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Fabrication of green and sintered bodies prepared by centrifugal compaction process using wet-jet milled slurries

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Abstract

Properties of green and sintered bodies prepared by high-speed centrifugal compaction process (HCP) using wet-jet milled slurries were investigated. At 30 vol.% solids loading, the relative packing density of compacted body prepared from wet-jet milled slurry was 67%. On the other hand, the density of compacted body prepared from ball-milled slurry was 56% at the same loadings. The difference in relative densities of the top and bottom of green bodies prepared from wet-jet milled slurry was decreased to one-third of the ones prepared from ball-milled slurry. Moreover, the linear shrinkage of the sintered bodies prepared from wet-jet milled slurries was almost constant at sintering temperatures above 1400 °C; it was found to be increasing for bodies prepared from ball-milled slurries with increasing sintering temperature. Furthermore, activation energy for grain-growth was estimated. The grain-growth of green bodies prepared from wet-jet milled slurries was promoted at lower-sintering temperatures.

Keywords: High-speed centrifugal compaction process (HCP); Suspensions; Milling; Al_2O_3; Grain growth

1. Introduction

Colloidal processing of fine ceramic powders is suitable to form dense and homogenous green compacts.[1–4] Especially, slip casting is one of the most common methods for compacting ceramic powder because of its ability to economically produce complicated shapes. However, the gypsum mold that is generally used in slip casting causes contamination of calcium and sulfur from molds into green bodies. Furthermore, it is well known that the casting rate is very slow.

The high-speed centrifugal compaction process (HCP) is a method for compacting fine ceramics or metal powders into various shapes of green bodies.[5–18] HCP provides dense and homogenous green bodies within a relatively short time as compared to other colloidal processing methods. HCP also provides green and therefore sintered compacts with low defects, because bubbles, large particles, and other impurities in slurries are removed under the strong centrifugal force. Therefore, HCP is characterized as a powerful colloidal processing method that leads to ceramic parts with high strength and high reliability. Tashima et al.[6,7] prepared alumina green bodies by HCP which after sintering showed a very high bending strength (1330 MPa) and excellent wear resistance.

However, the drawback of HCP is related to the density gradient during the consolidation stage.[2,13] Considering that the particles in slurry receive a strong centrifugal force during HCP, the heavier particles are settling down at first. If there are flocculated and/or agglomerated particles in a prepared slurry, the fabricated compacts have an inhomogeneous density as well. This phenomenon is the main reason why HCP is not widely used industrially. Furthermore, agglomerated particles also cause lower relative density in green and sintered bodies, higher linear shrinkage during sintering and lower bending strength. Therefore, it is important for HCP to prepare well-dispersed slurry. In general, ball-milling process is widely used for the preparation of ceramic slurries. However, it is known that the ceramic particles after ball-milling have tendency to re-flocculate due to the dominating attractive force which is caused by the increase in the active sites such as lattice defects induced on the surface of particles during ball-milling.[19,20] Therefore, it is difficult to prepare homogeneous green compacts by HCP.

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Recently, in chemical engineering and food technology fields, wet-jet milling has been developed as a new method of mixing and dispersion.[21] In this process, the particles in the slurry or solution collide mutually at high pressure and high speed. In addition, the cavitation, turbulent flow and shear flow are induced at the high speeds. This is how grain refinement, emulsification, and homogenization are achieved within a short period of time. In our previous works,[22–24] it has been demonstrated that the wet-jet milled slurries exhibited distinctly different stability behavior compared to ball-milled ones in terms of re-flocculation efficiency, rheological properties, and packing density. The viscosity of ball-milled slurries increased rapidly with time. On the other hand, the viscosity of wet-jet milled slurries were stable for a long time, indicating that re-flocculation of wet-jet milled particles is low.

In this work, the properties of green and sintered bodies prepared by HCP using wet-jet milled slurries were investigated. The homogeneity of green compacts prepared from HCP was studied. Moreover, the activation energy for grain growth during sintering will also be discussed.

