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Preparation and sintering pure nanocrystalline α-alumina powder

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Abstract

Seeding of aluminum nitrate solution with ≥0.5 wt.% of the 25 nm α-Al_2O_3 powder markedly accelerates the kinetics of α-alumina crystallization from the gel, the latter being formed in course of addition of ammonia to the solution. Depending on the amount of seeds and the way of their incorporation to the solution the complete transformation occurs from 800 to 930 °C. The resulting powder consists of 50–60 nm crystallites which, in turn, are agglomerated in 10–20 μm species, the size of crystallites being independent of the amount of seeds added. Grinding under mild conditions destroys agglomerates and leads to a nano-powder of which 95–99% dense bodies have been sintered at 1300–1400 °C.

Keywords: A. Sintering; α-Al_2O_3; Nanopowders preparation; Seeding

1. Introduction

Due to the steady growing attention concerning the use of oxide ceramic nanopowders in industry, there is a great deal of research in the field aimed at finding the most effective way of their preparation. Nevertheless, in spite of evident achievements in that direction, α-Al_2O_3 nanopowders are practically unavailable on the market, even though alumina is probably the most widely used ceramic material.

Difficulties in preparation of that powder evolved from fundamental features of alumina synthesis. The succession of physical–chemical transformations leading from the precursor to α-Al_2O_3 almost invariably involves the formation of thermodynamically metastable transition aluminas, that can be shown in the following chain:

$$\text{aluminum salt} \rightarrow \text{boehmite}\,(\gamma\text{-AlOOH}) \rightarrow \gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha\text{-}Al_2O_3$$

Phase transitions up to θ-Al_2O_3 are occurring in a topotactic way that is without large distortion of boehmite lattice but with the formation of porosity as a result of water molecules liberation and subsequent densification of the oxide. As to the last transformation to stable α-phase, it already involves a considerable structure rearrangement and thus requires passing over the high activation barrier to secure nucleation of the new phase.

With nucleation being the rate determining step for the phase transition, the product consists of a relatively small amount of coarse particles which, due to the high transition temperature, are also strongly interconnected within agglomerates. To obtain a nanopowder suitable for further ceramic processing from the strongly agglomerated product one should resort to intensive milling that is impossible with ceramic balls possessing low density and insufficient mechanical strength. When steel or tungsten carbide balls are used for milling alumina, the latter being generally acknowledged as a highly abrasive substance, contamination with wear material cannot be avoided.[1] Furthermore, chemical interaction between the powder being milled and the wear material occurs under intensive mechanical treatment[2] and interferes with or even makes impossible further purification. At present the most pure mechanochemically prepared α-Al_2O_3 nanopowders contain about 0.2 wt.% of both iron and chromium[3] that is unacceptable for majority of up-to-date ceramic materials.

Numerous attempts aimed at preventing from the formation of coarse powder by means of involvement organics to separate sterically growing nuclei of α-Al_2O_3 have been reported.[4–11] It is noteworthy that not all the paper cited contain sufficient and conclusive evidence of phase pure α-Al_2O_3 synthesized and that the use of considerable amount of organic additives makes the whole process far more expensive and well may result in

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contamination with carbon. In order to prevent the forming α-alumina particles from excessive growth during calcination a sol-freeze drying method has also been successfully used and resulted in about 40 nm powder judging from TEM and BET evidence.[12]

For the sake of reducing the size of resulting alumina powder a decrease of its crystallization temperature has been attempted as an alternant route. With this in mind 3 mass% of AlF_3 was mixed with $Al(NO_3)_3{\cdot}9H_2O$ salt[13] and authors claimed that thermal treatment of the dry mixture at no more than 650 °C yield phase pure α-Al_2O_3. Nevertheless comparison of crystallite size taken from X-ray pattern (25–30 nm) with 15 m^2/g specific surface area points to highly aggregated state of the powder and thus makes it hardly appropriate for the preparation of advanced ceramics.

