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Partical Size Reduction of Si_3N_4 Powder With Si_3N_4 Milling Hardware

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PARTICLE SIZE REDUCTION OF Si_3N_4 POWDER WITH Si_3N_4 MILLING HARDWARE*

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ABSTRACT

The grinding of Si_3N_4 powder using reaction bonded Si_3N_4 attrition, vibratory, and ball mills with Si_3N_4 media was examined. The rate of particle size reduction and the change in the chemical composition of the powder were determined in order to compare the grinding efficiency and the increase in impurity content resulting from mill and media wear for each technique. Attrition and vibratory milling exhibited rates of specific surface area increase that were approximately eight times that observed in ball milling. Vibratory milling introduced the greatest impurity pickup.

INTRODUCTION

Most commercially available Si_3N_4 powders require at least some form of milling to reduce the average particle size to the low micrometer or submicrometer range desirable for sintering. This particle size reduction can be achieved by milling the powder in a ball mill, attrition mill, vibratory mill, or fluid energy mill. The characteristics of these types of mills have been discussed by Greskovich¹ and Pentecost.²

The technique that is selected for milling Si_3N_4 powder will determine the grinding efficiency, the production capability, and the amount of contamination. Although increasing the grinding time leads to a decrease in particle size, it also leads to an increase in impurity pickup from mill and media wear. The rubber lined milling jars often used to mill Si_3N_4 powder

*Originally issued as Technical Memorandum 86864 entitled "Milling of Si_3N_4 with Si_3N_4 Hardware" in 1985.

may have contributed to that contamination.³ The use of a mill or mill liner of the same composition as the powder to be ground should minimize impurity pickup.

The milling of fine powders (<10 μm) is an inefficient process. As milling time progresses the tendency for particles to reaggregate under grinding pressure, the presence of energy absorbing flocs, the semifluid nature of the slurry, and the difficulty in concentrating the grinding energy on a small number of particles at a time lead to a decrease in the probability of a particle being involved in a successful breakage event.⁴ Eventually, a practical grinding limit (~0.1 to 1 μm particle size) is observed.^{1,4} Thus, grinding curves (specific surface area \bar{S} versus time) are expected to plateau after prolonged grinding.

The purpose of this study was to evaluate the milling of Si_3N_4 powder using Si_3N_4 mills and media. It goes beyond the work reported in Ref. 5 to include data on the viscosity of the milling fluids and media wear in ball milling.

METHODS

The Si_3N_4 powder used in this study had the composition shown in Table I. The as-received powder was riffled to eliminate sampling errors and put through a 150- μm sieve. Each powder charge was dried overnight at 100 °C before milling.

Ball milling was done in a reaction bonded silicon nitride (RBSN) jar mill.* It had an 11-cm inside diameter and a volume of 1250 cm^3 . One hundred grams of Si_3N_4 powder were combined with 800 g of 0.95- by 0.95-cm injection-molded RBSN cylinders[†] and 500 cm^3 of milling fluid (6 vol %

*Ceramic Systems, Inc., Detroit, Michigan.

†AiResearch Casting Co., Torrance, California.

solids). The mill was sealed with a rubber cap and rotated at a constant speed of 100 rpm. Samples containing approximately 6 g of powder were withdrawn from the stopped mill at 24, 96, 192, and 288 hr with a glass syringe. Although this sampling method reduced the solids contents of the mill and thus affected the grinding efficiency, all the experiments were equally affected. Ethanol, heptane plus 3 vol % ethanol, and n-butanol were the milling fluids. Single runs were made in heptane plus 3 vol % ethanol and n-butanol. The milling in ethanol was run in triplicate to assess reproducibility.

Vibratory milling was done in the same size RBSN jar mill as ball milling. A Pilamec Megapact laboratory vibrational mill^{*} was modified to hold the RBSN jar mill centered and supported 10.2 cm above the motor. The mill was cooled by opposing air jets. One hundred grams of Si_3N_4 powder were combined with 1600 g of slip cast RBSN 0.95- by 0.95-cm cylinders^{*} and 500 cm³ of milling fluid. The mill was sealed with a neoprene rubber cap secured by a metal hose clamp. The mill was operated at 55 Hz at an amplitude of 0.2 cm. Samples containing 6 g of powder were withdrawn from the stopped mill at 1, 2, 4, 6, and 12 hr with a glass syringe. Ethanol, heptane plus 3 vol % ethanol, and n-butanol were the milling fluids. Single runs were made in heptane plus 3 vol % ethanol and n-butanol. The milling in ethanol was run in triplicate.