2. Experimental

2.1. Materials and preparation of slurries

A commercially available high purity α-Al_2O_3 (AKP-20; Sumitomo Chemical, Japan) with an average particle size (D_{50}) of 570 nm was used in the present work. A commercially available NH_4^+ salt of poly (acrylic acid) (Aron A-6114, Toagosei Co., Ltd., Japan) was utilized as a dispersant. Aqueous slurries containing 10–50 vol.% of Al_2O_3 powder were prepared with 0.12 wt.% dispersant in distilled water. Optimal dispersant amount was determined from viscosity.[22,23] The slurries were stirred for 5 min before milling.

The slurries were pulverized by wet-jet milling process (PRE03-20-SP; Genus, Saitama, Japan). Fig. 1 illustrates the diagram of wet-jet milling process. Slurries were pumped into the collision unit at 200 MPa where the mutual collision of the particles took place.[22–24] For comparison, the ball-milled Al_2O_3 slurries were also prepared at same solids loading. Ball-milling was performed for 24 h at 60 rpm in a plastic bottle using high-grade Al_2O_3 balls with a diameter of 10 mm. The slurry to ball ratio was 1:2 in volume.

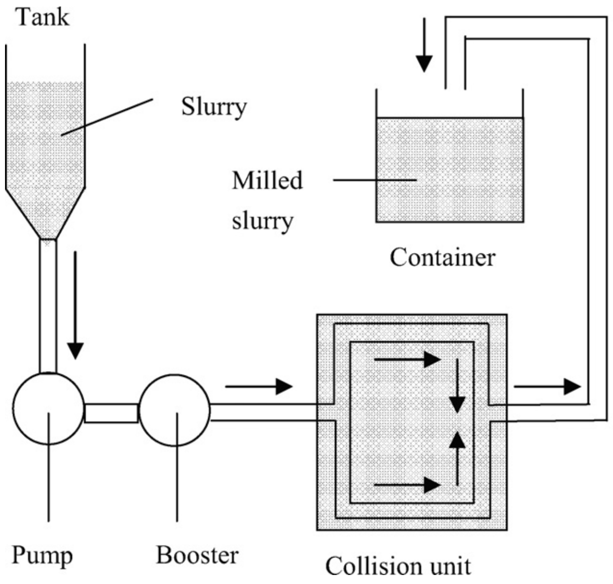

Fig. 1. Diagram of wet-jet milling.

2.2. Preparation of centrifugal compacts

In this work, the centrifugal tube with a diameter of 12 mm and a height of 51 mm was used. The prepared slurries were poured into a centrifugal tube to the height of 27 mm, which are then centrifuged at 57,000 × *g* for 30 min. After centrifugation, the supernatant was poured off, and then the compacted bodies in the centrifugal tubes were dried for 24 h at room temperature. The relative packing density was estimated by the ratio of the volume of green body after HCP and the volume of poured slurry. The relative density of green bodies was estimated by Archimedes method after calcination at 800 °C for 2 h in air. In order to investigate the density gradient of green bodies prepared from wet-jet milled and ball-milled slurries, the relative densities of the top and bottom for green bodies were estimated by Archimedes method. The green bodies were sintered at 1350, 1400, 1450, 1500 and 1550 °C for 2 h in air. The relative density of sintered bodies was also estimated by Archimedes method. The linear shrinkage was estimated from the ratio of compact size before and after sintering. After the surface of sintered bodies was polished, the specimens were thermally etched for 5 min. A field emission scanning electron microscopy (FE-SEM, Hitachi S-4300, Japan) was used to observe the microstructures of sintered compacts. The settling velocity of particles was estimated from the growth rate of clear zone during HCP.[14,15]

3. Results

Fig. 2 shows the appearance of compacted bodies after HCP at 50 vol.% solids loading. The compacted bodies prepared from wet-jet milled slurry showed a lower sediment height compared

