

ORIGINAL ARTICLE

Advancing spray granulation by ultrasound atomization

 Patrick Höhne  | Bjoern Mieller  | Torsten Rabe

BAM-Federal Institute for Materials Research and Testing, Division 5.5–Advanced Technical Ceramics, Berlin, Germany

Correspondence

Patrick Höhne, BAM-Federal Institute for Materials Research and Testing, Division 5.5–Advanced Technical Ceramics, Unter den Eichen 87, D-12205 Berlin, Germany. Email: patrick.hoehne@bam.de

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Abstract

The influence of the atomization technique on the suitability of granules for dry pressing is the focus of the presented investigations. Therefore, destabilized alumina, zirconia, and zirconia toughened alumina (ZTA) slurries were spray dried and the obtained granules were used to fabricate green and finally sintered bodies for evaluation. Granules made in a laboratory spray dryer with a two-fluid nozzle served as a reference. An ultrasonic atomizer was integrated into the same spray dryer and the influence on the granule properties was evaluated. Untapped bulk density, granule size distribution, and flowability are among the evaluated granule-related properties as well as the granule yield which is used as an indicator of the process efficiency. Yield and flowability as most important granule properties are clearly improved when atomization is realized with ultrasound. The investigated sinter body properties include porosity, sinter body density, and biaxial strength and are as well positively affected by switching the atomization technique to ultrasound. Therefore, the approach to improve the compressibility of granules by ultrasonic atomization, which leads to an improved microstructure, density, and strength of sintered bodies, has proven to be successful for single-component ceramics (alumina and zirconia) as well as for the multicomponent ceramic ZTA.

KEYWORDS

alumina, granules, spray drying, ultrasound, zirconia, zirconia-toughened alumina

1 | INTRODUCTION

Uniaxial or isostatic dry pressing is a simple and efficient way to produce compacts of ceramic materials. Dry pressing facilitates fast production rates, possibilities for automatization and low production costs. But there are certain demands to the raw material, the granules, for satisfying compact quality. The shaping step is of crucial importance, since defects introduced here into the green compacts cannot be reduced, removed, or compensated in the following process. A homogeneous die filling is the first requirement for high-quality compacts. Die filling performance is highly dependent on the

granule's flowability. In general, spherical granules should be within a diameter of about 50 to 100 μm to ensure good flowability.¹ Microstructural defects originating from spray dried granules have been extensively studied earlier.^{2–4} Defects are classified into crack-like and round-shaped defects, residues of hollow granules.² Both originate from incompletely destroyed granules under applied compaction pressure.

To evaluate the effect of granules on the compact quality, a detailed consideration of the granulation process is helpful. Granulation via spray-drying proceeds from a slurry containing the ceramic powder material and organic additives to improve slurry stability and compaction performance. Removal

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of the aqueous phase takes place after the slurry is atomized in a spray dryer. The slurry droplets start drying immediately after atomization, hence the final granule size is already limited by the droplet size. Two drying mechanisms are found in literature. Either the droplet's surface hardens due to material and organic additive migration toward the droplet surface, leading to an early size fixation of this particle,⁵ or the particles maintain the ability to shrink further while drying leading to a lower final size and softer granules. Two distinct granule morphologies result from the two drying mechanisms. The first mechanism described generally leads to larger particles. But more importantly, such granules are hollow and possess a hard densely packed shell which can only hardly be destroyed when dry pressing. In contrast, secondly described particles are smaller, nonhollow, and softer and often lead to a more homogeneous microstructure in sinter bodies.

A previous study⁶ aimed for the improvement of the granule compaction behavior by adjusting the slurry stability. Soft and nonhollow granules can be obtained by spray drying such precisely destabilized ceramic slurries. Since the initial slurry dispersion and processing step has been optimized, the focus shifted to the upcoming atomization step.