Attrition milling was carried out in a stirred attritor as previously described.⁶ The mill was a RBSN pot[†] with a 19.5-cm inside diameter and a 6000 cm³ volume. Four hundred grams of Si_3N_4 powder were combined with 6150 g of sintered, 0.64-cm-diameter, Si_3N_4 balls^{*} and 1800 cm³ of ethanol. Samples containing 6 g of powder were withdrawn from the stopped mill at 1, 2, 4, 6, and 12 hr with a glass syringe. The rotational speeds of the stirring arms

^{*}Pilamec, Clearwell Glos., England.

[†]Wesgo Division of GTE Products Corporation, Belmont, California.

that were compared were 80, 100, and 122 rpm. Single runs were made at 80 and 122 rpm, and the milling at 100 rpm was run in triplicate.

The samples removed from the mills were air-dried in glass Petri dishes. The dried, flaked samples were broken up in an agate mortar and pestle. One-gram specimens were placed in glass bottles for analysis.

Specific surface area was determined by the three-point BET method with a precision of ± 5 percent. Particle size distributions were determined using the Microtrac small particle analyzer.* Particle shape was observed by transmission electron microscopy (TEM). Trace impurities were quantified by inductively coupled plasma (ICP) emission spectrometry. Values are reported at a precision of ± 10 percent. Carbon was determined by heating the sample in an induction furnace and measuring the evolved CO_2 using a gas chromatograph. Oxygen content was determined by fusing the sample, converting the O to CO and measuring CO by infrared spectroscopy. Carbon and oxygen values are reported at a precision of ± 5 percent.

RESULTS AND DISCUSSION

Particle size reduction, as measured by an increase in specific surface area \bar{S} , was linear with time for all three milling techniques, within the range of milling times evaluated in this study. This linear relationship was determined by least-squares regression analysis of the data ($r^2 > 0.95$). Similarly regressed lines are shown in all figures. Figure 1 shows that in ethanol the rates of increase of \bar{S} for attrition and vibratory milling were essentially the same and were about a factor of eight greater than the rate for ball milling. Oxygen pickup with time, again in ethanol, was also linear (Fig. 2) and was greatest with vibratory milling, intermediate for attrition milling, and lowest for ball milling. In terms of new surface created

*Leeds and Northrup, St. Petersburg, Florida.

(Fig. 3), the oxygen pickup rates for ball and attrition milling were equivalent and much lower than for vibratory milling.

Chemical analyses (Table II) indicates that the major impurities in both the as-received and milled powders were oxygen, carbon, iron, and aluminum. Some of the milled powders also contained appreciable amounts of yttrium and magnesium. Other impurities were detected but their change in concentration with milling time was masked by the limit of accuracy of the analysis technique and thus are not reported. The precision of chemical analyses on repeat ethanol runs in the attrition, ball, and vibratory mill was $<\pm 5$ percent. Specific surface area \bar{S} is also included in Table II. The precision of \bar{S} analyses on repeat runs with ethanol in the attrition, ball, and vibratory mill was $<\pm 1$ percent.

Particle size and shape of the as-received Si_3N_4 powder and powders milled in ethanol to equivalent surface areas in the attrition, vibratory, and ball mills are shown in Fig. 4. The lower limit of particle size, as observed by transmission electron microscopy, was $0.02 \mu\text{m}$ in all powder samples. The as-received powder consisted of flakelike individual particles and agglomerates with a maximum particle diameter of $15 \mu\text{m}$. Vibratory, ball, and attrition milled powders were also roughly flakelike with maximum particle sizes on the order of 6, 2, and $4 \mu\text{m}$, respectively.

Differential particle size distribution (PSD) analyses for Si_3N_4 powder milled in ethanol are shown in Fig. 5. The as-received powder exhibited a bimodal distribution with a broad peak at $2 \mu\text{m}$ and a sharper peak at $\sim 6 \mu\text{m}$. The milled powders ($\bar{S} \sim 17 \text{ m}^2/\text{g}$) had much narrower distributions and mean particle sizes of $\sim 1.0 \mu\text{m}$. For particle sizes $< 0.46 \mu\text{m}$ all of the milled powders were quite similar. At the large end of the particle size range, the ball milled powder was finer than attrition milled powder, which, in turn, was finer than the vibratory milled powder. The attrition milled powder was

bimodal with a primary peak at 1.0 μm and a secondary peak at 3.7 μm . Longer ball milling time (288 hr) moves the particle size peak to a lower value and narrows the PSD.