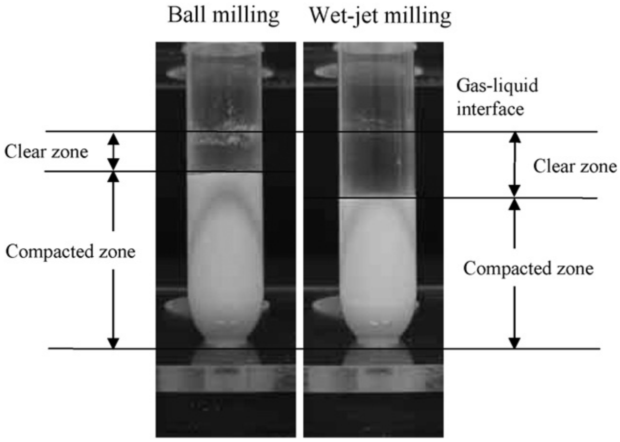

Fig. 2. Appearance of compacted bodies after HCP using ball-milled and wet-jet milled slurries. Solids loading: 50 vol.%.

Table 1
Sediment heights of compacted bodies after HCP prepared from ball-milled and wet-jet milled slurry at the various solids loading

Solids loading (vol.%)	Ball-milling (mm)	Wet-jet milling (mm)
10	7.25 ± 0.07	6.17 ± 0.09
20	11.27 ± 0.16	9.74 ± 0.06
30	14.98 ± 0.07	13.21 ± 0.04
50	21.36 ± 0.18	19.17 ± 0.11

Initial height: 27 mm.

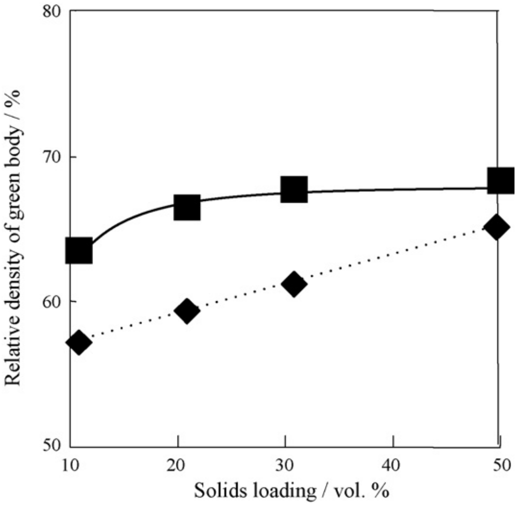

Fig. 3. Relative density of green bodies as a function of solids loading. (Square) Wet-jet milling; (Diamond) Ball-milling.

to the one from ball-milled slurry. Table 1 presents the sediment heights of compacted bodies after HCP prepared from ball-milled and wet-jet milled slurry at the various solids loading. Irrespective of the solids loading, the sediment height of compacted bodies prepared from wet-jet milled slurry was lower than that of ones prepared from ball-milled slurry. Table 2 summarizes the relative packing density of powder compacts, which were estimated by the ratio of the volume of green body after HCP and the volume of poured slurry prepared at various solids loading. The sediments prepared from wet-jet milled slurries have very high relative packing density, as compared to the ones prepared from ball-milled slurries.

Fig. 3 shows the relative density of green bodies prepared from ball-milled and wet-jet milled slurries as a function of solids loading. The relative densities of green bodies prepared from wet-jet milled slurries were significantly higher than those of green bodies prepared from ball-milled ones. The green bodies prepared from wet-jet milled slurries have a relatively constant relative density of about 67%. On the other hand, the relative density of green bodies from ball-milled ones is dependent on the solids loading.

Table 2
Relative packing density of compacted bodies prepared at various solids loading

Solids loading (vol.%)	Ball-milling (%)	Wet-jet milling (%)
10	49.1	60.4
20	52.4	65.9
30	56.2	67.6
50	61.5	71.0

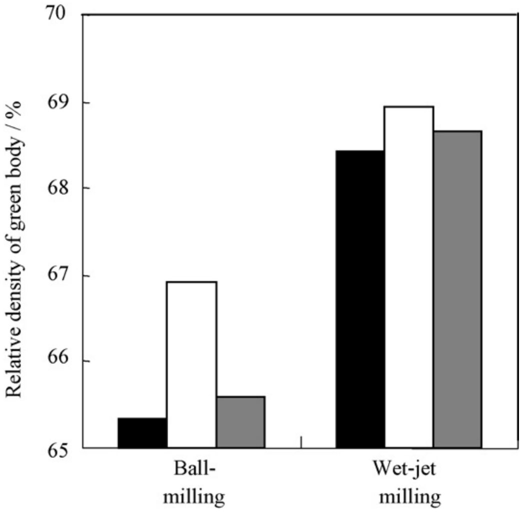

Fig. 4. Relative densities of the top and bottom regions of green bodies. (Black bar) top; (White bar) bottom; (Gray bar) overall of green body. Solids loading: 50 vol.%.