Early investigations including ultrasound atomization with a standing wave setup were already published in the mid '90s.⁷ Here, the reduced energy input and fast droplet solidification was highlighted and the applicability for metal melts was presented. For medical applications, ultrasound atomization into an atmosphere of reduced pressure was published for microencapsulation, but allowed only very low slurry feed rates of approximately 1 mL/min for sufficient atomization efficiency.⁵ An extensive study has been published by Ramisetty et al,⁸ who investigated the effect of ultrasound atomization efficiency and classified the atomization into two categories. The correlation between ultrasound atomization parameters such as frequency and power dissipation and slurry properties such as surface tension and viscosity lead to two atomization phenomena: capillary wave controlled or cavitation controlled mechanism related to lower and higher atomization energy, respectively.

Already possessing the ability to produce soft nonhollow granules, investigations of the atomization step were carried out to check for further improvement by atomization with ultrasound. The slurry destabilization as such, is mandatory for furtherly investigating the improvements by the atomization techniques. Therefore, the developed destabilized slurries of alumina, zirconia, and ZTA materials of the former study were the starting point for the presented investigations. Within this study, the effect of the atomization technique on the granule performance is compared between atomization with a conventional two-fluid nozzle and an ultrasound atomizer maintaining most of the drying parameters for both techniques. This study is associated with the low-energy atomization mechanism described by Ramisetty et al⁸ and enhances former studies of droplet formation by investigations

focusing on the atomization technique based property changes of the produced spray dried granules.

2 | EXPERIMENTAL

2.1 | Materials and slurry preparation

Alumina powder AES-11 (Sumitomo Chemical) or/and zirconia powder TZ-3YS-E (Tosoh Corporation) were used as received. Determined raw material properties are listed in Table 1.

Organic additives for slurry preparation and granulation were Dolapix CE64 (dispersing agent), Optapix AC95 (binder), and Zusoplast 9002 (lubricator), all used as received from Zschimmer & Schwarz GmbH & Co KG.

Aqueous slurries of three different powder batches were prepared: alumina, zirconia, and zirconia toughened alumina (ZTA), a mixture of 80 wt% alumina +20 wt% zirconia. Alumina or zirconia grinding bodies were added at a weight ratio of 2:1 (grinding body mass to powder mass) to deionized water (1 μ S/cm) followed by the addition of 0.577 wt% dispersant (as received, related to powder mass). Similarities in particle size and especially specific surface allow weight-based dosing of equal amounts of the surface active organic compounds for both powders. As final component, the ceramic powder was added. Mixing and homogenization of the slurry were conducted in a polyethylene container. Slurries were homogenized for 14 hours with 40 rpm. After removing the grinding bodies by sieving, binder (1.0 wt%) and lubricator (0.5 wt%) were added using a pipette while stirring. Nitric acid (10.6 wt%) was added as last step using a pipette to destabilize the slurry. The alumina slurries were destabilized with $V_{\text{acid}} = 14.9 \mu\text{L/g}$, zirconia slurries with $V_{\text{acid}} = 9.0 \mu\text{L/g}$, and the ZTA slurries with $V_{\text{acid}} = 12.0 \mu\text{L/g}$ (relating to the powder mass).

Although prepared as similarly as possible, the different powder materials led to differences in various slurry characteristics. The solids content, for example, needed to be reduced for both single-component materials to achieve a sufficient degree of destabilization while at the same time

TABLE 1 Properties of alumina and zirconia powders

Powder properties	Alumina (type AES-11)	Zirconia (type TZ-3YS-E)
True density, $\text{g}\cdot\text{cm}^{-3}$	3.94	5.93
Specific surface, $\text{m}^2\cdot\text{g}^{-1}$	6.46	6.71
Mean particle size d_{50} , μm	0.58	0.53
Phase content	$\alpha\text{-Al}_2\text{O}_3$	tetragonal + low amount monoclinic

maintaining a suitable viscosity. In contrast, for the ZTA batches a suitable viscosity and sufficient degree of destabilization has been achieved with less pH adjustments and a higher solids content. The viscosities of the ZTA batches were, despite the higher solids content, comparable with the ones of the alumina batches.

The zirconia batches, on the other hand, had a significantly lower viscosity with the same solids content as alumina slurries. The lower viscosity is due to the lower volume concentration of the solid, since the same mass-based solid concentration results in a lower volume fraction for zirconia than for alumina due to zirconia's higher true density. However, increasing the zirconia powder concentration led to slurries susceptible to agglomeration with minimal pH fluctuations. So, the solids content and thus the viscosity had to be kept so low for safe processing.

