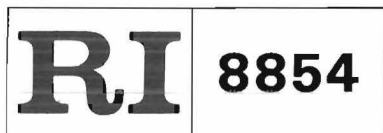


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Bureau of Mines Report of Investigations/1984

# **Use of Bureau of Mines Turbomill To Produce High-Purity Ultrafine Nonoxide Ceramic Powders**

By Dale E. Wittmer



UNITED STATES DEPARTMENT OF THE INTERIOR

**Report of Investigations 8854**

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**UNITED STATES DEPARTMENT OF THE INTERIOR**  
**William P. Clark, Secretary**

**BUREAU OF MINES**  
**Robert C. Horton, Director**

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# UNITS OF MEASURE ABBREVIATIONS USED IN THIS REPORT

$^{\circ}\text{C}$	degree Celsius	$\text{m}^2$	square meter
$\text{cm}^3$	cubic centimeter	$\text{m}^2/\text{g}$	square meter per gram
$\text{cm}^3/\text{h}$	cubic centimeter per hour	min	minute
g	gram	mm	millimeter
$\text{g}/\text{cm}^3$	gram per cubic centimeter	$\mu\text{m}$	micrometer
$\text{g}\cdot\text{h}^{-1}\cdot\text{cm}^{-3}$	gram per hour per cubic centimeter	nm	nanometer
$\text{g}\cdot\text{s}^{-1}\cdot\text{cm}^{-1}$	gram per second per centimeter	pct	percent
		r/min	revolution per minute
h	hour	s	second
in	inch	wt pct	weight percent

# USE OF BUREAU OF MINES TURBOMILL TO PRODUCE HIGH-PURITY ULTRAFINE NONOXIDE CERAMIC POWDERS

By Dale E. Wittmer<sup>1</sup>

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## ABSTRACT

Nonoxide ceramic materials could substitute for high-temperature alloy steels containing imported critical and strategic materials such as cobalt, chromium, and nickel if their high-temperature properties can be improved. An important result of this substitution would be a reduction of the Nation's need for imports of critical and strategic materials. In this Bureau of Mines investigation, the primary objective was to produce high-purity ultrafine (particles  $<5\text{ }\mu\text{m}$  equivalent spherical diameter (ESD)) alpha-silicon carbide ( $\alpha\text{-SiC}$ ) powders with improved high-temperature properties using the Bureau's patented turbomilling process. A secondary objective was to determine the feasibility of using polymer mill construction materials as a means of eliminating metal contamination from the turbomill.

Ultrafine  $\alpha\text{-SiC}$  powders with BET (Brunauer-Emmett-Teller) surface areas from 30 to 35  $\text{m}^2/\text{g}$  ( $\sim 0.06\text{ }\mu\text{m}$  particle ESD) were produced in an all-polymer turbomill. These high-purity powders (with 1-pct B and 1-pct C additions) hot-pressed to greater than 99 pct of their optimal density and exhibited properties commensurate with those of commercially available  $\alpha\text{-SiC}$ .

The most promising of the construction materials tested for the major wear surfaces of the turbomill was ultrahigh-molecular-weight (UHMW) polyethylene, a polymer thermoplastic.

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## INTRODUCTION

The utilization of high-purity ultrafine SiC for high-temperature applications could reduce the Nation's dependence on critical imported chromium, nickel and cobalt presently used in high-temperature alloys. Sinterable ultrafine SiC powders are presently required to meet high-temperature design criteria for conventional and advanced heat engines, heat exchangers, military applications, and many environments where conventional ceramic materials are known to fail. Pressureless sintering of SiC powders offers an alternative to hot-pressing or hot isostatic pressing for obtaining shapes that require little finishing. However, high-purity ultrafine  $\alpha$ -SiC powders that can be pressureless sintered and have the required high-temperature properties have not been produced by conventional grinding techniques.

The use of turbomilling (also referred to as attrition grinding) to produce ultrafine particles is well documented (1-22).<sup>2</sup> This technique, originated and patented by the Bureau of Mines (3), consists of intense milling and agitation of a slurry composed of a relatively coarse milling medium, the material to be

milled, and the suspending liquid. Stan-czyk and Feld, in a previous report (23), presented diagrams of the turbomill and turbomill process, summarized the Bureau's research on turbomilling, and discussed commercial applications of the process.

In past studies, the turbomills used were of all-metal construction, which resulted in metal contamination of the materials being milled. In this study, a different approach, known as autogenous turbomilling, was investigated. In autogenous turbomilling, the mill medium and material to be milled are identical or similar in composition; this eliminates contamination of the fine material produced. In this study, the Bureau investigated the feasibility of using polymer mill construction materials to produce high-purity ultrafine  $\alpha$ -SiC powders without metal contamination from the mill. The report presents the results of wear testing on six polymer construction materials and the preparation of ultrafine  $\alpha$ -SiC powders using UHMW polyethylene as the preferred polymer construction material.