The PSD analysis indicated maximum particle sizes of 15 μm for as-received powder, 2.6 and 5.3 μm for ball and vibratory milled powder, respectively, in any of the milling fluids, and 3.7 μm for attrition milled powder at 100 rpm in ethanol. At 80 and 122 rpm the attrition milled powder had a maximum size of 5.3 μm . The agreement between TEM and PSD for powder milled in ethanol by the three techniques is excellent.

Attrition Milling

Experiments in the ball mill and vibratory mill indicated little difference between milling in heptane plus 3 vol % ethanol and in pure ethanol. Butanol was more contaminating than the other fluids and provided slower grinding rates. Since it was believed that similar results would be observed in attrition milling, the attritor was run only in ethanol and the rotational speed of the stirring arms was chosen as the parameter to vary.

Rotational speed of the stirrer influenced the particle size reduction of Si_3N_4 powder milled in ethanol. At 100 rpm the rate of reduction (grinding rate) as measured by increase in surface area was 1.0 (m^2/g)/hr; at 80 and 122 rpm the grinding rate was of the order of 0.75 (m^2/g)/hr. Based on energy input to the powder charge, an increase in grinding rate with speed would be expected. The increased rate at intermediate speed is due to a more optimum stirring action of the powder slurry-ball charge. Powder size distribution analysis supports the observed rates. More fine particles (<0.25 μm) were present in the powder milled at 100 rpm; more coarse particles (>2.6 μm) were present in powders milled at 80 and 122 rpm.

The primary impurity pickup was oxygen from the oxidation of the new surface created as a result of particle fracture during milling. As shown in

Table II, the amount of oxygen increased with milling speed. In terms of new surface created, however, the lowest oxygen pickup ($0.06 \text{ wt } \%/(\text{m}^2/\text{g})$) occurred at 100 rpm. The increase in yttrium and aluminum content of the milled powder is attributed to wear of the milling media which, as shown in Table I, contained 9.7 wt % yttrium and 1.4 wt % aluminum. Contaminant pickup from the media was roughly equivalent at 80 and 100 rpm and greater at 122 rpm. Although aluminum may be a detrimental impurity in Si_3N_4 , yttrium is a commonly used sintering aid and thus could be compensated for by adjustment of the mill powder charge. Carbon content increased from 0.28 to about 0.7 wt % at all three milling speeds. Retention of milling fluid on the milled powder is the source for the carbon increase. However, most of the carbon can be removed by heating in an inert atmosphere. The very slight (0.01 wt %) increase in iron content of the milled powders is indicative of a very slight, uniform wear of the milling container at all milling speeds.

Vibratory Milling

Changing the milling fluid altered the particle size reduction of Si_3N_4 powder in the vibratory mill. For ethanol and heptane plus 3 vol % ethanol the grinding rate was $1.0 (\text{m}^2/\text{g})/\text{hr}$. For n-butanol, however, the rate dropped to $0.8 (\text{m}^2/\text{g})/\text{hr}$. Particle size distribution analysis shows that along with the lower grinding rate, n-butanol also yields fewer fines ($0.25 \mu\text{m}$) than the other two fluids. The lower grinding rate and lower yield of fines is consistent with an observed greater coating of the media with n-butanol slurries than with ethanol or heptane-ethanol slurries.

As with attrition milling the primary impurity pickup was oxygen. The highest oxygen level (5.2 wt %) occurred with heptane plus 3 vol % ethanol; with ethanol and n-butanol only 4.5 wt % oxygen was measured. Taking into consideration the new surface area created, heptane plus 3 vol % ethanol and n-butanol exhibited an oxygen pickup of $0.3 \text{ wt } \%/(\text{m}^2/\text{g})$, and with ethanol

it was 0.24 wt %/(m²/g). A high grinding rate combined with low oxygen pickup per unit increase in surface area as obtained with ethanol is most desirable.

The oxygen pickup in the vibratory mill is twice as high as in the ball and attrition mills. An increase of this magnitude is clearly not due to either increased mill wear or the increased rate of comminution. Since the vibratory mill is the most energetic mill of the three, more heat is evolved during the grinding process. Although mill temperature was not monitored, we speculate that a higher slurry temperature might lead to greater oxidation of the powder surface.

