PROCESSING VARIABLES OF ALUMINA SLIPS AND THEIR EFFECTS ON THE DENSITY AND GRAIN SIZE OF THE SINTERED SAMPLE

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ABSTRACT

High densities and small grain size of alumina ceramic bodies provide high strength and better mechanical properties than lower density and larger grain size bodies. The final sintered density and grain size of slip-cast, alumina samples depends greatly on the processing of the slip and the alumina powder, as well as the sintering schedule. There were many different variables explored that include initial powder particle size, slurry solids percent, amount and type of dispersant used, amount and type of binder used, and sintering schedule. Although the experimentation is not complete, to this point the sample with the highest density and smallest grain size has been a SM8/Nano mixture with Darvan C as the dispersant and Polyvinyl Alcohol (PVA) as the binder, with a solids loading of 70 wt% and a 1500 °C for 2 hours sintering schedule. The resultant density was 98.81% of theoretical and the average grain size was approximately 2.5 μm.

INTRODUCTION

Some of the mechanical properties of ceramics have always been attractive to manufacturers. Their hardness, durability, and ability to operate effectively at high temperatures are unsurpassed by any metal, but their brittleness and difficulty in manufacturing complex shapes have been repelling factors to manufacturers.[1]

Slip-casting of ceramics has made it possible to create complex shapes while maintaining the desirable characteristics of the ceramic. Slip-casting is a relatively simple and inexpensive method used to produce ceramics. The alumina powder is dispersed in an aqueous solution then poured into a gypsum mold. The mold removes the water from the solution leaving the powder in a tightly packed green state. Because of the diversity in the sizes and shapes of the molds, the ceramic can take on any number of shapes. Slip-casting can also yield high densities, small grain sized ceramics. These slip-cast ceramics are very close in density and grain size to hot-pressed ceramics.

If the desired high densities and comparable grain sizes can be achieved through slip-casting, the mechanical properties should be comparable to hot-pressed samples, with the added bonus that complex shapes can be achieved at a greatly reduced cost. To achieve the high densities and grain size needed to obtain the wanted mechanical properties, there are many different processing procedures. The variable ingredients and sintering schedules can be applied in any number of combinations. Our purpose in doing this experiment is to find which combinations of processing, ingredients and sintering schedule provides the highest fired density and smallest grain size for slip-cast alumina bodies.

MATERIALS AND METHODS

The different alumina powders used in the experiment were Baikalox SM8 with a primary particle size of 150 nm, Baikalox CR6 with a primary particle size of 250 nm, and NanoPhase Technologies NanoTek with a primary particle size of 30 nm.[2] The different dispersants used were Darvan C and Darvan 821. The binders used were solid Polyvinyl Alcohol (PVA) and Rhoplex B-60A. The water used was consistent throughout and was de-ionized.

To prepare the slurry, the powder was prepared several different ways. It was used with one, or a combination of several, of the following: out of the bag with no further processing; sieved through a –100 mesh sieve; and heat-treated at 800 °C for 4 hours in air. The sieve was used to break up the soft agglomerates of particles and remove the hard agglomerates, both of which can be detrimental during sintering. The heat treatment was used to remove any impurities that may have been in the as-received powder.

The dispersants were added in quantities ranging between 0.5 wt% of solids and 2.0 wt% of solids. The binders were added according to their recommended dilutions. The PVA was dissolved in water before it could be added to the solution at between 0.1-2.0 wt% of solids. In order to dissolve the PVA, the water had to be heated to approximately 80 °C.

The ingredients were added in the following order: water (with binder if PVA was used), binder, dispersant, then alumina powder. After the wet ingredients are added, solid alumina cylinders are added to the mixture to mix them together. The

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alumina powder is then slowly added allowing for complete wetting of the powder after each small addition of powder. Mixing the powder slowly and allowing saturation of the particles between additions allows faster addition of all the powder.