## ACKNOWLEDGMENT

The author expresses his appreciation to Neil N. Ault, technical director of Norton Co., Worcester, MA, for supplying

SiC samples and chemical analyses of the powders produced in this study.

PREPARATION OF ULTRAFINE HIGH-PURITY  $\alpha$ -SiC POWDERS USING AUTOGENOUS TURBOMILL

The turbomill (figs. 1 and 2) consists of a cylindrical container, a cage-like stator composed of vertical bars fixed to rings at top and bottom, a cage-like rotor composed of vertical bars fixed to an upper disk attached to a drive shaft, and a frame that holds the motor and machine components. The turbomill used in this study was a 5-in-diam<sup>3</sup> unit.

## TURBOMILL CONSTRUCTION MATERIAL EVALUATION

Six polymer materials were selected for testing as turbomill construction materials. The objective was to identify material that could be used to eliminate metal contamination of the turbomilled powders by the wear surfaces

<sup>2</sup>Underlined numbers in parentheses refer to items in the list of references at the end of this report.

<sup>3</sup>To convert inches to millimeters, multiply by 25.4.

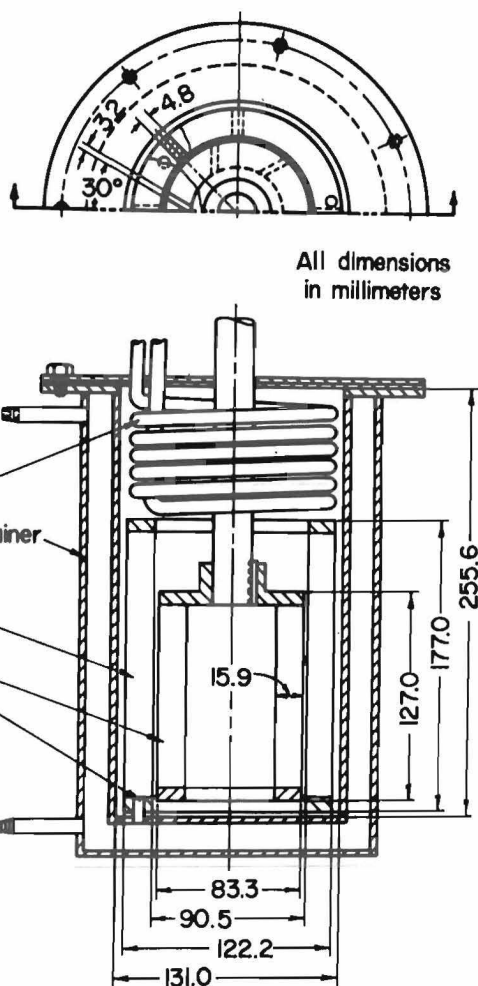


FIGURE 1. - Machine drawing of 5-in-diam (131.0-mm) turbomill.

of the mill. The materials tested were--

UHMW polyethylene--which has the highest impact strength of any thermoplastic, very low moisture absorption, high abrasion resistance, and a very low coefficient of friction.

Teflon<sup>4</sup>--a polytetrafluorethylene with the lowest friction coefficient of any solid, low water absorption, and excellent corrosion and impact resistance.

Torlon--a poly (amide-imide) with high impact strength, excellent

<sup>4</sup>Reference to specific products does not imply endorsement by the Bureau of Mines.

temperature properties and dimensional stability, and good chemical resistance.

Celcon--an extruded acetal thermoplastic with a high melting point and excellent mechanical and chemical properties.

Ryton--a polyphenylene sulfide material with good physical, thermal, and chemical properties.

Polyurethane--a highly flexible polymer with excellent tear, abrasion, oil, and solvent resistance and very high noise and vibration dampening capabilities.

Replaceable rotor blades (approximately 4.875 by 0.625 by 0.250 in) were machined from sheets of each candidate polymer for use in the standard 5-in turbomill. (Other parts of the turbomill were metal.) Prior to the actual testing, a 1-h initial milling was conducted to round off any sharp corners or edges of the polymer materials present after the machining process. The rotor blades were removed after successive milling times of 1, 2, and 4 h and evaluated for weight loss, dimensional changes (volume), and high-wear areas.

The coarse milling medium used for the polymer evaluation tests was minus 28-plus 48-mesh prerounded<sup>5</sup> black  $\alpha$ -SiC (supplied by Norton Co.), the material milled was minus 200-mesh black  $\alpha$ -SiC, and the suspending liquid was distilled water. Other milling parameters were total solids, 70 pct; coarse-to-fine ratio, 2.5:1; grinder speed, 1,600 r/min; water temperature, 28° C; and bottom clearance of rotor, 0.125 in. A typical analysis for Norton's black  $\alpha$ -SiC is given in table 1.

<sup>5</sup>Prerounding consisted of previous milling for 8 h in distilled water with a total solids content of 70 pct and a 2.5:1 ratio of minus 16-plus 30-mesh to minus 200-mesh  $\alpha$ -SiC followed by wet screening to remove the rounded minus 28-plus 48-mesh fraction.