Apparently, identical slurry parameters were impossible to realize across the different materials. Nevertheless, slurries for the spray drying experiments were prepared as similar as possible. Recipes were kept identical for each material, leading to slurry compositions that were nozzle independent but necessarily material dependent. More detailed description of slurry preparation and slurry recipe development including the effect of the destabilization were previously published.⁶

2.2 | Granulation processes using spray dryer with different nozzles

Granulation via spray drying was carried out in a laboratory spray dryer (Atomizer Minor, Niro, Søborg/Denmark). The spray dryer was run in fountain mode, using an external mix two-fluid nozzle with a slurry feed orifice diameter of 1.5 mm (access shown at left bottom in Figure 1). Pressurized air was used as atomizing fluid, vortexed within the nozzle by a swirl disk right before exiting the nozzle, resulting in twirl of the atomized slurry.

The same spray dryer was used for granulation with ultrasound atomization. The spray dryer is designed to be run in top feed cocurrent mode as well, in case a rotary atomizing wheel is used. This access was used for the atomization with ultrasound but required further changes in the setup for implementation (US atomizer position marked at spray dryer's top in Figure 1). A fitting element was designed to introduce the ultrasound atomizer into the laboratory spray dryer's lid where the rotary atomizing wheel is usually located. The fitting element mainly consisted of two rings (one fixed at spray dryer and the other holding the nozzle) connected with a corrugated stainless-steel pipe. The corrugated pipe allowed necessary nozzle adjustments in position, angle, and height while sealing at the same time, important to avoid premature drying of the slurry by the hot gas entering through the annular gap around the US atomizer (arrows at spray dryer's top in Figure 1). Dried product was collected at two different

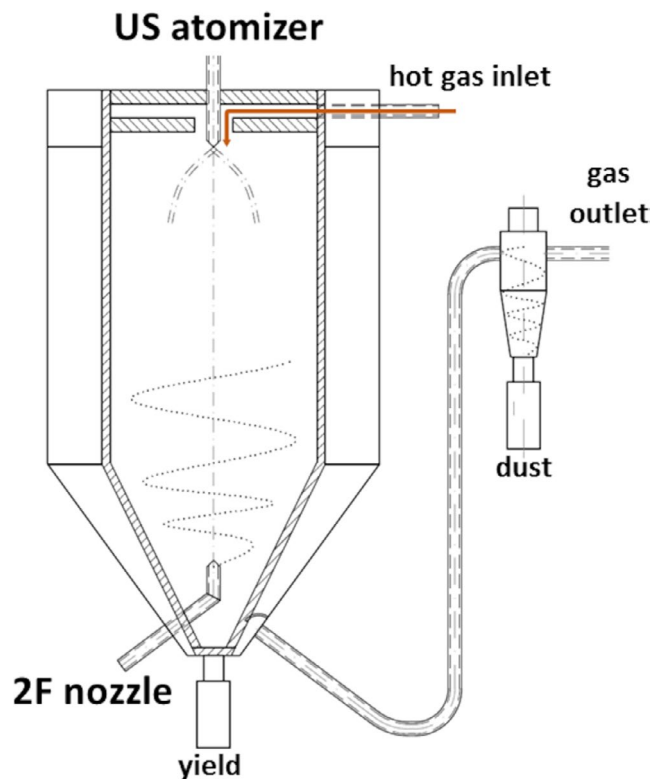


FIGURE 1 Schematic drawing of Niro laboratory spray dryer with atomizer positions, hot air inlet, and product collectors (yield and dust) [Color figure can be viewed at wileyonlinelibrary.com]

positions, depending on the granule size. The coarse granules were collected at the spray dryer's bottom and considered as process yield, while the fine dust-like particles, on the other hand, are carried out by the exhaust gas stream and collected in the cyclone (marked in Figure 1 as yield and dust, respectively).

Constant process parameters for spray drying with two-fluid nozzle were the pressurized air flow rate of 40 L/min, and the spray dryer's gas outlet temperature (100–105°C). The process parameters for spray drying with ultrasonic nozzle were the frequency of 20 kHz and the gas outlet temperature which was kept in the same range as for the spray drying granulation with two-fluid nozzle. The amplitude needed to be adjusted for sufficient nebulization.