Table II also shows a significant increase in magnesium content, from 0.01 wt % in the as-received powder to as much as 0.07 wt % in powder vibratory milled in heptane plus 3 vol % ethanol, and a concomitant decrease in aluminum content. An increase in magnesium content combined with a decrease in aluminum content is indicative of wear of the milling media. We believe that the higher media wear associated with the use of heptane is related to the low measured viscosity of this fluid (<0.5 cP) relative to ethanol (1.15 cP) and n-butanol (2.75 cP).

Mill wear is indicated by an increase in iron content. The highest iron value (1.4 wt %) occurred with heptane plus 3 vol % ethanol. It would appear that a low-viscosity milling fluid also promotes higher mill wear. As with attrition milling, the major source of carbon pickup was from the retention of milling fluid on the milled powder. An additional source of carbon in vibratory and ball milling was wear of the mill cap, which was neoprene. The slightly greater carbon content noted in Table II with the use of heptane plus 3 vol % ethanol is associated with a strong reaction between this mill fluid and the neoprene cap. Although most of the carbon associated with the retention of mill fluid can be removed by heating in an inert atmosphere, it

is not known what effect the introduction of rubber from the mill closure will have on samples prepared from the milled powder. It is likely that rubber from the mill cap will produce some form of defects, most likely voids, in samples prepared from the milled powder.

Ball Milling

The effect of milling fluid on the ball milling of Si_3N_4 was investigated in this study. It is recognized that mill speed and powder charge are important parameters in fine particle comminution. In other experiments the maximum grinding efficiency during ball milling was achieved with mill speeds greater than 90 rpm and solids loading in the range 5 to 15 vol %.⁷

Ball milling was the least efficient and therefore the most time consuming means of particle size reduction for Si_3N_4 powder. In all three fluids the grinding rate was $0.12 \text{ (m}^2/\text{g)}/\text{hr}$. At longer milling times (288 hr), however, the grinding rate with n-butanol was slightly lower than for the other two fluids. In terms of particle size distribution, n-butanol yielded more coarse particles ($>2 \mu\text{m}$) and a slightly wider particle size distribution than was observed with either ethanol or heptane plus 3 vol % ethanol. Oxygen contamination of powder prepared with the three fluids increased in the order ethanol, heptane plus 3 vol % ethanol, n-butanol (Table II). However, within the accuracy of the analysis, these differences are not significant. The pickup also increased, from a minimum value of $0.05 \text{ wt \%}/(\text{m}^2/\text{g})$ in ethanol to a maximum value of $0.07 \text{ wt \%}/(\text{m}^2/\text{g})$ in n-butanol. This is similar to the level achieved with attrition milling. For longer milling times (288 hr) the pickup values for ethanol and heptane plus 3 vol % ethanol were the same ($0.05 \text{ wt \%}/(\text{m}^2/\text{g})$), while for n-butanol pickup was slightly greater ($0.06 \text{ wt \%}/(\text{m}^2/\text{g})$). Again, the differences are not significant.

Media wear, in the case of ball milling, was monitored by the change in weight of the media charge. The greatest weight loss occurred with heptane

plus 3 vol % ethanol followed by ethanol and then n-butanol. This is in the same order as observed with vibratory milling.

Mill wear as determined by iron pickup was essentially equivalent in all three fluids with ethanol perhaps yielding a slightly lower amount. As with the vibratory mill, carbon pickup in excess of that from the retained milling fluid was due to wear of the mill cap which in this case was rubber. The larger carbon content of the powder milled in n-butanol is related to a very strong attack and deterioration of the rubber mill cap with this fluid.

SUMMARY

This study was conducted to evaluate the use of Si_3N_4 hardware for the particle size reduction of Si_3N_4 powder by attrition milling, ball milling, and vibratory milling and yielded the following results:

1. Particle size reduction as measured by increase in surface area is linear with time.
2. The attrition and vibratory mill produce new surface area at approximately the same rate, which is about eight times faster than a ball mill.
3. Oxygen pickup is linear with time, occurring at about the same rate with respect to surface area in ball milling and attrition milling. The rate of oxygen pickup is significantly greater for vibratory milling.
4. Mill wear is much greater with the vibratory mill than with either the ball mill or attrition mill.
5. Both ethanol and heptane plus 3 vol % ethanol permit faster particle size reduction than is achieved with n-butanol.
6. For equivalent powder surface area, the size of the largest particles in the final powder increases in the order: ball mill, attrition mill, and vibratory mill. For particle sizes $<0.46 \mu\text{m}$, all of the milled powders were quite similar.