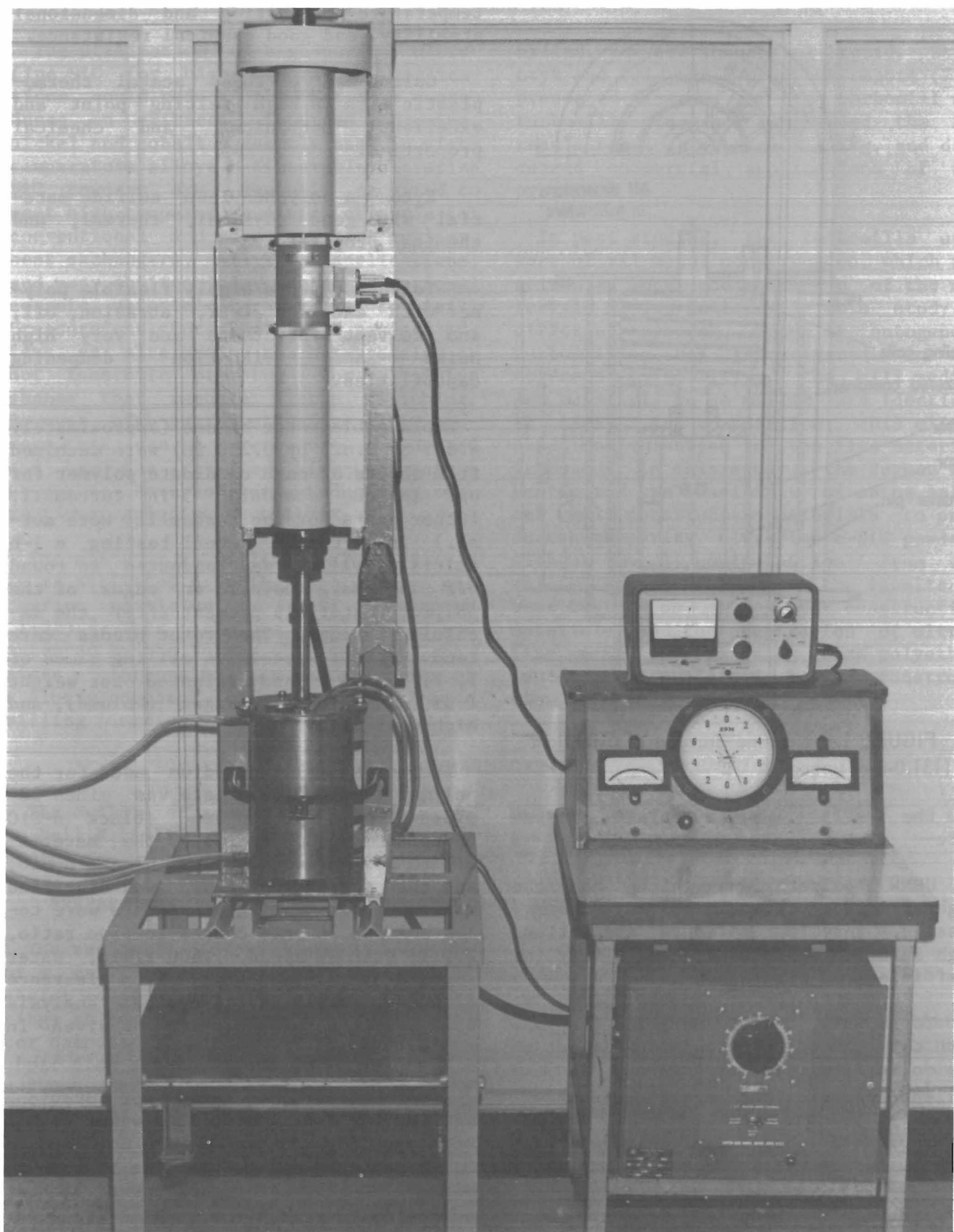


FIGURE 2. - Five-in-diam turbomill assembly.

TABLE 1. - Typical compositions of black and green  $\alpha$ -SiC, percent

Constituent	Black $\alpha$ -SiC	Green $\alpha$ -SiC
SiC.....	97.6	98.6
SiO <sub>2</sub> , free.....	.8	.63
C, free.....	.6	.36
Si, free.....	.5	.15
Fe.....	.2	.08
Al.....	.2	.08
Ca.....	.05	.05
V.....	ND	.04
Mg.....	.05	.03

ND Not determined.

Source: Norton Co.

Figure 3 shows five of the six polymer materials tested after 8 h total milling time. The polyurethane material (not

shown) was eliminated from further testing due to its high flexibility, which caused the blades to deform and contact the stator at the test milling speed. (Coating the stator and rotor units with this abrasion-resistant polymer was ruled out as an alternative due to the very critical dimensional tolerances of these parts in the 5-in-diam mill.) Visual examination indicated that UHMW polyethylene and Celcon showed the least wear and that Ryton showed the most wear after the 8-h test.