Seeding of the precursor is a common route to decrease crystallization temperature of a substance and it has been attempted for α-alumina powder preparation. As an example, upon taking 10 wt.% of α-alumina powder fraction with mean particle size equal to 0.1 μm the authors were successful in diminishing the temperature of the end product formation from about 1200 °C down to 1025 °C,[14] thus having limited grain growth to afford the powder with particle size in the range 0.1–0.4 μm. Numerous researches of the followers summarized in[15] did not improve the latter results substantially. Probably the most surprising results has then been published in,[16] where with the aid of 100 nm α-alumina seeds and polyethyleneglycol the preparation of as fine as 10 nm α-Al_2O_3 powder announced at 3 min exposure at 900 °C. The authors unfortunately did not present electron diffraction images of the powder and the X-ray pattern cause some doubts in phase purity of the product. Of recent investigations are the use of nano-aluminum as a seeding agent resulted in plate-like particles at 1100 °C[17] and the combined effect of seeding with 5% nano-sized α-Al_2O_3 and adding organics to separate growing particles[18] brought about 30–40 nm α-Al_2O_3 powder from supersaturated sodium aluminate solution. In latter case the temperature of complete $\theta \rightarrow \alpha$-$Al_2O_3$ transformation proved to be 1150 °C assuming that the resulting powder must have consisted of relatively hard aggregates and sodium impurity may also have expected.

It is to be noted that the majority of studies cited above are focused only on the phase purity and the mean crystallite size of the product and the data given either did not contain information related to suitability of the powder for ceramic processing or testified its highly aggregated nature. The only exceptions are[6] where successful sintering of nanostructured α-Al_2O_3 ceramics reported after uniaxial pressing of the 10 nm powder at a pressure of 1120 MPa and sintering at about 1400 °C,[8] where starting from 30 nm powder after 200 MPa CIP and 1400 °C sintering 98.5% relative density has been achieved, and[16] where with 10 nm powder using nearly the same as in[16] conditions ceramics with 99% theoretical density and 0.93 μm mean grain size has been prepared.

Further diminishing the temperature of α-phase formation, compared to that given in,[14] has been attempted in the present paper by the use of 25 nm α-Al_2O_3 seeds prepared mechanochemically.[19] The work has been carried out with the object to develop an inexpensive procedure leading to the nanopowder suitable for ceramic industry.

2. Experimental

Aluminum nitrate of the grade “pure for chemical analysis” containing 9 H_2O molecules and 25% ammonia “chemically pure” were used as starting reagents. As a seeding agent α-Al_2O_3 nanopowder with the mean particle size of 25 nm and Fe content of 0.15 wt.% has been prepared as in.[10] On vigorous stirring the nitrate solution slightly acidified (pH 3.5) water suspension of the nanopowder was dropwise added. The solution then was heated up to 70 °C and the quantity of ammonia estimated on the base of Eq. (1) was dropwise introduced, with the whole mixture being stirred in a continuous manner.

$$Al(NO_3)_3 + 3NH_4OH \rightarrow Al(OH)_3 + 3NH_4NO_3 \qquad (1)$$

The resulting gel was set aside overnight, dried on a hot plate and then subjected to heating in a furnace up to various temperatures with the rate of 5°/min followed by isothermal exposure.

X-ray diffraction analysis was carried out with a DRON-4 diffractometer (CuKα1 radiation, graphite monochromator, rate 0.25–0.5°/min). Relative phase contents and crystallite dimensions were determined by the Rietveld procedure using the Powdercell program, an α-Al_2O_3 sample annealed at 1600 °C being taken as a standard. Since high quality description of the peaks attributed to transition aluminas was rather involved, the quantitative data regarding the percentage of α-Al_2O_3 in the product should be considered as little more than a crude approximation.

The specific surface area of powders was measured according to the BET procedure after drying in the flow of inert gas at 200 °C by means of “Katakon” analyzer, and the density by the use of Quantachrome-MVP-D160E. For size distribution analysis water suspension (pH 3–4) has been made by vigorous ultrasound treatment and then placed in a vial of NICOM 380 DLS nanosizer. Powder electron microscopic studies were performed using the JEM-2000FX2 microscope and sintered nanostructures analyzed with TM-1000 and X-23000BU devices. Polished surfaces were thermally etched at 1250 °C and 1300 °C for 30 min for pellets sintered at 1300 and 1400 °C, respectively. The etching temperature of 1200 °C proved to be insufficient for high quality images and we forced to use 1250 °C though some grain growth was observed at this temperature on the fracture surface. Powder SEM images were kindly supplied by Prof. Yu D. Kotov.

Mechanical treatment was carried out in an AGO-2M planetary mill (Novic Co., Russia) with mill pots lined with zirconia (Huachuang Fine Ceramics Co.) as well as milling balls were made of ZrO_2 by TOSOH Co. The ball to powder mass ratio was about 1:30.