2.3 | Shaping and sintering of samples

Spray-dried granules were pressed to cylindrical green compacts with a diameter of 20 mm and a height of about 2 mm with uniaxial pressure of 100 MPa using a hand lever press (Paul-Otto Weber GmbH).

Thermal processes were performed in a box furnace (FHT 16/8, Ceram-Aix), starting with a slow heating rate of 1 K/min and a dwell time of 30 minutes at 450°C for debinding.

TABLE 2 Slurry properties and test parameters for spray drying experiments with two-fluid (2F) and ultrasound (US) nozzle types

Batch composition	Nozzle type	Solids content		Viscosity (at 100 s ⁻¹)	Slurry feed	Amplitude
		wt%	pH	Pa·s	ml·min ⁻¹	%
Alumina	2F	65	7.8	0.220	20	—
	US	65	7.8	0.171	12	83
Zirconia	2F	65	6.6	0.015	15	—
	US	65	6.5	0.016	13	87
ZTA	2F	70	8.3	0.195	20	—
	US	70	7.8	0.304	13	95

Afterwards, temperature was increased with heating rate of 5 K/min up to the maximum process temperature of 1580°C (60 minutes dwell time) for Al₂O₃ and ZTA and 1500°C (30 minutes dwell time) for ZrO₂, respectively. A cooling rate of 5 K/min was hold up to 300°C.

2.4 | Testing procedures

The particle size distribution of starting powders was determined in aqueous dispersions with a Mastersizer 2000 (Malvern). Powders were suspended in deionized water and treated with ultrasound for 3 minutes. The test suspensions were cooled to room temperature before the measurement. The true density was determined by gas pycnometry using Helium (AccuPyc II 1340, Micromeritics). The specific surface area (BET) was determined by measuring the nitrogen adsorption (NOVA 2200, Quantachrome Instruments) and using the skeletal density determined by gas pycnometry. Phase content was measured by X-ray diffraction (Bruker D8).

Slurry viscosity was monitored using a rotational rheometer (Physica MCR 300, Anton Paar).

The size distribution of spray-dried granules was measured by laser diffraction with Mastersizer S (Malvern) using the dispersion unit for dry samples with a feed rate of 20% and a pressure of 0.16 bar. The granule shape was determined with light optical microscope Axiotech 30 (Zeiss). Granule flowability was measured as mass over time with the “Apparatus for determination of bulk density & flow rate” of Landgraf Laborsysteme HLL GmbH using fresh powder for each measurement. Granules used for flowability determination were collected in a cylinder of defined volume to gravimetrically determine the untapped bulk density. Both methods were carried out according to standards, respectively.^{9,10}

The green density of the dry-pressed, cylindrical samples was determined by measuring mass and geometry. The sintered density was measured by Archimedes method.¹¹ Light optical microscope Axiotech 30 was used for microstructural investigations. Evaluation of sinter body cross sections

regarding porosity was carried out by image processing with PxF Workbench software with phase fraction expansion module (PixelFerber).

Biaxial strength of sintered samples was determined with a ball-on-three-balls (B3B) setup.^{12,13} In the B3B test, one surface of a disk-shaped specimen is supported on three balls equidistant from its center. The opposite face is centrally loaded with a fourth ball normal to the orientation of the plane. The sample holder was used in a testing machine (Zwick/Roell) which can apply a maximum force of 5 kN. The preload on each specimen was 30 N and the loading rate was 0.03 mm/s.

3 | RESULTS

Slurries for all granulation experiments were prepared identically as described in the experimental section.

Corresponding slurry properties such as solids content, pH, and viscosity are listed in Table 2. Single material batches were processed with a solids content of 65 wt% while ZTA was processed with 70 wt% solids content. The pH values were around 8 for alumina-based slurries and around 6.5 for zirconia slurries. The viscosities of the alumina and ZTA slurries were 0.17 up to 0.3 Pa·s at a shear rate of 100 s⁻¹ while the zirconia slurries had a much lower viscosity of 0.02 Pa·s at the same shear rate.