The rate of wear, in terms of change in blade volume per unit of time (cubic centimeter per hour), was determined for each polymer, and the results are given in table 2. The amount of polymer wear as a function of milling time (grams per hour per cubic centimeter) was also

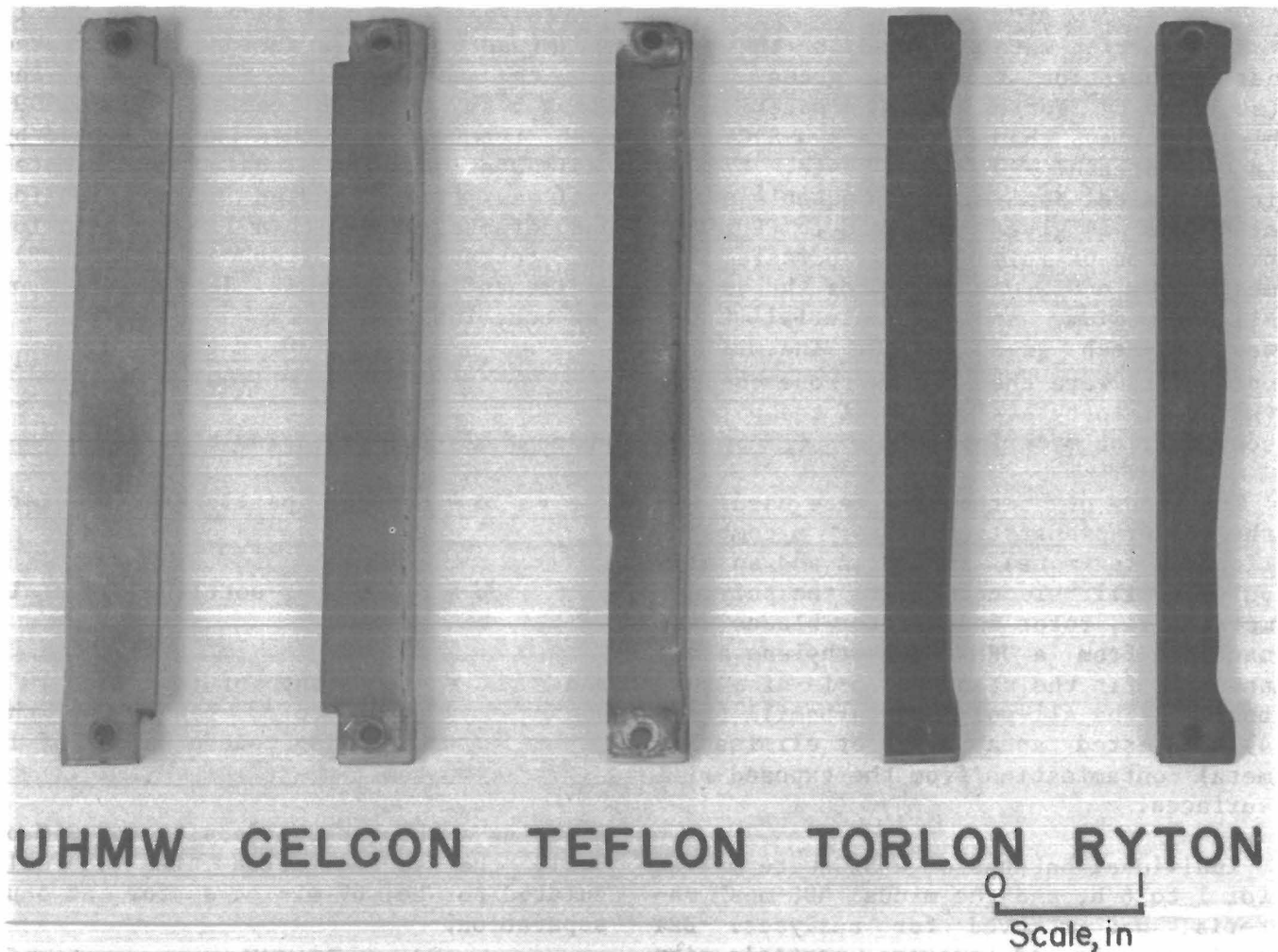


FIGURE 3. - Polymer test specimens after 8-h milling time.

determined, based on a grinder volume of 2,000 cm<sup>3</sup>, and these results are also given in table 2. Based on these results, UHMW polyethylene was selected as the construction material for the all-polymer turbomill.

TABLE 2. - Results of wear testing of polymer rotor blades after 8 h of turbomilling

Polymer	Wear	
	cm <sup>3</sup> /h	g·h <sup>-1</sup> ·cm <sup>-3</sup>
UHMW polyethylene...	0.02	7.028 × 10 <sup>-6</sup>
Celcon.....	.04	2.812 × 10 <sup>-5</sup>
Teflon.....	.05	5.355 × 10 <sup>-5</sup>
Torlon.....	.05	3.580 × 10 <sup>-5</sup>
Ryton.....	.10	8.131 × 10 <sup>-5</sup>

#### POWDER PREPARATION

##### Materials and Conditions

The starting material used in the powder preparation tests was green  $\alpha$ -SiC (supplied by Norton Co.), a material of higher purity than the black  $\alpha$ -SiC used in the material evaluation tests. A typical chemical analysis for Norton's green  $\alpha$ -SiC and is given in table 1. For the preparation of high-purity powders, 30-mesh green  $\alpha$ -SiC was used for the coarse milling medium, and the material milled was 200-mesh green  $\alpha$ -SiC. The milling parameters were the same as those used in the preliminary evaluation of the polymer construction materials.