Powders compaction has been carried out by dry uniaxial pressing in a steel die. Pellets have been made of 6.5, 10, and 25 mm in diameter and with the height of 2.5–3 mm. Upon measuring the green density they were sintered in the air in VP04/17 furnace or in Netsch DIL402C dilatometer. Heating rate was

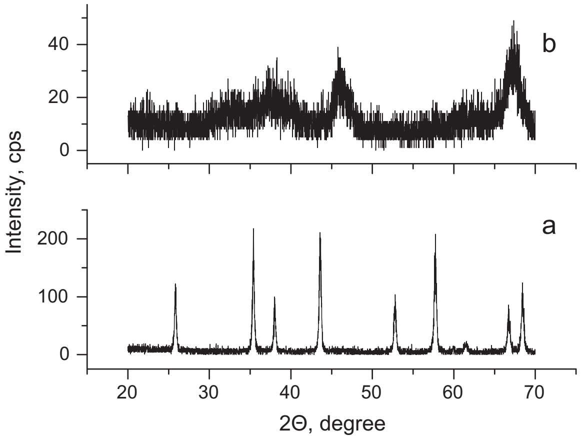

Fig. 1. X-ray patterns of gels with 1.5 wt.% (on dry product) seeds (a), and unseeded (b) annealed for 30 min at 930 °C.

Table 1
Effect of seeds concentration (calculated on dry product) on crystallization and crystallite size of α-Al_2O_3.

Seeds concentration, wt.%	Percentage of α-phase after 870 °C, 20 min	Percentage of α-phase after 910 °C, 20 min	α-Phase crystallite size after 910 °C, 20 min
1	13	68	62
1.5	40	100	61
3	40	100	69
8	42	100	58
10	40	100	55

5°/min and the dwell time 1.5 h. In order to determine the green density the mass was divided by the volume being calculated from geometric measurements whereas sintered densities were estimated by water displacement technique.

3. Results and discussion

3.1. Powder preparation

Fig. 1 represents X-ray patterns of gels, with and without seeds, being heated under similar conditions. The evident difference between the two patterns is that the former gel has transformed in well crystallized α-Al_2O_3 with the mean crystallite size of about 55 nm and specific surface area (*S*) equal to 32–34 m^2/g, the latter corresponding to 44–47 nm particles (*D*) in accordance with formula $D = 1.5/S$; whereas the unseeded gel has transformed to transition aluminas.

The sequence of X-ray patterns taken at various temperatures and consequently corresponding to various stages of the seeded gel transformation indicates ammonium nitrate as being the only crystalline phase after 200 °C followed by amorphous product resulted from its decomposition at 350 °C. A short-term heating at 870 °C gives rise to X-ray peaks attributed mainly to transition aluminas but at 900 °C the dominating phase is α-Al_2O_3. The transition in stable α-phase is still not complete under that conditions which is additionally confirmed by the specific surface area value exceeding 150 m^2/g, the latter can by no means be in agreement with the widths of X-ray responses. Further elevation of the temperature by 30° (or increase of the dwell time at 910 °C) results in X-ray evidence signifying that the α-Al_2O_3 formation is over.

Data given in Table 1 indicate the crystallite size of the resulting oxide as being substantially unaffected by the concentration of seeds introduced in the gel. This observation leads one to the conclusion that seeds do not constitute nuclei of the new forming phase of α-Al_2O_3, since otherwise the final crystallite size had to fall with the increased number of nuclei.

Furthermore, as, for example, with 1.5% of seeds introduced in the gel, to fill up the entire volume of material the growing nuclei must increase their volume by a factor of more than 65, which corresponds to 4 fold increase of their size. Thus, if only artificially introduced seeds are growing, the resulting crystallite size of α-phase cannot be less than 100 nm, which argues clearly with the experiment.

It is also deserved to be mentioned that according to data,[4] in spite of the fact that the size of seeds added has been 100 nm the most of grains being formed at 1025 °C, the degree of conversion being 95% at the moment, were about 50 nm and became 0.1–0.4 μm only by the end of the transformation.