7. Mill and media wear in ball and vibratory milling are highest with a low viscosity milling fluid.

CONCLUDING REMARKS

Based on the results summarized above, we believe that attrition milling offers a fast method for the production of fine Si_3N_4 powders with a minimum amount of detrimental impurity pickup. Improving the quality of the Si_3N_4 mills and media would reduce wear and thus lower the total impurity content of all milled powders.

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TABLE I. CHEMICAL ANALYSIS OF STARTING Si_3N_4 POWDER
AND MILLING HARDWARE (WT %)

	C	O	Fe	Y	Al	Mg
Powder ^a	0.28	1.7	0.56	---	0.36	0.01
Milling ^b containers Media	.04	2.0	3.0	---	.30	.03
Attritor ^c	.006	7.2	---	9.7	1.4	---
Vibratory mill ^b	.02	2.0	.46	---	.25	.20
Ball mill ^d	.03	1.0	.80	---	.20	.01

^aKawecki Berylco Industries, Reading, Pennsylvania - CP85.

^bCeramic Systems Inc., Detroit, Michigan.

^cWesgo Division of GTE Products Corporation, Belmont, California.

^dAiResearch Casting Co., Torrance, California.

TABLE II. - CHEMICAL ANALYSIS AND SPECIFIC SURFACE AREA OF MILLED POWDERS

Type	Time, hr	Speed	Fluid	Chemical analysis, wt %						Specific surface area, S, m ² /g
				C	O	Fe	Y	Al	Mg	
As received	---	-----	-----	0.28	1.70	0.56	-----	0.36	0.01	5.2
Attrition	12	80 rpm	Ethanol	0.73	2.30	0.57	0.20	0.39	0.01	14.2
		100 rpm	Ethanol	.71	2.40	.57	.24	.39	.01	17.0
		122 rpm	Ethanol	.69	2.50	.57	.37	.42	.01	14.9
Vibratory	12	55 Hz	Ethanol	0.80	4.50	1.30	-----	0.30	0.04	17.1
			Heptane	1.00	5.20	1.40	-----	.27	.07	18.2
			Butanol	.78	4.50	1.20	-----	.31	.03	14.6
Ball	96	100 rpm	Ethanol	0.73	2.20	0.60	-----	0.33	0.01	16.8
			Heptane	.72	2.40	.63	-----	.36	.01	17.3
			Butanol	1.00	2.50	.62	-----	.35	.01	16.3
Ball	288	100 rpm	Ethanol	1.20	3.00	0.62	-----	0.34	0.01	31.6
			Heptane	1.30	3.00	.70	-----	.34	.01	32.0
			Butanol	1.80	3.20	.64	-----	.35	.01	28.5