When all the ingredients are added to the mixture the slurry is allowed to roll mix for several hours. After several hours of roll mixing to allow for complete wetting, the slurry is treated with an ultrasonic horn for 3 minutes. This ultrasonic treatment breaks apart all the soft agglomerates in the slurry allowing for a uniform and tightly packed cast piece. The slurry is then allowed to roll mix at least over night.

The gypsum mold is prepared by using water and plaster of paris at 30 parts plaster to 21 parts water. The molds are prepared in the desired shape and allowed to dry completely before use.

Before the slurry is poured into the mold, it is vacuum treated to remove any air bubbles. It is then poured into the mold and left to dry. After the slurry is completely dry, it is placed in a furnace to sinter according to one of the following sintering schedules:

1. Ramp to 800 ° C, hold for 4 hours, ramp to 1500 °C hold for 2 hours, cool down;
2. ramp to 800 °C, hold for 2 hours, ramp to 1500 ºC hold for 1 hour, cool down; and
3. ramp to 800 °C hold for 2 hours, ramp to 1400 °C hold for 2 hours, cool down.

The included 800 °C hold is to burn out the binder in the sample and is ramped at a rate of 10 °C/min. The ramp from 800 °C to the final sintering temperature is between 6 °C/min and 10 ºC/min.

The final sintered densities for each sample were found using Archimedes density principle, provided the density exceeded 92% of the theoretical density.

RESULTS

Our project consisted of making alumina ceramic pieces with the many different combinations of variables. Most of these variables had some effect on the final sintered density of the sample. Table 1 shows the effect of dispersant type on the fired density. Only small differences were observed. However, since Darvan C produced a slightly denser sample, it was used for the remainder of the experiment.

Table 2 shows that the samples with PVA as the binder were ≤ 1 percentage point higher in fired density than samples using B-60A. PVA was used nearly all of the time because of this. However, more complicated shapes held together better with B-60A so it was used in these circumstances.

Table 3 shows that the average particle size of the powder had an impact on the final density of the sample. The CR6 powder was slightly less dense than was the SM8 powder, with the SM8 powder having the smaller initial powder particle size. As with the binder the difference was ≤ 1 percentage point higher in relative theoretical density.

Table 1. Dispersants vs. Density

Sample	Dispersant	% Theoretical Density
Al-PVA-70-C	Darvan C	98.81
Al-PVA-70-821	Darvan 821	98.77

Table 2. Binders vs. Density

Sample	Binder	% Theoretical Density
Al-B60A-0.7	Rhoplex B-60A	97.54
Al-PVA-70-C	PVA	98.81

Table 3. Particle size vs. Density

Sample	Powder	% Theoretical Density
Al-B60A-0.7	CR6	97.54
Al-B60A-SM8-B-15	SM8	98.60

Table 4. Solids wt% vs. Density

Sample	Solids wt%	% Theoretical Density
Al-01-02	50	>92.0
Al-04-0.6	60	96.18
Al-PVA-70-C	70	98.81
Al-B60A-80-C	80	97.74

Table 5. Sintering Schedule vs. Density

Sample	Sintering Temp.	Sintering Time	% Theoretical Density
Al-B60A-SM8-B-14	1400°C	2 hr	95.34
Al-B60A-SM8-B-15	1500°C	1 hr	98.60