Slurries were spray dried in a laboratory spray dryer either with a two-fluid nozzle (abbreviated as “2F”), atomized with pressurized air, in fountain mode or in cocurrent top feed mode, atomized with an ultrasound nozzle (abbreviated as “US”). The slurry feed rate was lower for all ultrasound atomized batches, while the reduction is more pronounced for alumina containing slurries. The atomization in the spray dryer was optically evaluated and was classified satisfying, when no macroscopic droplets were expelled. The ultrasound atomizer required tailored amplitudes for each batch for good atomization results (see Table 2).

The granule yield shown in Figure 2 is the granule mass obtained at the spray dryer's bottom relative to the processed

slurry's solids fraction. The fine dust-like fraction obtained at the cyclone is unsuitable for later shaping by dry pressing and therefore not considered in the granule yield. For all batches, the ultrasound atomization shows higher yields compared to the granulation with two-fluid atomization ranging from an increase of absolute yield from 62% to 77% (zirconia) to an increase from 47% to 78% (ZTA).

The characterization of the spray dried granules is summarized in Table 3. The flowability is clearly improved by the use of the ultrasonic atomizer for all materials. In contrast, the granule's mean particle size and the width of the size distribution as shown in Figure 3 (and listed in Table 3) are inconsistently affected by the atomization technique. The untapped bulk density of the granules is only weakly affected by the used atomization technique, but shows a slightly increasing trend for the ultrasound atomization of single material batches.

The previously developed and published slurry destabilization⁶ led to a reduction of the fraction of donut-like shaped granules. Such donut-like shape is a common granule morphology of spray dried granules but possesses an unfavored hard shell which leads to defects in the final sample body. As exemplarily shown in Figure 4 for alumina, neither the granules produced by two-fluid atomization (left) nor the granules from ultrasound atomization (right) show a relevant fraction of the abovementioned donut-like shaped granules.

Each granule batch was dry pressed as described in the experimental part to produce disk-like sample bodies for strength and density determination. The green compact's densities are only marginally affected by the atomization method without an explicit trend (right column in Table 3).

The sinter body properties are positively affected by the ultrasound atomization. The porosity is reduced for all

materials when the atomization is realized with ultrasound, while this effect is more pronounced for alumina and ZTA materials. The porosity determined by image analysis of the microstructure images (Figure 5) show a final porosity of only 0.1% for both alumina containing materials (Table 4). The zirconia material shows a similar trend, but less pronounced with a final porosity of 0.9%. The sinter body density as a more representative value shows a significant improvement for all materials when the atomization was carried out with ultrasound (Table 4). The corresponding microstructure images for all materials are shown in Figure 5. The images of alumina and ZTA materials (left and right column) visualize the improvement by means of the reduction in pore size and number very well. In contrast, the improvement in pore content and density by atomizing the zirconia slurry with ultrasound is not obvious in the microstructure images (center column).

Presented improvements of the sinter body microstructure are supported by results of biaxial strength measurements for the different materials, which are listed in Table 4. The results of the minimal, mean, and maximum strength of the sintered bodies originating from the ultrasound atomization, show a tendency toward higher values for all materials.

4 | DISCUSSION

The spray drying process involves atomization as a droplet formation step performed using two different techniques. On the one hand a conventional, industrially widespread two-fluid nozzle, on the other hand an atomization with an ultrasound nozzle. The ultrasound atomization led to an increase in the yield of the processable coarse granule fraction. The smallest increase in yield was observed for alumina from 47% to 69%, while the largest increase was achieved for ZTA, where the yield for experiments with ultrasound atomization increased from 47% (two-fluid nozzle atomization) to 78%. At the same time, the unprocessable dust-like fraction, collected in the cyclone of the spray dryer, was reduced. The reduction of the dust-like fraction led to the same production rate despite lower slurry quantity by reduced pump speed (see Table 2). Thus, the efficiency of the spray drying process, where atomization was realized with ultrasound, was higher for all materials.