Fig. 4 shows the relative densities of the top and bottom regions of green bodies prepared from wet-jet milled and ball-milled slurries at 50 vol.% solids loading. The relative density of the bottom region of green body prepared from ball-milled slurry was high as compared to the one of the top region. The difference in relative densities of the top and bottom regions was 1.6%, indicating that the density gradient occurred in HCP. On the other hand, the relative density of the bottom region of green body prepared from wet-jet milled slurry was almost similar to the one of the top region. The difference in relative density from top and bottom region was 0.5%. The difference in relative densities of the top and bottom of green bodies prepared from wet-jet milled slurry was decreased to one-third of the ones prepared from ball-milled slurry. Hence, wet-jet milled slurry leads more to homogeneous green compacts.

Fig. 5 shows the relative density of sintered bodies as a function of sintering temperature. To obtain a high relative density above 99%, sintering temperatures exceeding 1500 °C were required in the case of ball-milled green body, whereas wet-jet milled slurries were densified at temperatures as low as 1450 °C.

Fig. 6 shows the linear shrinkage of sintered bodies as a function of sintering temperature. Linear shrinkage of sintered bodies fabricated from ball-milled slurries increased linearly with increasing sintering temperature. On the other hand, the linear shrinkage of sintered bodies prepared from wet-jet milled slurries was very low (12%) and independent of sintering temperature.

Fig. 7 illustrates the microstructures of compacts sintered at 1350, 1400, 1450 and 1550 °C. Sintered ceramics prepared from ball-milled slurry had many pores when sintered at 1350 °C, whereas the ones prepared from wet-jet milled slurries were pore free and easily densified. Irrespective of the sintering temperature, the grain size of sintered body prepared from wet-jet milled slurry was larger than that of sintered body

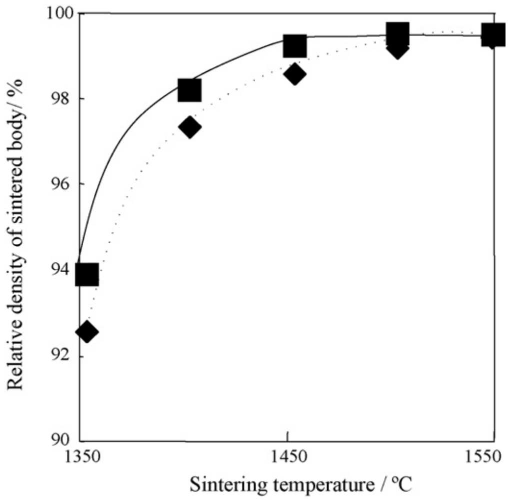

Fig. 5. Relative density of sintered bodies as a function of sintering temperature. (Square) Wet-jet milling; (Diamond) Ball-milling. Solids loading: 30 vol.%.

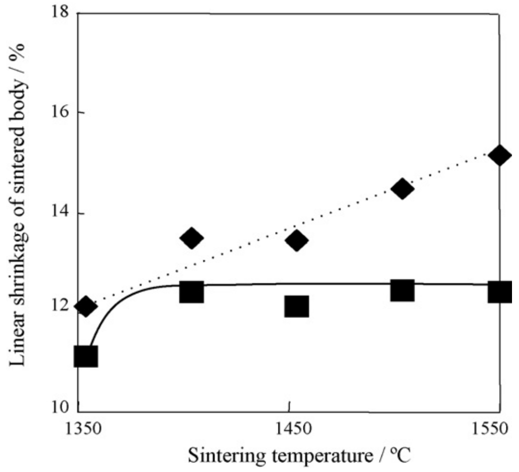

Fig. 6. Linear shrinkage of sintered bodies as a function of sintering temperature. (Square) Wet-jet milling. (Diamond) Ball-milling. Solids loading: 30 vol.%.