That the seeds themselves are not nuclei for emerging phase is consistent with the microscopic observation that there is a space separation between them and much smaller α-Al_2O_3 nuclei.[20]

As an alternant explanation the seeds may be suggested to exert an influence on the structure of alumohydroxide gel or that of transition aluminas forming around, reducing thus the activation energy of the transformation to α-Al_2O_3. In this case it seems reasonable to expect an increase in efficiency of the matter with increasing the number of seeding particles rather than their mass concentration. That is, the fewer the number of seeding particles the larger will be the distance between them and consequently the lower the probability of α-Al_2O_3 nucleation in the most distant from seeds areas. Therefore, lower number of seeds requires using higher temperature for complete transformation to α-alumina for a particular time interval or longer time if the temperature is predetermined.

That the above hypothesis holds was confirmed by 5.5-h exposure at 850 °C of the gel containing but 0.5 wt.% seeds followed by 8-h exposure at 880 °C. No peaks assigned to transition aluminas can be detected from the resultant X-ray pattern and the crystallite size, despite long thermal treatment of the powder, was estimated to be 50 nm again.

As to the constancy of crystallite size of alumina produced within the temperature range having been studied, the following idea can be put forward. The density of α-Al_2O_3 lattice is markedly higher than that of the gel or transition aluminas. This difference will lead to generation of unavoidable stresses in the matrix of the precursor when α-Al_2O_3 particle is nucleated. Under such a low temperature as we used the relaxation of the stresses is considerably arrested, if possible at all, and will rise in value with the growth of the nucleus until a certain critical limit. Beyond this limit the matrix can no longer withstand the stresses and newly formed alumina particle splits off the matrix and stops growing.

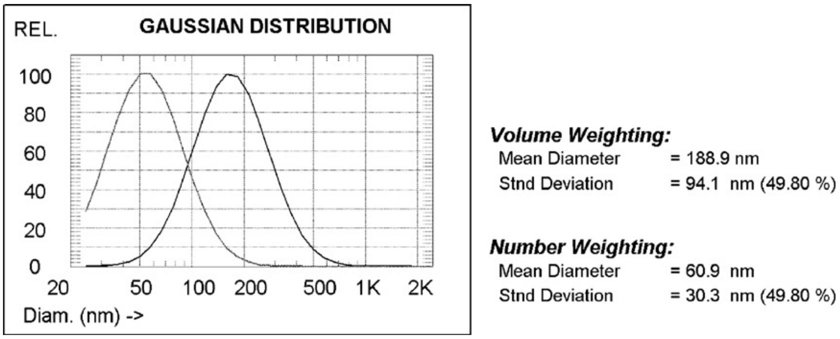

Fig. 2. Number (left curve) and volume (right curve) particle size distribution in α-Al_2O_3 powder obtained at 900 °C and grinded for 30 min at 6 g acceleration.

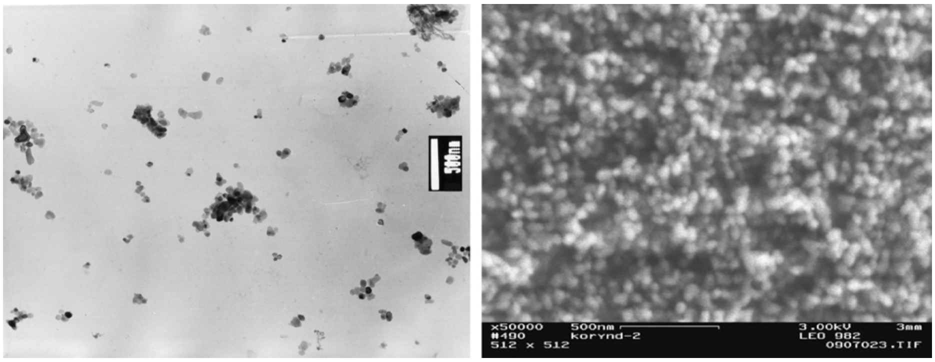

Fig. 3. TEM (left) and SEM images of the powder obtained at 900 °C and grinded for 5 min at 4 g acceleration.

The idea accounts for independence of α-Al_2O_3 particle size of the quantity of seeds added. At low temperatures the rate of diffusional flow is too small to supply enough material for appreciable increase of the size of detached particle during the time of the experiment. It appears that at 1025 °C, that is under conditions adopted in,[4] α-alumina particles having initially the size of 50 nm must have had enough time to grow up to 100–400 nm, whereas at ≤930 °C diffusion is to slow to provide the growth.