Granule flowability as important granule property was as well improved by ultrasound atomization for all tested materials. This effect is much more pronounced for the alumina (increased by 57%) and zirconia (increased by 130%) materials than for ZTA (increased by 15%). For alumina and zirconia such improvement could be expected considering the increase of the granule sizes stated in Table 3. For the ZTA material the flowability was as well positively affected, although the measured granule size apparently decreased. Optical microscopy

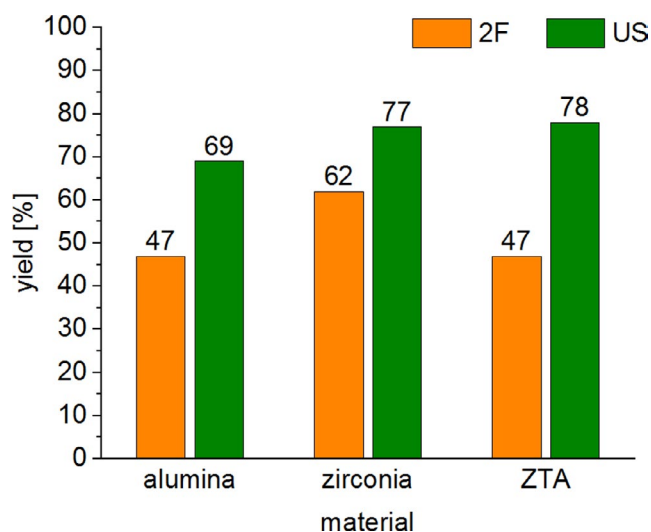
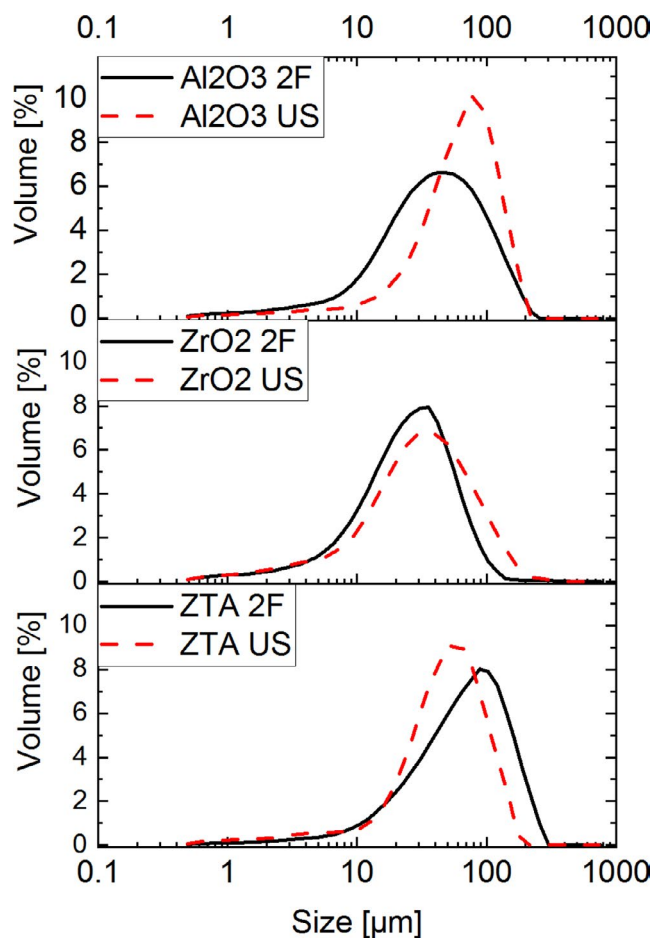


FIGURE 2 Achieved granule yields of the spray drying with different nozzles and materials [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 3 Characteristics of granules and pressed green compacts utilizing two-fluid or ultrasound atomization

Powder composition	Nozzle type	Granules	Granules	Granules	Flowability g·s ⁻¹	Untapped bulk	Rel. green
		d ₁₀ µm	d ₅₀ µm	d ₉₀ µm		density g·cm ⁻³	compact density %
Alumina	2F	10	37	90	0.66	1.10	56.4
	US	21	70	138	1.04	1.17	56.4
Zirconia	2F	7	27	65	0.56	1.43	48.2
	US	7	34	97	1.29	1.49	47.7
ZTA	2F	19	74	170	0.93	1.23	54.9
	US	13	47	100	1.07	1.22	54.6