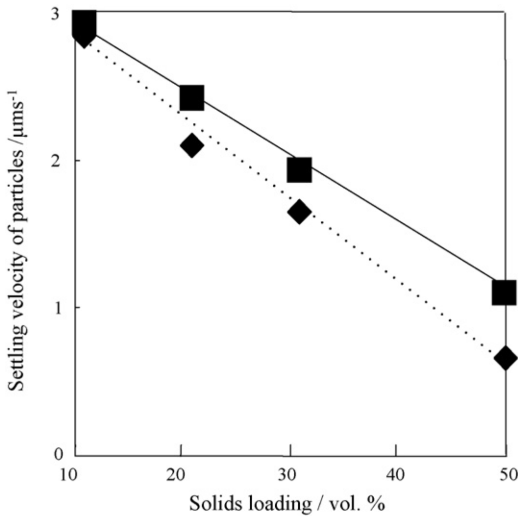

Fig. 8. Settling velocity as a function of solids loading in slurries. (Square) Wet-jet milling; (Diamond) Ball-milling.

prepared from ball-milled one. Hence, green bodies prepared from wet-jet milled slurry were easily densified at lower sintering temperatures. Moreover, wet-jet milled slurry leads to a more homogeneous microstructure, as compared to that of prepared from ball-milled one.

4. Discussion

During HCP, the particles in the slurry are settling down mainly due to the centrifugal force. Fig. 8 shows the settling velocity estimated from the growth rate of clear zone during HCP as a function of solids loading in ball-milled and wet-jet milled slurries. It dramatically decreases with increasing solids loading in slurries. The settling velocity of particles in wet-jet milled slurries is faster than that of ball-milled ones. Suzuki et al. have been reported[9,14,15] that counter flow of medium becomes considerable to compensate down-going flow of particles with increasing solids loading in the slurry. Thus, the

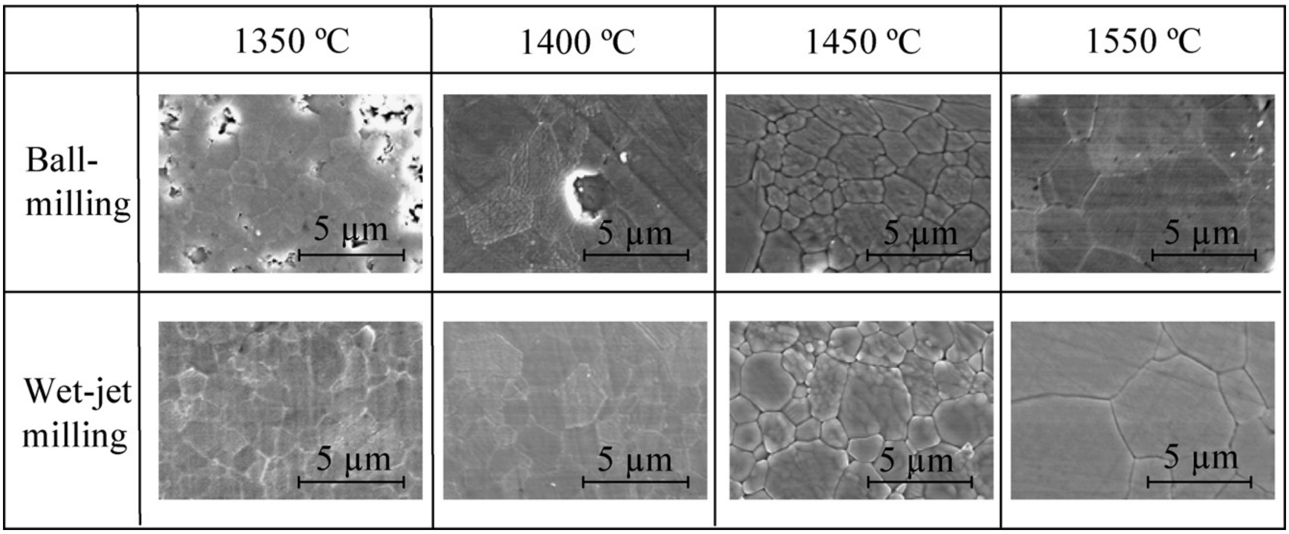

Fig. 7. Microstructure of compacts sintered at 1350–1550 °C.

settling velocity apparently decreases as the solids loading in slurries were increased.

