (μm)	8.2	10.6
Grain size exponent	1.18	1.15
Specific surface area (m ² /g)	1.20	0.95
Linear rate of glass removal (nm/s)	16.3	12.1

^a Prepared from a concentrate containing ca. 87.3% CeO₂, 6.7% La₂O₃, 5.8% Nd₂O₃ and a trace of Pr.

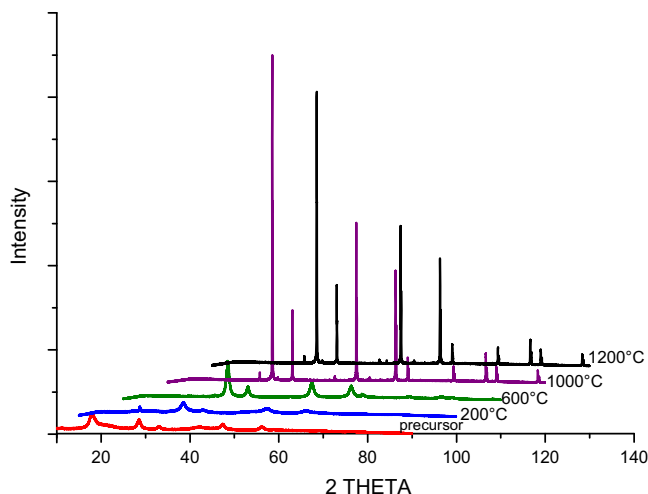


Fig. 4. XRD patterns of the carbonate precursor and cerium oxides annealed at various temperatures.

segregation and growth of individual sub-micron primary particles, which finally developed into a distinct rough surface structure at 1200 °C.

The TEM investigations are in good agreement with the SEM results, as shown in Fig. 2. The thin plate-like particles (Fig. 2b) consist of randomly oriented nano-crystals with various sizes and thicknesses, as shown by the dark and brighter spots (Fig. 2c). The random orientation of the primary crystals was also confirmed by the appearance of various d-spacings in high-resolution images (Fig. 2c,d) of the selected areas. The interspatial distance between adjacent layers $d=0.33$ nm was ascribed to plane (111), and the distance $d=0.27$ nm belonged to plane (200) of cubic cerium oxide.

The polishing tests showed that the efficiency of cerium oxide is strongly influenced by the calcination temperature. When prepared by calcination at temperatures lower than ca. 650 °C, cerium oxide exhibited a poor polishing efficiency both in terms of the removal rate and the quality of the polished surface; conversely, the cerium oxides prepared by calcination at temperatures in the range of 700–1200 °C exhibited a good glass-polishing efficiency. The respective dependence of the linear rate of removal on the calcination temperature is shown in Fig. 3. The properties of the cerium oxides prepared from carbonate and oxalate precursors were comparable with an exception of the bulk density (see Table 1).

The rapid rise in the polishing efficiency in the temperature range of ca. 600 to 700 °C corresponds well to the changes in crystallinity of cerium oxide, as determined from the XRD measurements. The diffraction peaks become sharper and narrower with increasing calcination temperature, demonstrating that the crystalline structure becomes more ordered (see Fig. 4). The crystallite sizes calculated using the Scherrer formula [39] increased from ca. 10 nm for the samples annealed at temperatures below 600 °C to more than 100 nm for the samples annealed at temperatures above 700 °C. These values correspond well with the sizes of the sub-micron particles, as estimated from the TEM images in Fig. 2.

The relation between the polishing efficiency and the crystallinity of cerium oxide may be explained in terms of certain mechanical aspects of the polishing mechanism; larger and more compact crystallites generate greater pressure, and thus, extensive local deformations and phase transformations facilitate the smoothing of the surface [20,42]. However, these mechanical aspects cannot fully explain the observed dependencies and a high quality of the polished surfaces achieved with the polishing powders prepared by calcination at high temperatures. As reported by Peng et al. [43], to obtain a defect-free surface, polishing