**FIGURE 3** Size distribution of the spray dried granules [Color figure can be viewed at wileyonlinelibrary.com]

delivered images of ZTA granules which are similar to the alumina ones shown in Figure 4, for two-fluid and ultrasound atomized slurries, respectively. The reduction in the granule size presented in Table 3 is unfortunately affected by the dispersion step of the laser diffraction measurement, leading to the destruction of granules, indicated by an increase of the particle fraction below 1 µm, which is in the same order of magnitude as the primary particles. So, the granules are obviously softer due to the ultrasonic atomization and possess a

narrower particle size distribution as indicated by the images in Figure 4.

The increased flowability means at the same time an improved die filling behavior making the granules more easily processable, leading to more homogeneous green compacts, although the green density is not increased. In contrast, sintered samples made of granules of the ultrasound atomized slurries possess a higher density. The sinter body densities increase by 0.9%, 0.2%, and 1.6% for alumina, zirconia and ZTA, respectively. The microstructures shown in Figure 5 support the improvements in sinter body densities as listed in Table 4. The evaluation of the microstructure by pore fraction analysis of images from optical microscopy shows enhancements for all sinter bodies originating from slurries atomized with ultrasound. The porosities from image analyses deliver porosities as low as 0.1% for alumina and ZTA materials. The porosity calculated from true and sinter body densities delivers porosities of 0.9% for alumina and 0.2% for ZTA materials. As the micrographs (Figure 5) show, the pore sizes are reduced for all materials. The large pores disappear almost completely, and the difference of the pore fraction is probably due to the fact that mainly small pores below 1 µm remain which are not detected in the microstructure evaluation. The zirconia material reaches only down to a porosity of 0.9%. Although this value is higher than the ones for both alumina containing materials, it is already a very good value but indicates that the zirconia material still has potential for improvements that could be addressed by further slurry adjustments. Since the determination of the true density of zirconia is complicated due to the phase transition, from tetragonal to monoclinic, a comparison between optically determined porosity and porosity calculated from true density and sinter density is renounced.

The sinter body's biaxial strength is as well positively affected by the ultrasound atomization and expresses a tendency toward higher strength values for all materials processed with ultrasound. This tendency is given for maximum, mean, and minimum biaxial strength (listed in Table 4). Due to the limited number of specimens, the results of the strength determination can therefore only be seen as a trend.

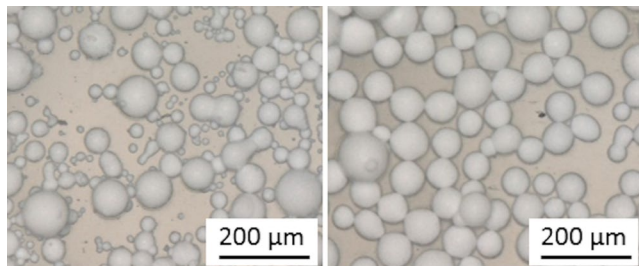


FIGURE 4 Granules of alumina (spray drying with two-fluid atomization left and ultrasound atomization right) [Color figure can be viewed at wileyonlinelibrary.com]

5 | CONCLUSION

The effect of the atomization technique on the suitability of granules for dry pressing, properties of green compacts, and

finally sintered bodies was investigated for alumina, zirconia, and ZTA materials. Therefore, all slurries were atomized in destabilized condition, which is essential as prior slurry optimization step. Granules of the different materials were first produced with a two-fluid nozzle in a laboratory spray dryer and used as reference. An ultrasonic atomizer was successfully integrated into the same spray dryer processing slurries of identical composition for each material.

The relevant granule and sintered body properties were strongly positively affected. Using ultrasonic atomization higher granule yield, better granule flowability, higher sinter body density, reduced porosity, and improved biaxial strength were achieved compared to two-fluid atomization.

Therefore, the strategy to improve the quality of sintered bodies by atomizing with ultrasound has been proven successful for single-component ceramics (alumina and zirconia) as well as for the multicomponent ceramic material ZTA.