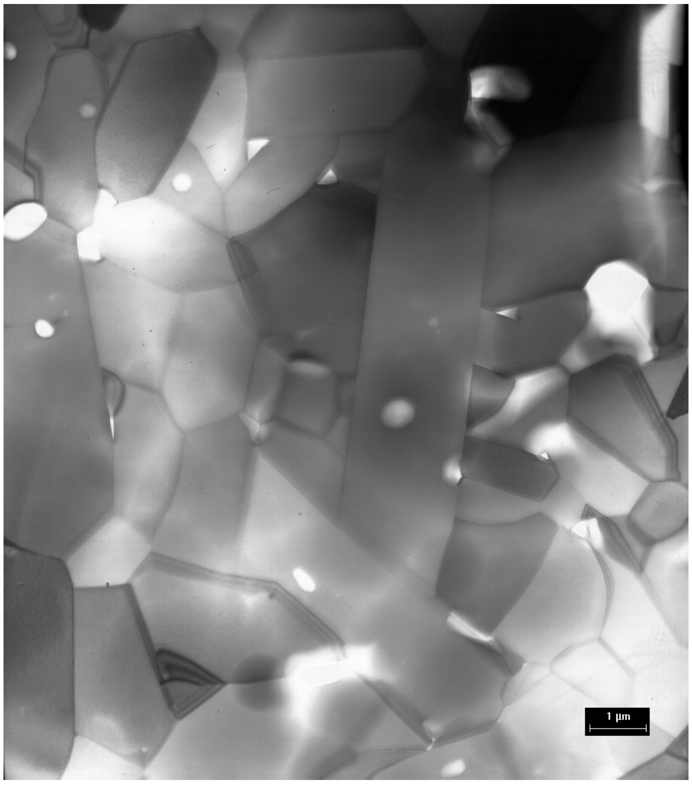

Figure 1. Al-B60A-0.7 with an average grain size of ~5.5mm, % theoretical density of 97.54% and CR6 powder.

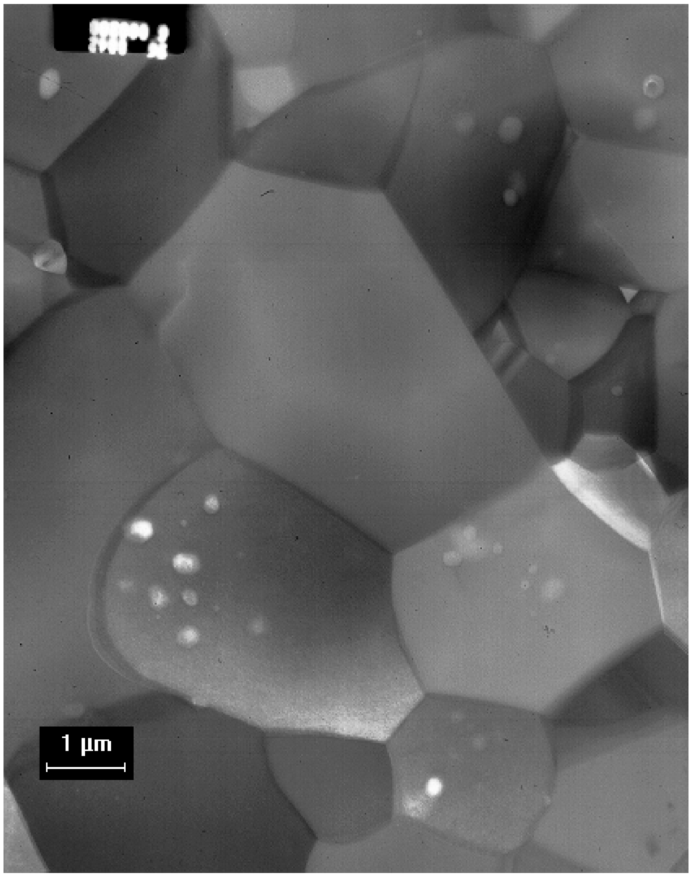

Figure 2. Al-PVA-70-C with an average grain size of ~6mm, % theoretical density of 98.81% and SM8 powder.