The relationship between settling velocity and solids loading (φ) is represented by Buscall et al.[25] as

$$V_\mathrm{p} = V_0(1-\varphi/\varphi_{\max})\varphi^{\max\times k} \tag{1}$$

where V_p is the settling velocity of particles, V_0 the settling velocity of particles in dilute slurry, φ the solids loading of slurry, $\varphi_{\max}$ the maximum packing density of particles, and k is the empirical constant. Assuming that the particles are close-packed, $\varphi_{\max}$ is 0.74. V_0 in Eq. (1) coincides the equilibrium settling velocity (V) in Stoke's law (Eq. (2))[14,15]

$$V = \frac{(\rho_\mathrm{p}-\rho_\mathrm{m})Cd^2}{18\eta} \tag{2}$$

where V is the equilibrium settling velocity of particles, ρ_p the density of particle, ρ_m the density of medium, C the centrifugal acceleration, d the diameter of particle, and η is the viscosity of slurry. From Eqs. (1) and (2),

$$V_\mathrm{p} = \frac{\{(\rho_\mathrm{p}-\rho_\mathrm{m})Cd^2\}\{(1-\varphi/\varphi_{\max})\varphi^{\max\times k}\}}{18\eta} \tag{3}$$

In previous work,[22,23] we have reported that the particle size distribution of wet-jet milled Al_2O_3 is identical with that of ball-milled one, and the mean particle size is 570 nm. And then, we also demonstrated that the viscosity of wet-jet milled slurries was much smaller than that of ball-milled ones (Table 3). Moreover, the viscosity of wet-jet milled slurries was nearly constant for a long time, whereas viscosity of the ball-milled slurries increased with time. Thus, wet-jet milled slurries were more stable than ball-milled ones. When solids loading in slurries are similar, the settling velocity of particles is determined by only the viscosity (η) in Eq. (3). Therefore, the settling velocity of wet-jet milled slurry is faster than that of ball-milled one during HCP.

The relative densities of green bodies prepared from wet-jet milled slurries were significantly higher than those of green bodies prepared from ball-milled ones. Moreover, the relative density of green bodies prepared from ball-milled slurry was proportional to the solids loading. On the other hand, in the case of wet-jet milled slurry, the relative density of green bodies was steady above 20 vol.% solids loading (Fig. 3). Recently, we have demonstrated by AFM colloid probe measurements that the repulsion force of wet-jet milled Al_2O_3 particle was larger than that of ball-milled one.[23] The larger repulsion forces led to the non-agglomerated state and the stable dispersed slurry. Therefore, the green body with high relative density, which was prepared from the wet-jet milled slurries, was fabricated by HPC.

Table 3
Viscosity of wet-jet milled and ball-milled slurries at various solids loading

Solids loading (vol.%)	Ball-milling (mPa s)	Wet-jet milling (mPa s)
10	12.2	1.42
30	25.1	3.24
50	45.5	11.0

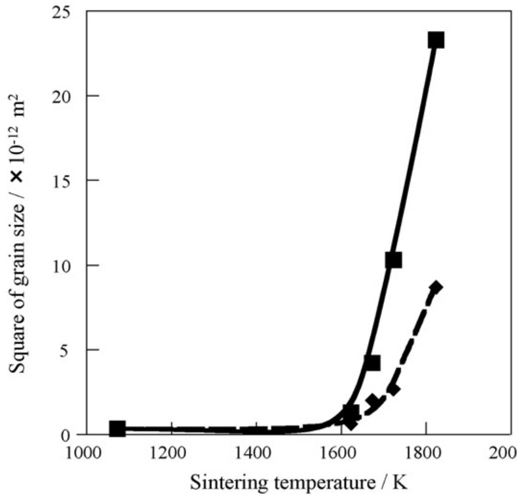

Fig. 9. Square of grain size as a function of sintering temperature. (Square) Wet-jet milling. (Diamond) Ball-milling.