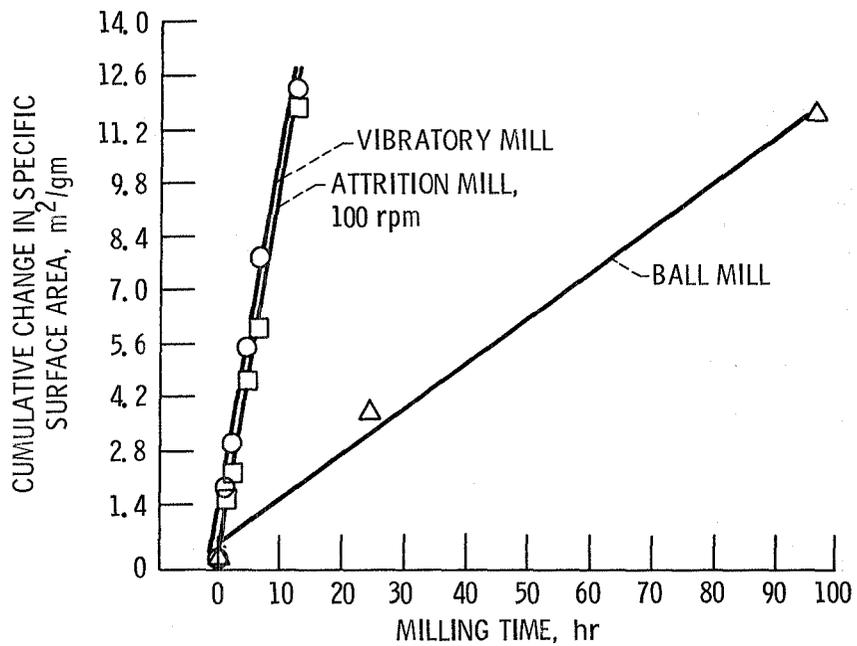


Figure 1. - Cumulative change in specific surface area (\bar{S}) as a function of milling time in ethanol.

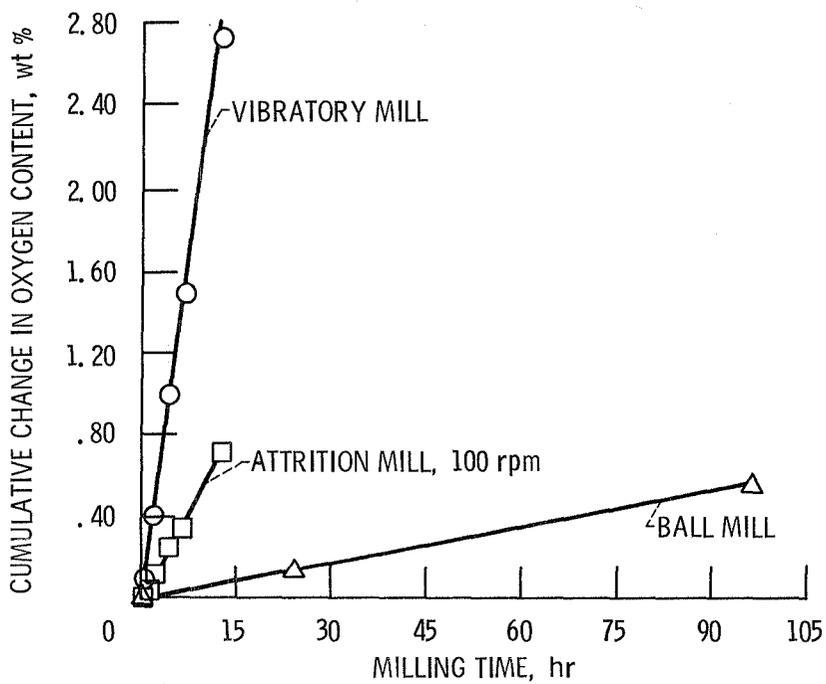


Figure 2. - Cumulative change in oxygen content as a function of milling time in ethanol.

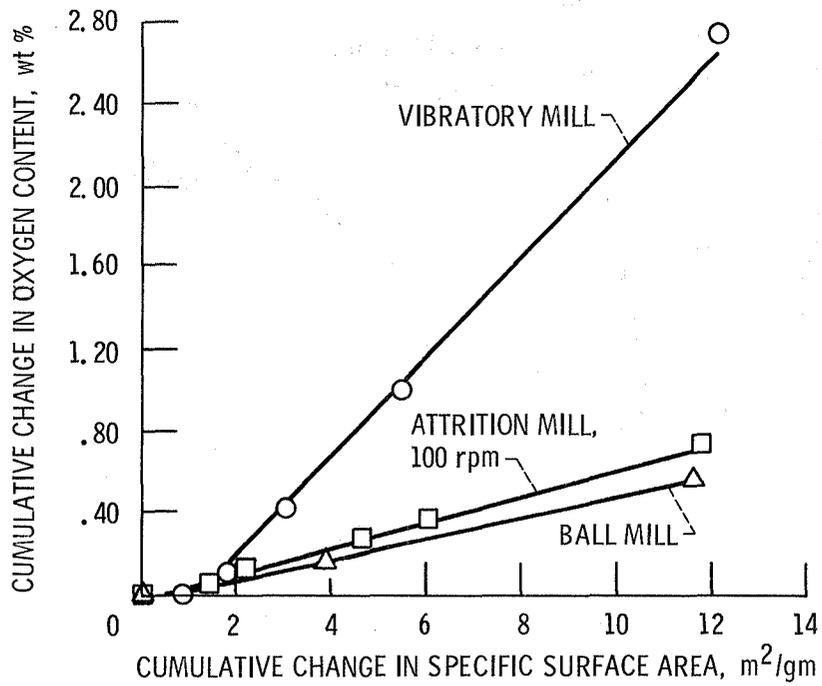


Figure 3. - Cumulative change in oxygen content as a function of cumulative change in specific surface area (\bar{S}).