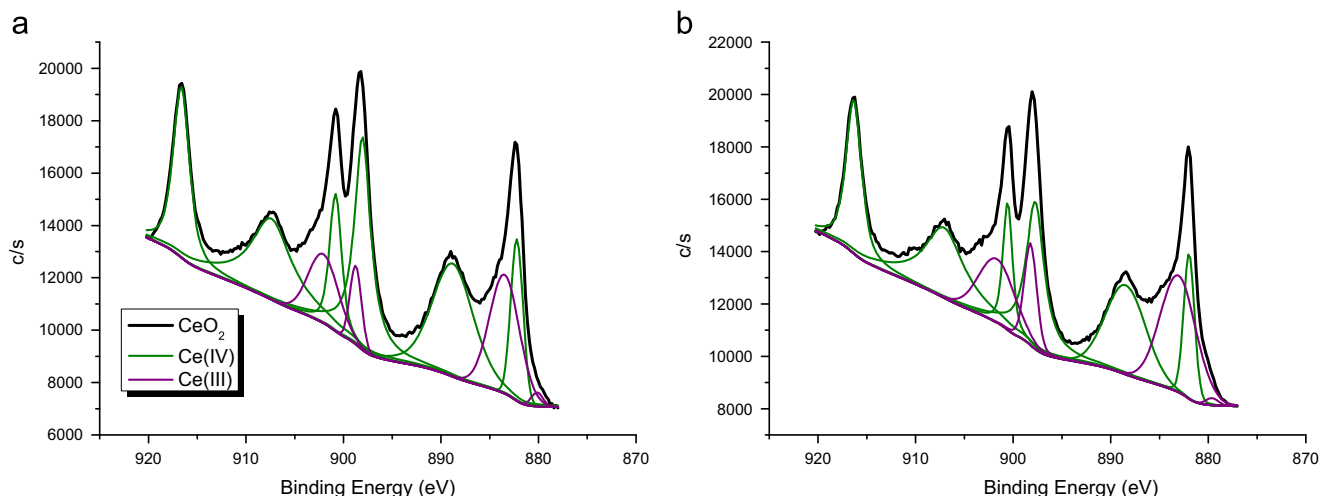


Fig. 5. XPS patterns of the polishing powders annealed at 600 °C (a) and 1200 °C (b).

must be performed in an elastic mode, which requires certain chemical interactions to occur; the presence of the Ce–O–Si bonds were confirmed using FTIR and XPS techniques.

Chemical–mechanical polishing is considered a multi-step process that can be described as follows[7,42]:

- i) A temporary bond is formed between the Ce atoms in the polishing agent and the Si atoms on the polished substrate via surface hydroxyl groups;
- ii) The Si–O bonds are elongated and dissociated; and
- iii) The dissolved silica is removed via adsorption onto the grains of polishing agent.

These steps are facilitated by mechanical stress factors and local phase transformations; however, chemical reactions certainly also play an important role. As mentioned above, the high polishing efficiency was attributed to the presence of the Ce^{3+} ions rather than to the Ce^{4+} ions. Ozawa et al. [44] conducted extensive computer simulations of the CMP process, which confirmed the presence of Ce^{3+} ions at the surface of a ceria-based polishing agent and showed that the Si–O bonds are weakened in the presence of trivalent Ce.

Note that cerium oxide often contains non-negligible amounts of trivalent cerium cations (i.e., as much as 40%) that are responsible for its catalytic and biological activity [45,46]. In the ceria-based polishing agents, the Ce^{3+} ions acts as a reactive species [29] towards the silica surface and also generates the oxygen vacancies and other surface non-stoichiometric effects that further facilitate the dissolution kinetics of cerium oxide [47]. Using XPS techniques, we estimated the contents of the Ce^{3+} ions in the polishing agents in dependence on the calcination temperature; examples of these XPS patterns are shown in Fig. 5. The contents of Ce(III) in the polishing powders ranged from ca. 22 to 32% and increased with increasing calcination temperature especially in the temperature range of ca. 800 to 1000 °C (see Fig. 2).

While an increase in the removal rate for the cerium oxides annealed at temperatures between 500 and 700 °C may be related to the increase in the crystallite sizes (and thus reflects mainly the changes in the mechanical properties of the polishing agents), an increase in the removal rate for the cerium oxides annealed at temperatures about 1000 °C may be related to the Ce(III) content (and thus reflects the chemical reactivity of the polishing agents). A characteristic “hump” on the removal rate vs. calcination temperature curves (although less pronounced) was observed also for the polishing agent containing ca. 82% of cerium oxide [26], which further support the above considerations on the role of trivalent cerium. This hypothesis was supported by correlation tests - the Spearman correlation coefficients were 0.756 and 0.833 for the dependencies of the removal rate on the crystallite size and the Ce (III) content, respectively, considering the cerium oxides annealed at temperatures above 400 °C. The specific surface area (which is strongly correlated with the crystallite sizes - the correlation coefficient being as high as 0.976 in an absolute value) may be also related to the removal rate, whereas the particle sizes, as determined by the photosedimentation method, exhibited almost no influence on the polishing efficiency.

4. Conclusions

Highly effective polishing agents may be prepared via the thermal decomposition of cerium oxalates or carbonates. The glass polishing efficiency depends strongly on the calcination temperature; only cerium oxides prepared at temperatures higher than ca. 700 °C can be used as polishing agents. Conversely, the calcination time plays only a minor role in the final polishing

efficiency, providing that it is longer than ca. 0.5 h; the respective supporting plots are shown in [Supplementary material](#). Strong correlations were found between the polishing efficiency and crystallinity of cerium oxide. The superior polishing efficiency of cerium oxide annealed at 1050 °C was found to be related to the increased content of Ce(III) ions at the surface of the cerium oxide.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at <http://dx.doi.org/10.1016/j.wear.2016.05.020>.

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