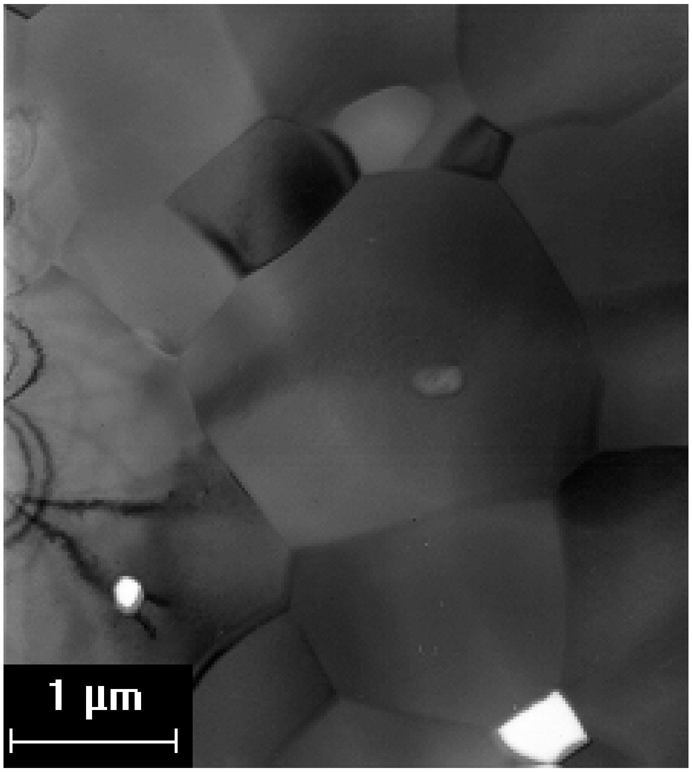

Figure 3. Al-PVA-SMNAN with a % theoretical density of 98.81% and an average grain size of ~2.5 mm.

The slurry solids percent had the greatest impact on the final sintered density of the sample. Table 4 shows that the lower solids percent loading yields a much lower final density, which can be as many as 8 percentage points when the solids loading is dropped to 50 solids wt%. This table also shows that when the solids loading was increased to 80 solids wt% the density dropped ~1 percentage point from 70 solids wt%.

The final variables are the sintering temperature and time. This has an effect on the final density but also has an effect on the sintered grain size. Table 5 shows that the ceramic has a much higher density when sintered at 1500 °C as compared to 1400 °C, the difference being ~3 percentage points of the theoretical value.

The grain size differed between samples, but not enough data has been collected, particularly for the samples in Table 5, to come to any conclusions as to which of the processing variables and sintering schedules provides the smallest average grain size. Several of the discussed samples have been observed through a Transmission Electron Microscope (TEM), and Figures 1 & 2 show their grain structure and average grain size. Also Figure 3 shows a bimodal sample which is the best sample produced to this point in terms of average grain size and density.

The results regarding the heat treatment, sieving, and ultrasonic treatment of the powder showed that these variables produced insignificant effects on the final density.

DISCUSSION AND CONCLUSION

The results of this experiment show that some of the variables make little difference in the final density of the sample, while others make a noticeable difference. Also, some of these differences are easily explained while others are not as clear. The variables that make a difference are the binder used, grain size of the powder, sintering schedule, and solids percent loading. The two dispersants used made very little difference.

From the previously stated results, an optimum processing schedule would be as follows: SM8 aluminum oxide powder at 70 wt% of solids , DI water, Darvan C at 1.5 wt% of solids, PVA at 0.5 wt% of solids , and a sintering temperature of 1500 °C for 1 hour. This schedule would yield the highest density alumina slip-cast sample. Highest density does not necessarily equate to best mechanical qualities, and mechanical testing is on-going to research which of the samples will provide the best mechanical properties.

From the results up to this point, it appears that we will be able to produce high density, small grain size slip-cast alumina ceramics, comparable to hot-pressed alumina samples, and expected to have comparable mechanical qualities. As the research continues, the sintering schedule will be optimized to achieve the needed characteristics and unnecessary processing steps will be eliminated, providing us with a high quality alumina ceramic at a fraction of the cost of hot-pressed alumina ceramics. It will also allow complex shapes to be made, which cannot be made through hot-pressing.

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