Two kinds of turbomills were used for the powder preparation tests: a combination polymer-metal turbomill and an all-polymer mill. To construct the polymer-metal mill, rotor and stator blades were machined from a UHMW polyethylene stock sheet to fit the standard 5-in-diam turbomill. The all-polymer turbomill (fig. 4) was tested as a means of eliminating metal contamination from the exposed mill surfaces.

Individual batches of  $\alpha$ -SiC were milled for 1 to 6 h, and the minus 400-mesh material was removed for analyses. BET surface area and average particle size

were measured, and spectrographic chemical analyses were performed. Wear of the UHMW polyethylene rotor and stator blades was also determined after 40 h of total milling time. To eliminate metal contamination by the exposed mill surfaces, an all-polymer turbomill was also constructed (fig. 4).

Turbomilling of  $\alpha$ -SiC in nonaqueous solutions was performed with both decane and silicone oil. Although ultrafine powders were prepared in these solutions, problems encountered were dispersion, recovery of fines, and poor heat transfer. Further evaluation of turbomilling in nonaqueous media was not conducted. All subsequent milling was conducted using distilled water as the milling fluid.

##### Experimental Work

After completion of each milling trial, the minus 400-mesh fraction was separated from the total batch. Separation of the minus 5- $\mu$ m fraction from the minus 400-mesh fraction was then accomplished by sedimentation. The minus 400-mesh material was first adjusted to 5-pct solids and deflocculated with a 1.0-pct solution of tetrasodium pyrophosphate (TSPP). Sedimentation time was determined from the equation

$$t = \frac{C}{D^2 (\rho - 1.00)}, \quad (1)$$

where  $t$  = sedimentation time, s,

$D$  = average particle diameter,  $\mu$ m,

$\rho$  = density of settling material, g/cm<sup>3</sup>,

and  $C$  = a constant of  $2.29 \times 10^{-3}$  g·s<sup>-1</sup>·cm<sup>-1</sup>, based on the settling column length of 14 cm.

Assuming a theoretical density for SiC of 3.21, the sedimentation time was calculated to be 69 min 4 s for a 5- $\mu$ m separation.