One comes to the conclusion that crystallization of α-Al_2O_3 at the temperatures being lower that 950–1000 °C should not be affected by the decrease of seed particle size, the mass of seeds being the same, in terms of the resulting size of alumina particles in the product but the kinetics of phase transition is accelerated with the reduced size of seeds.

It is important to conceive that the effectiveness of a seeding agent is determined not only by the size of composing crystallites, 25 nm in our case, but by the uniformity of crystallite distribution over the precursor of α-phase. Since a dry nanopowder always consists of particle aggregates, the latter do not unavoidable breaking apart in preparation of the water suspension, seeds are likely inhomogeneously distributed in the gel under consideration.

With this in mind, water suspension with seed particles has been subjected to 10 min centrifuging at the acceleration of 2000 × *g*, having thus isolated 10% of the finest seed fraction. It is noteworthy that centrifuging is likely to remove mainly aggregates of seeds because the crystallite size of the finest fraction proved to be slightly larger than that of initial seed sample, 28 and 25 nm, respectively. The fine fraction was added to the nitrate solution in a quantity calculated with the aim of providing 2 wt.% of seeds regarding to α-alumina to be produced. As a result 30 min exposure at no more than 900 °C was enough to convert the gel into 100% α-Al_2O_3 and the mean size of the crystallites in the product was again roughly the same (52 nm) with specific surface area value equal to 32 m^2/g.

We attempted to achieve further improvement of the seeding suspension, regarding the presence of aggregates in it, by subjecting seeds to mechanical treating in the mill with a quantity of aluminum nitrate. In such a manner prepared mixture was further manipulated to the suspension, 10 min treatment with the salt at 15 g having been shown to be ineffective in respect to the seed crystallite size. Such a procedure with 5 mass% of nano-powder being used turned out to be capable of lowering the temperature of 100% transition to α-alumina down to 850 °C for 30 min. The resulting powder possessed specific surface area

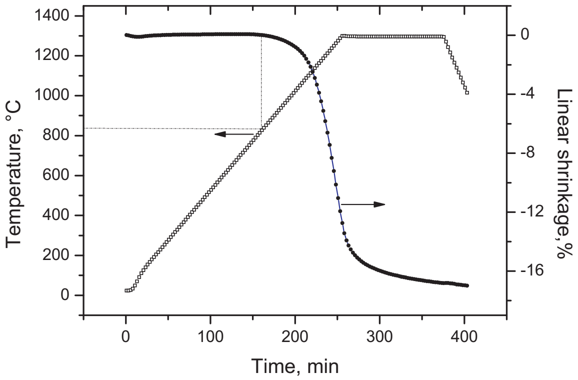

Fig. 4. Dilatometric sintering curve of the powder dry pressed at 230 MPa.

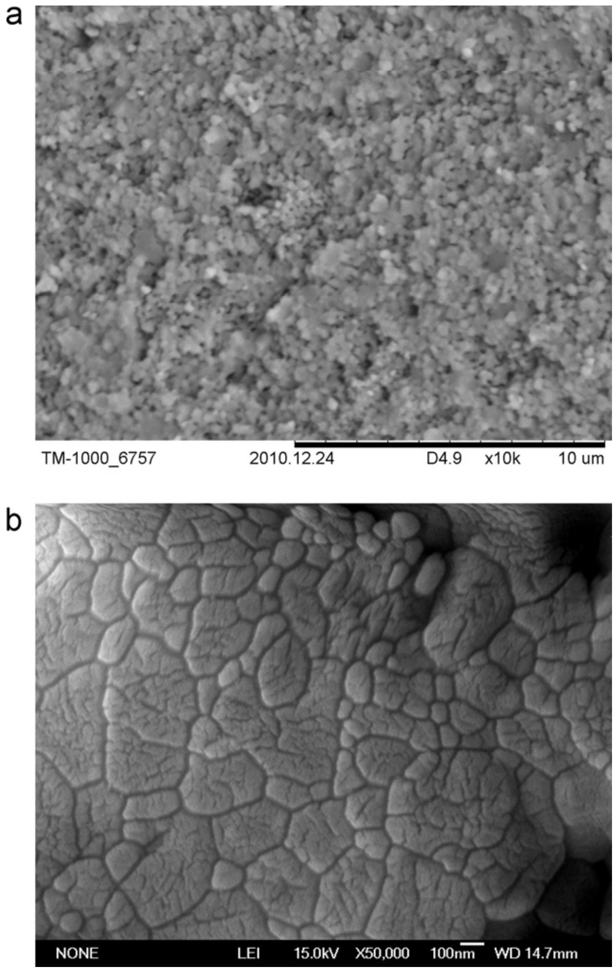

Fig. 5. SEM images of fracture surface (a) and polished and etched section (b) of ceramics sintered of ceramics sintered at 1300 °C.