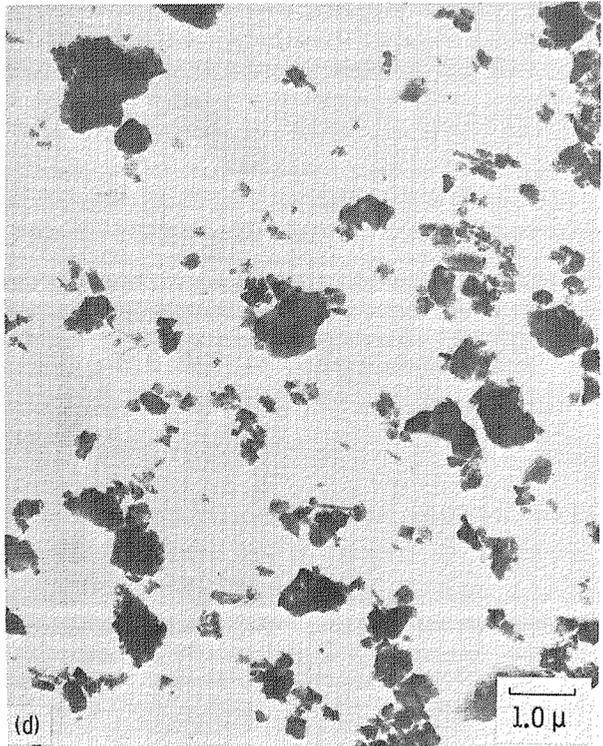
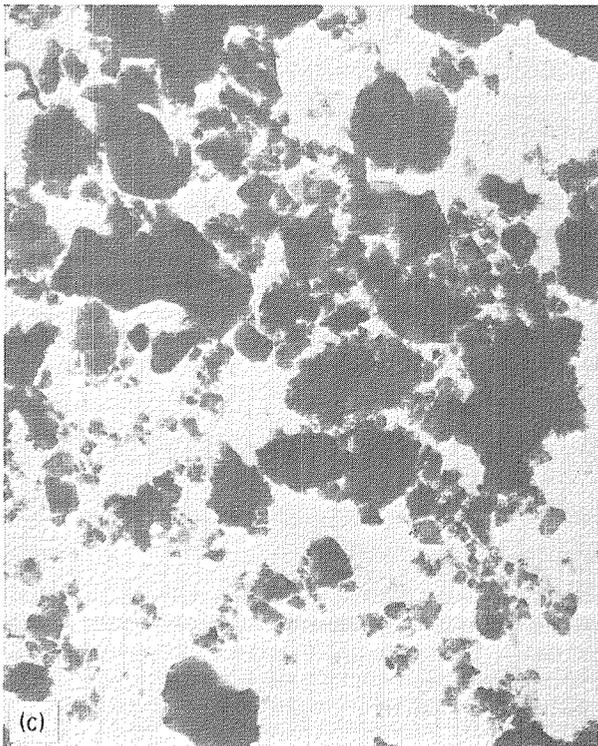
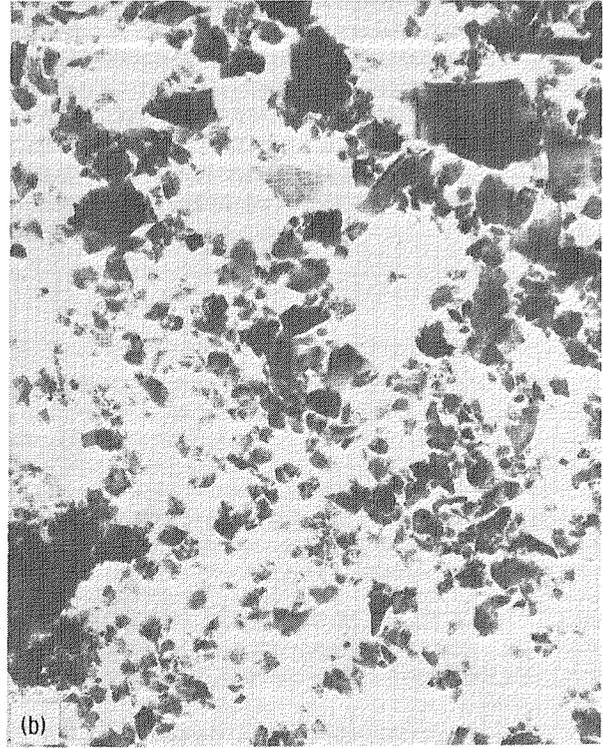
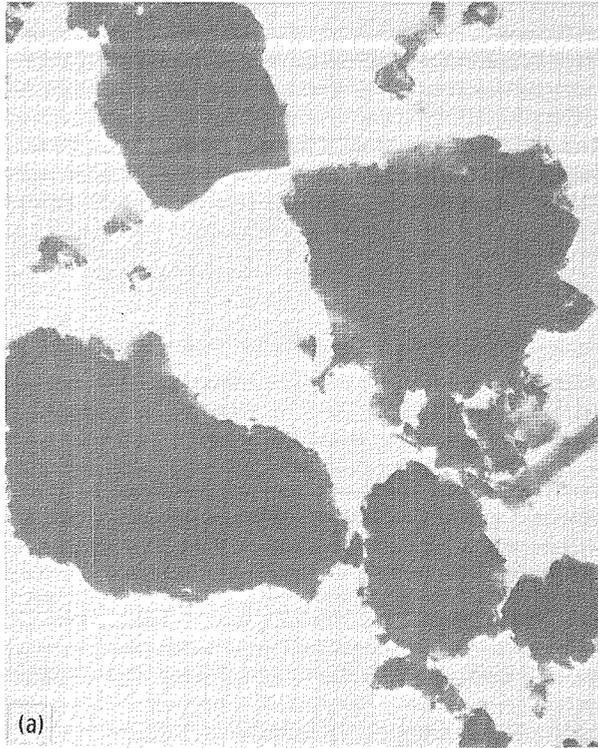