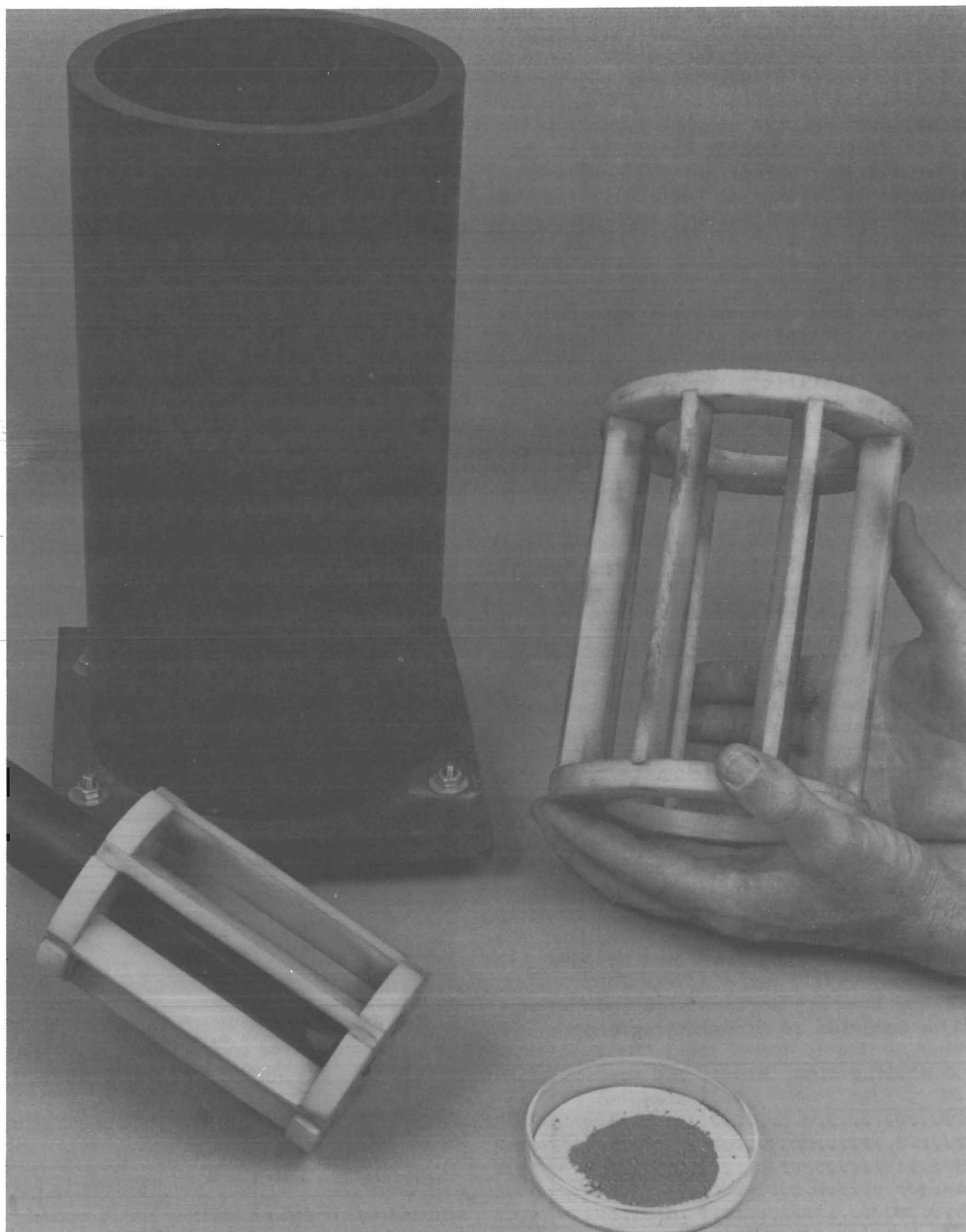


FIGURE 4. - All-polymer rotor and stator after 40-h milling time.

After the minus 5- $\mu\text{m}$   $\alpha\text{-SiC}$  fraction was obtained, the mixture was flocculated using a 3.0-pct solution of HCl. After settling for 25 h, the supernatant liquid was removed with a syphon. At this stage, the solids content was approximately 15 pct. Further consolidation (to approximately 30 pct solids) was accomplished by drying at 50° C. Prior to powder characterization, the materials were dried at 100° C for 24 h and then sized to minus 400 mesh.

Surface area measurements were made by nitrogen adsorption using a BET surface area analyzer. The ESD was determined from the specific surface area and density of SiC (3.217 g/cm<sup>3</sup>) by the equation

$$d = \frac{6}{S \rho}, \quad (2)$$

where  $d$  = ESD,  $\mu\text{m}$ ,

$S$  = specific surface area, m<sup>2</sup>/g,

and  $\rho$  = material density, g/cm<sup>3</sup>.

Scanning electron microscopy (SEM) was used to compare the observed particle size to the calculated ESD and to determine the state of agglomeration. Chemical analyses were conducted by the Norton Co. using spectrographic analysis.

### Results and discussion

The results of the specific surface area measurements and the associated ESD's are given in table 3 as a function of milling time. The data show that the surface area increased up to the 4-h

TABLE 3. - BET surface area and ESD as a function of turbomilling time

Milling time, h	BET surface area, m <sup>2</sup> /g	ESD, nm
1.....	22.8	81.8
2.....	30.0	62.2
3.....	33.4	55.8
4.....	33.8	55.2
5.....	31.1	60.0

milling time and then decreased with increased milling time. This was due to an increase in comminution of the coarse milling medium. This effect is also shown in figure 5, which shows both the BET surface area and the coarse fraction (plus 100 mesh) lost to fines as a function of milling time.

SEM analyses of the dried powders confirmed the ESD data and revealed the presence of large agglomerates composed of the particles smaller than 1  $\mu\text{m}$ . The average agglomerate size was found to be approximately 2  $\mu\text{m}$ , using a Coulter Counter (Norton Co.), regardless of the BET surface area. This agglomeration may present a problem in future sintering studies due to the affects of agglomeration on packing, sintering, and densification. The SEM photomicrographs in figure 6 show the initial 200-mesh  $\alpha\text{-SiC}$  compared to the ultrafine agglomerated powder produced by turbomilling for 6 h.

Contamination of the  $\alpha\text{-SiC}$  powders by the UHMW polyethylene was not detectable by infrared spectroscopy. Because the density of the polyethylene was lower than that of water, any polymer contamination would have been removed during sedimentation (along with the supernatant liquid). The wear rate for

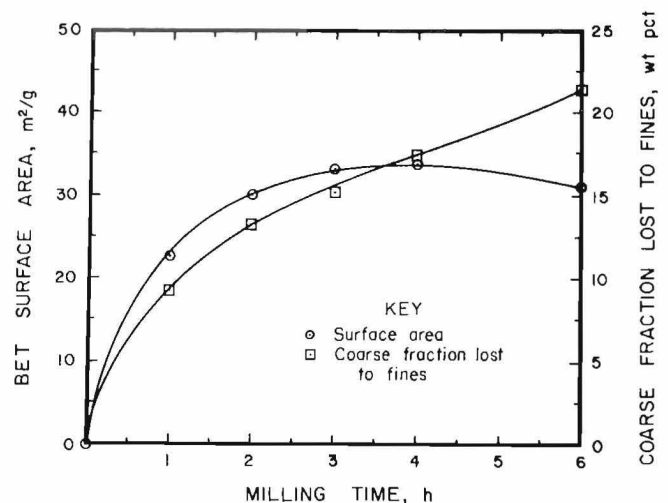


FIGURE 5. - BET surface area and coarse fraction lost to fines as a function of milling time.