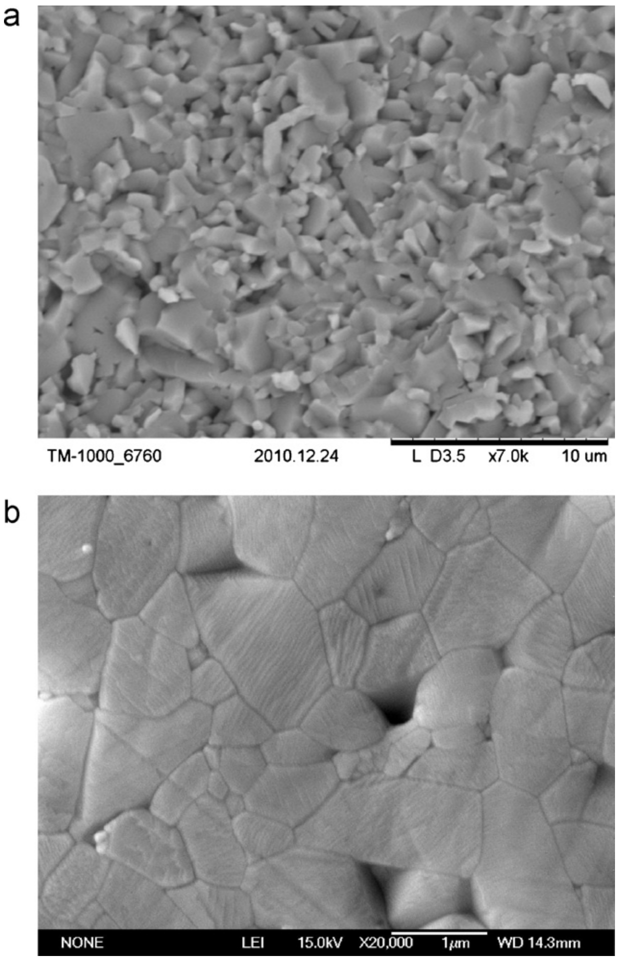

Fig. 6. SEM images of fracture surface (a) and polished and etched section (b) of ceramics sintered at 1400 °C.

value of 32 m^2/g and mean crystallite size of 60 nm. It is of importance that subsequent 30 min annealing at 900 °C exerts no influence on the crystallite size, thus confirming that mass transfer under given conditions cannot provide any notable growth of the particles.

To use 3% of seeds instead of 5% in the last instance decreases the rate of alumina formation and X-ray pattern after 850 °C annealing of the powder detects small broad bands of transition aluminas together with responses attributed to 53 nm α-Al_2O_3. The surface area in this case is also too high (60 m^2/g) to be in fair agreement with α-Al_2O_3 crystallite size. Yet the subsequent annealing of the powder at 900 °C does ensure the completion of the phase transformation without notable affecting the crystallite size.

Thus with properly selected seeding procedure the temperature of a complete transition to α-alumina can be decreased down to 850 °C and just 0.5–1 wt.% seeds is enough to produce α-alumina at temperatures as low as $\leq$900 °C. The resulting oxide consists of nano-crystallites 50–60 nm in size. It comes to clear also that the addition of this amount of a seeding agent makes it possible to prepare a product with the level of contamination at least 0.01 times as much as that in seeds, the latter being 0.15% in our case. Since the technique of purification of the seeds down to 0.02% is available, the method described in the paper removes in reality limitations on the purity of the resulting nanopowder.