Considering the principles of HCP, the presence of flocculated and/or agglomerated particles in slurry caused the density gradient in compacts (Fig. 4). Therefore, a well-dispersed and stable slurry is strongly required to develop HCP. In the case of ball-milled slurry, the flocculated and/or agglomerated particles in the slurry may settle first. Hence, the difference in relative density of the top and bottom regions for green bodies prepared from ball-milled slurries was large. On the other hand, the difference in the top and bottom regions for green bodies prepared from wet-jet milled slurries was very small, indicating that the density gradient could be decreased. Wet-jet milled slurry leads to homogeneous green compacts on HCP.

The activation energy for the sintering process can be readily determined from the sintering data.[26] Grain growth kinetic is represented as

$$G^2 - G_0^2 = Kt \tag{4}$$

where G is the grain size at sintering time t and G_0 is the initial grain size. When $t = 0$ then, $G = G_0$. In general, $G \gg G_0$ and then Eq. (4) becomes

$$G^2 = Kt \tag{5}$$

In this work, the sintering time was 2 h. So, the grain size is a function of K only.

$$G^2 = 2K \tag{6}$$

In reaction kinetics, K is represented as follows:

$$K = (8\chi\gamma_b V_\mathrm{M}/N_\mathrm{A}h)\exp(-\Delta G^*/RT) \tag{7}$$

where N_A is Avogadro's number, h the Planck's constant, γ_b the surface energy, V_M the molar volume and χ is the distance moved by an atom across the grain boundary. Substitution of Eq. (7) into Eq. (6) reveals the correlation between grain size and sintering temperature (T):

$$G^2 = (16\chi\gamma_b V_\mathrm{M}/N_\mathrm{A}h)\exp(-\Delta G^*/RT) \tag{8}$$

Table 4
Fitted equation and activation energy for grain growth (ΔG^*) estimated from Fig. 9

	Fitted equation	Activation energy for grain growth, ΔG^* (J/mol)
Ball-milling	$Y = 9.3 \times 10^{-2} \exp(-4.2 \times 10^4/X)$	5.1×10^3
Wet-jet milling	$Y = 5.1 \times 10^{-4} \exp(-3.1 \times 10^4/X)$	3.7×10^3

where ΔG^* is the activation energy for grain growth. Fig. 9 shows the relationship between the square of grain size and the sintering temperature for the sintered bodies prepared from ball-milled and wet-jet milled slurries. The data was fitted to the equation, $Y = a \exp(-b/X)$. Table 4 summarizes the fitted equation and the activation energy for grain growth (ΔG^*) in this work. In the case of the sintered bodies prepared from ball-milled slurries, the activation energy for grain growth was 5.1×10^3 J/mol, whereas it was 3.7×10^3 J/mol as for the sintered bodies prepared from wet-jet milled slurries. Hence, the activation energy for grain growth of green bodies prepared from ball-milled slurry was about 1.5 times as compared to the one from ball-milled slurry. Therefore, in the case of wet-jet milled samples, the grain-growth during sintering was promoted as compared to ball-milled ones.

5. Conclusion

In this work, we studied the properties of green and sintered compacts prepared by HCP using wet-jet milled Al_2O_3 slurry. Wet-jet milled slurries showed a lower sediment height as compared to the ball-milled ones at the same solids loading. Green bodies prepared from wet-jet milled slurries have a relatively constant relative density of about 67%. The difference in relative densities of the top and bottom regions of green bodies prepared from wet-jet milled slurry was decreased to one-third as compared to the green bodies prepared from ball-milled slurry, indicating that the density gradient could be decreased by using wet-jet milled slurry. Furthermore, the linear shrinkage of the sintered bodies prepared from wet-jet milled slurries was low and almost constant when sintered above 1400 °C. From the sintering behavior, the activation energy for grain growth of green bodies prepared from wet-jet milled slurry was equal to three-fourth of the one obtained from ball-milled slurry. The most important feature is that, a well-dispersed slurry leads to a homogeneous green body in HCP method. Wet-jet milled slurry is very suitable for HCP.

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