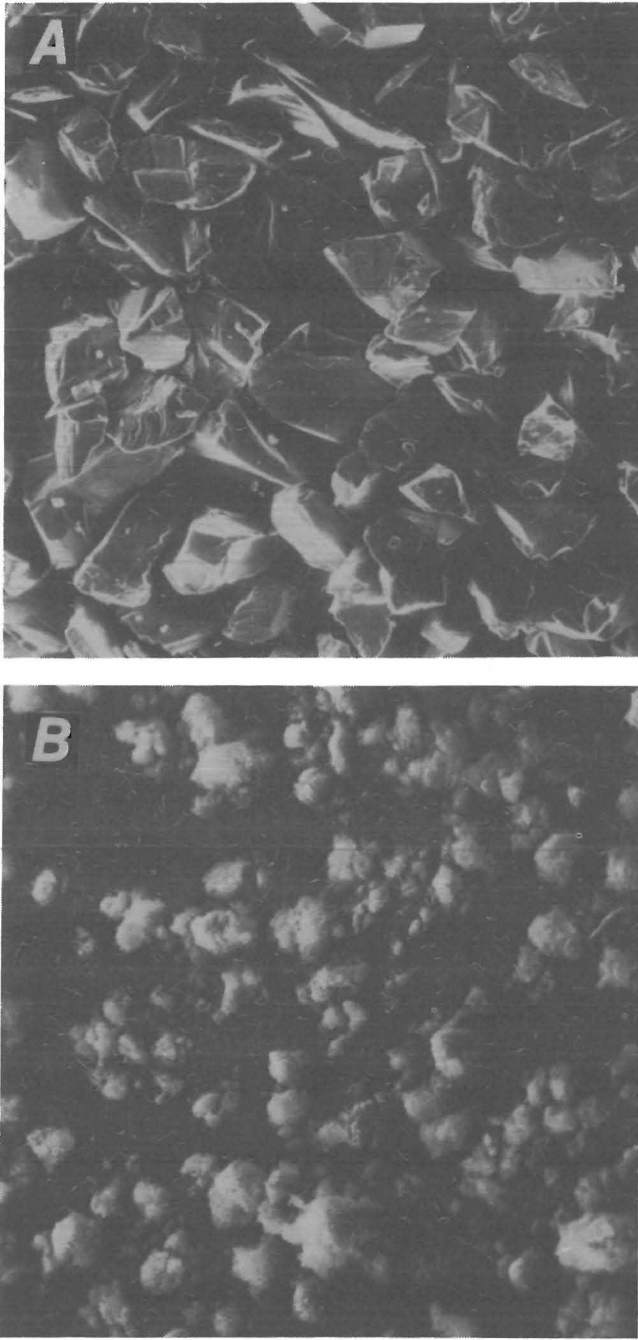


FIGURE 6. - *A*, As-received 200-mesh  $\alpha$ -SiC grain (X 85); *B*, ultrafine powder prepared by turbomilling for 6 h (X 12,000).

polyethylene rotor blades was  $0.03 \text{ cm}^3/\text{h}$  after 40 h of milling and  $0.02 \text{ cm}^3/\text{h}$  after 8 h of milling.

Turbomilling with the combination polymer-metal turbomill, with metal rotor and stator parts exposed to the milling

medium, was found by spectrographic analyses to increase iron content from 0.08 pct in the starting material to approximately 1 pct after milling for 1 to 6 h (table 4). After milling for 40 h, abrasion of the metal surfaces was quite severe, as shown in figure 7. It is not known whether the iron was present as a bulk impurity and/or as a particle surface contaminant. Other elements identified spectrographically, which are likely associated with the metal wear surfaces, were Zr, Mo, and Ni.

TABLE 4. - Spectrographic analyses of turbomilled  $\alpha$ -SiC powders, weight percent

Element	Mill type	
	Polymer-metal	All polymer
Fe.....	~1.0	0.08
Ni.....	.08	.02
Al.....	.04	.03
Mo.....	Trace	ND
Zr.....	<.01	ND
Ca.....	.01	<.01
Ti.....	.01	<.01

ND Not detected.

Source: Norton Co.

Iron contamination was reduced from approximately 1 pct to less than 0.4 pct by leaching the powders in 5-pct HCl at  $50^\circ \text{C}$  for 2 h, followed by rinsing and filtering through five cycles with distilled water. BET surface areas of the acid-leached powder were approximately  $25 \text{ m}^2/\text{g}$ .