3.2. Powder properties

The crystallization product consists of light-yellow spongeous granules 1–3 mm in size. The density of the granules estimated by gas densimetry technique equals to 3.92 g/cm^2 indicating that the closed porosity does not exceed 2%. Particles in the granules being produced at such low temperatures obviously linked to one another by limited number of Al–O–Al bridges which can be easily destroyed. Several minutes grinding in a mortar or in the planetary mill operating at acceleration as low as 4–6 g effect the breakdown of inter particle bridges, the surface area value being nearly unaffected. The size distribution curve of the powder thus produced (the distribution being suggested to fit to a Gauss' function) supports that the majority of the particles have their size in the range of 50–60 nm (Fig. 2), although the distribution is rather broad and the maximum on the volume distribution curve shifts by larger particles. If one analyzes the correlation function without requiring the distribution to be that of Gauss' curve the size distribution becomes

Table 2
Sintered density as a function of sintering temperature, grinding parameters, and compaction pressure.

Mode of grinding	Compaction pressure, MPa	Fractional green density, %	Fractional density after 1300 °C, %	Fractional density after 1400 °C, %
Mortar, 10 min	225	51.7	94.9	94.6
Mortar, 10 min	300	53.2	94.9	97.9
Mortar, 10 min	375	54.5	95.1	97.9
Mill, 5 mm balls, 10 g, 15 min	225	51.4	95.6	99.9
Mill, 5 mm balls, 10 g, 15 min	300	52.7	96.6	99.1
Mill, 5 mm balls, 10 g, 15 min	375	54.2	96.9	
Mill, 10 mm balls, 4 g, 5 min	225	53.2	92.6	94.6
Mill, 10 mm balls, 4 g, 5 min	225	55.2	89.6	94.4

bimodal with 99% of the particles having the size of about 40 nm. The presence of large particles indicates either insufficiency of the grinding time used to completely destroy granules or, which seems more likely, formation of secondary aggregates directly in the suspension. The last assumption based upon the fact that the distribution curves are very much similar whichever time of grinding within the range 2–30 min was used.

Fig. 3 presents evidence by transmission and scanning electron microscopy that unequivocally supports the vast majority of particles constituting the nano-powder as being 50–60 nm in diameter. It can be inferred that the product of low-temperature crystallization does consist of loosely packed granules that undergo ready breakdown into separate ∼50 nm particles by mild mechanical treatment.

Nano-powders produced by grinding the granules can be easily compacted by dry uniaxial pressing to give at about 250–300 MPa rather sinteractive green bodies with 52–54% theoretical density, in contrast to results of[6,8,16] where the powders exhibit quite a poor formability. The onset of intensive shrinkage of the latter has been clearly detectable on the dilatometric curve at 820 °C (Fig. 4) and 1300 °C is enough to achieve 95% fractional density (Table 2). As it is seen from Table 2 the short-term grinding in a mortar appears to be insufficient for all interparticle bridges to be destroyed which affects adversely on the residual porosity at 1400 °C. Whereas grinding in the mill with 5 mm light ceramic balls enables one to yield satisfactory results. At the same time grinding conditions have a marked effect on sintering behavior of the powder that is exemplified by the use of heavier 10 mm balls. Large heavy balls are most likely favors fine particle agglomeration that facilitates the compaction process but, from the other hand, brings about the phenomenon of differential sintering reducing the final density. It seems, as it has been supposed in,[3] the optimal milling conditions of a nano-powder for ceramics should not provide completely disaggregated state since interparticle friction will then hamper compaction. Instead it should result in a powder consisted of "soft" aggregates which facilitates compaction at low pressures and then fall to pieces as the pressure increases.

That the variation in sintering temperature causes drastic alterations in the material microstructure is evidenced by electron micrographs taken from the specimens consolidated at 1300 and then at 1400 °C (Figs. 5 and 6). The size of the grains changes from submicron to micron range that will evidently influence mechanical as well as other functional properties of the material.

To find optimal experimental conditions in terms of obtaining as much sintered density as possible at the lowest temperature had not been the subject of the present work and will be dealt with elsewhere.

4. Conclusions

Addition of 0.5–10 wt.% of 25 nm α-Al_2O_3 powder to aluminum nitrate solution markedly accelerates the kinetics of stable α-phase formation caused by heating the precipitate formed as a result of interaction between the solution and ammonia. The single phase material was obtained at little more than 850 °C if as large as possible number of seeds has been homogeneously distributed in the precursor. The resulted alumina formed within 850–930 °C temperature range consists of about 50–60 nm crystallites, the latter being independent of the amount of seeds introduced. The crystallites constitute loose spongy easily destroyed granules. Grinding of these granules under mild avoiding contamination conditions gives rise to the nanopowder with sufficiently high sinterability to get dense nanostructured ceramics by pressureless sintering at 1300 °C.

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