Figure 4. - Transmission electron micrographs of Si₃N₄ powders (a) as received, and after milling to a specific surface area of 17 m²/g in the (b) attrition mill, (c) vibratory mill, and (d) ball mill.

MILLING METHOD	SPECIFIC SURFACE AREA, S , m^2/g	MILLING TIME, hr
◇ AS-RECEIVED	5.2	0
◇ BALL	17	96
○ VIBRATORY	17	12
□ ATTRITION	17	12
△ BALL	31.6	288

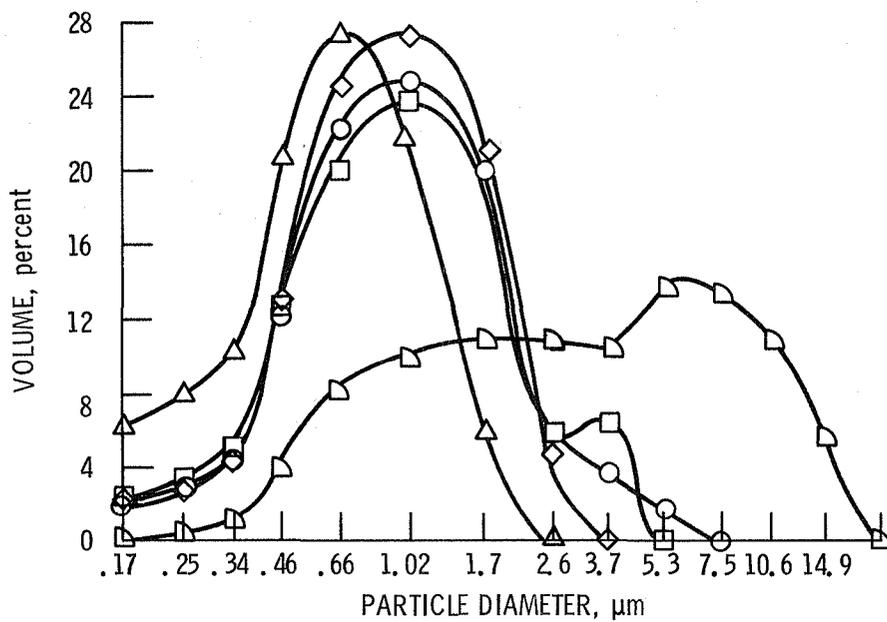


Figure 5. - Differential particle size distributions of Si₃N₄ powders. Milling fluid, ethanol.

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