Ultrafine powders produced in the all-polymer turbomill had iron contents equivalent to the starting material and BET surface areas of 30 to  $35 \text{ m}^2/\text{g}$ . With additions of 1-pct B and 1-pct C, these powders were hot-pressed at the Naval Research Laboratories, Washington, DC. Densities greater than 99 pct of the theoretical density and properties similar to those of the best commercially available powders were obtained during preliminary sintering evaluations. After 40-h total milling time, the all-polymer mill parts showed negligible wear.

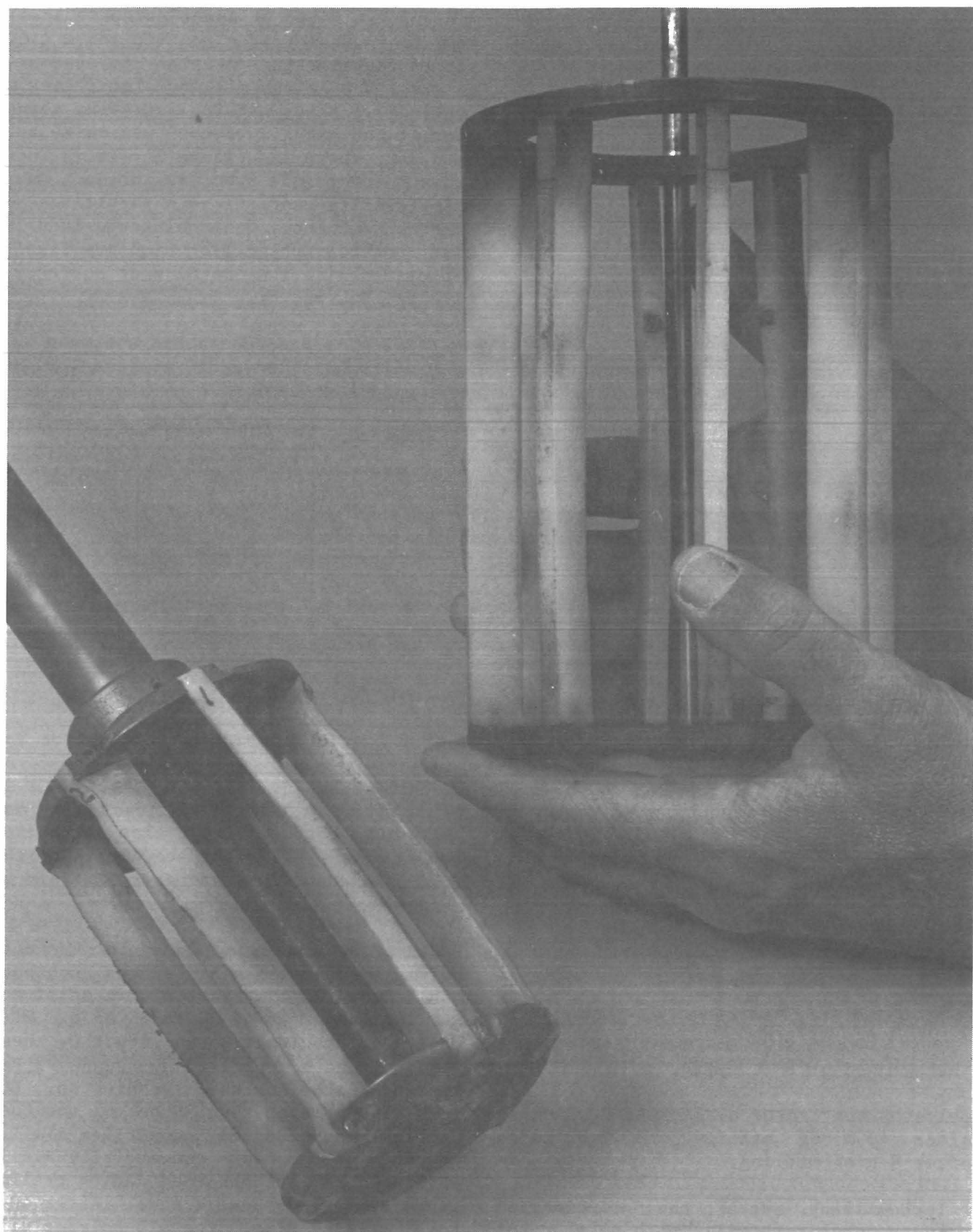


FIGURE 7. - Combination polymer-metal rotor and stator after 40-h milling time.

## CONCLUSIONS

1. Ultrafine  $\alpha$ -SiC powders were prepared by autogenous turbomilling. Powders with BET surface areas of 30 to 35 m<sup>2</sup>/g were obtained after 3 to 6 h of milling.

2. Iron contamination in the ultrafine  $\alpha$ -SiC powders was eliminated through the use of all-polymer turbomill construction materials.

3. UHMW polyethylene was selected as the best polymer construction

material for the high-wear surfaces of the turbomill.

4. Preliminary sintering results indicated that turbomill-produced ultrafine  $\alpha$ -SiC can be hot-pressed to greater than 99 pct of its theoretical density and exhibit properties similar to commercially available  $\alpha$ -SiC powders.

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