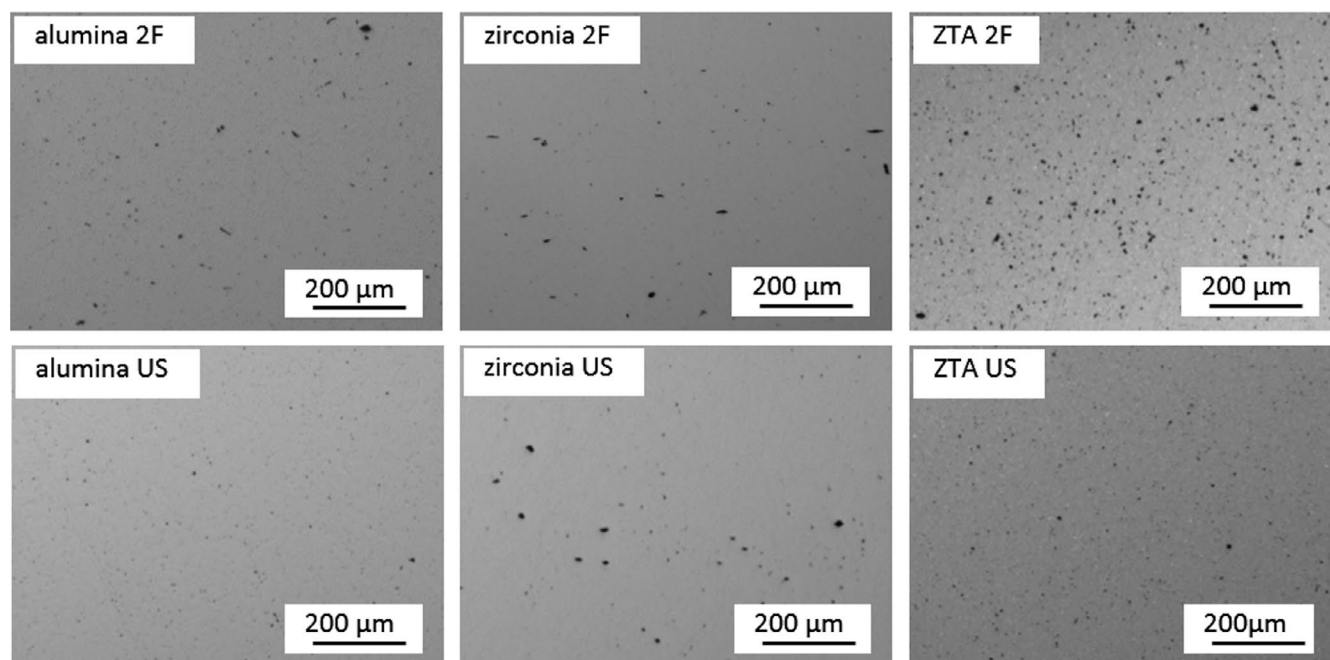


FIGURE 5 Microstructure of sintered alumina (left column), zirconia (center column), and ZTA (right column) samples made of spray dried granules with two-fluid atomization (top row) and ultrasound atomization (bottom row)

	Nozzle type	Porosity %	Sintered density $\text{g}\cdot\text{cm}^{-3}$	Mean strength MPa	Minimal strength MPa	Maximum strength MPa
Alumina	2F	0.7	3.860 ± 0.009	$499 \pm 12\%$	427	556
Alumina	US	0.1	3.904 ± 0.011	$539 \pm 5\%$	502	568
Zirconia	2F	1.2	5.978 ± 0.008	$818 \pm 25\%$	563	1011
Zirconia	US	0.9	5.992 ± 0.005	$980 \pm 12\%$	847	1124
ZTA	2F	0.6	4.163 ± 0.007	$644 \pm 16\%$	519	751
ZTA	US	0.1	4.229 ± 0.009	$725 \pm 15\%$	557	765

TABLE 4 Characteristic values of sintered alumina, zirconia and ZTA sample bodies made of the differently atomized slurries

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ORCID

Patrick Höhne  <https://orcid.org/0000-0003-3963-4062>

Bjoern Mieller  <https://orcid.org/0000-0002-0784-9790>

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