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**OAK RIDGE
NATIONAL
LABORATORY**

LOCKHEED MARTIN

**Development of Improved Processing and
Evaluation Methods for High Reliability
Structural Ceramics for Advanced Heat
Engine Applications Phase II**

PHASE II FINAL REPORT

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CERAMIC TECHNOLOGY PROJECT

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LOCKHEED MARTIN ENERGY RESEARCH CORPORATION
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DEVELOPMENT OF IMPROVED PROCESSING AND EVALUATION METHODS FOR HIGH
RELIABILITY STRUCTURAL CERAMICS FOR ADVANCED HEAT ENGINE
APPLICATIONS, PHASE II

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ABSTRACT

The research program had as goals the development and demonstration of significant improvements in processing methods, process controls, and nondestructive evaluation (NDE) which can be commercially implemented to produce high reliability silicon nitride components for advanced heat engine applications at temperatures to 1370°C.

In Phase I of the program a process was developed that resulted in a silicon nitride - 4 w% yttria HIP'ed material (NCX 5102) that displayed unprecedented strength and reliability. An average tensile strength of 1 GPa and a strength distribution following a 3-parameter Weibull distribution were demonstrated by testing several hundred buttonhead tensile specimens.

The Phase II program focused on the development of methodology for colloidal consolidation producing green microstructure which minimizes downstream process problems such as drying, shrinkage, cracking, and part distortion during densification. Furthermore, the program focused on the extension of the process to gas pressure sinterable (GPS) compositions.

Excellent results were obtained for the HIP composition processed for minimal density gradients, both with respect to room-temperature strength and high-temperature creep resistance. Complex component fabricability of this material was demonstrated by producing engine-vane prototypes. Strength data for the GPS material (NCX-5400) suggest that it ranks very high relative to other silicon nitride materials in terms of tensile/flexure strength ratio, a measure of volume quality. This high quality was derived from the closed-loop colloidal process employed in the program.

INTRODUCTION

Highlights of the silicon nitride processing methodology that was developed in Phase I of the program are discussed in a previous report.¹ In Phase I, work focused on the attainment of high strength and reliability through the identification and subsequent control of strength degrading flaw populations. The material produced by the resultant process has been designated NCX-5102 and consists of silicon nitride - 4% yttria composition which is densified by use of glass encapsulating HIP'ing.

In the report¹, an account is provided of developmental efforts which resulted in a colloidal processing methodology for the production of high strength, high reliability silicon nitride components for advanced heat engine applications at temperatures to 1370°C. The process includes an innovative closed-loop system of uninterrupted colloidal processing, from milling to shape forming, which effectively circumvents traditional sources of contamination. The effort concentrated on the near-net-shape fabrication of large-aspect-ratio, axisymmetric buttonhead tensile bars. This geometry served as a challenging forming requirement, sufficient for development of a general shape-forming capability and it also provided a direct means to assess tensile strength levels attributable to the process.

The process demonstration provided experience and data which suggest that NCX-5102 truly ranks as a world-class, structural silicon nitride material. The test program established a mean tensile strength of 997 MPa for a set of 320 tensile rods. Competing risk data analysis suggests that the strength distribution associated with process-related, intrinsic strength-impairing flaws is best represented by a 3-parameter Weibull model with a threshold strength equal to 665 MPa. Approximately one half of the data fall into this category. The other half primarily involved machining-damage-related failure origins and define a distinctly different strength distribution.

In Phase II of this advanced processing program, efforts were focused on refinement of the forming aspects of this reliable process and extension of the process to non-HIP sinterable compositions.

The Phase II plan was structured with three tasks:

- Task 1 - Reliability optimization through density gradient reduction in aqueous colloidal forming and glass encapsulating HIP'ing.
- Task 2 - Application of the high reliability colloidal processing technique to a gas pressure sinterable (GPS) composition.
- Task 3 - Demonstration of representative complex component fabrication in the HIP system.

Key activities within Task 1 involved development of an understanding of the density gradient by examining the slurry properties and developing a mathematical model to simulate the casting process. Technical challenges with respect to Task 2 involved control of hydrolysis of the specific sintering aid in an aqueous medium and subsequent casting of this suspension. Accomplishments are discussed below in sections related to the three tasks.

FORMING USING HIGHLY LOADED SUSPENSIONS AND HOT ISOSTATIC PRESSING

The overall effort of this task was to help develop a robust net-shape-forming technology for complex-shape heat engine and gas turbine components. As identified earlier in Phase I¹ of the Processing for Reliability Program, density gradients in the cast components resulted in overall lower process yield due to non-uniform shrinkage during drying (cracking) and HIP densification (part distortion). This problem was substantially addressed in the Phase I program through understanding and manipulation of the casting-rate² behavior of the slip. In the current (Phase II) program, the effort was directed towards a more comprehensive and complete analysis of all factors that influence the casting behavior of an aqueous silicon nitride suspension. However, greater emphasis was placed on the development of a highly loaded suspension approaching solid content ~ 80W% (55V%). Use of a highly loaded suspension during shape forming is anticipated to alleviate spatial density gradients by a) minimizing particle segregation, b) controlling casting rate, and c) potentially totally eliminating drainage of fluid during green forming. Various factors that influence the solids content, namely, type of surfactant, particle size and surface area of the powder, powder surface chemistry, comminution procedure, etc. were examined throughout this investigation. The task was divided into the following four sub tasks: i) Powder Processing, ii) Density Gradient Control, iii) Casting Process Simulation and Modeling and iv) Effect of Mold Material. Progress towards each of these sub tasks is described below:

CLOSED LOOP POWDER PROCESSING

In order to obtain high solids loading, the sedimentation behavior of Si_3N_4 powder with five different surfactants (A, B, C, D, E) was measured with time. As seen in Table 1, the final sedimentation density was increased from 1.25 g/cm³ in the case of no surfactant to a value of approximately 1.50 - 1.55 g/cm³ with 3 of the surfactants. The optimum surfactants were utilized in the closed-loop processing methodology development during Phase I of this program. In addition, particle size distribution and the surface area of the powder were manipulated extensively to analyze their effects on solids loading achievable. The detailed results of this effort are described below under density gradient control.

Table 1: Surfactant effects on sedimentation density

NO SURFACTANT		SURFACTANT A		SURFACTANT B	
wt %	Density g/cc	wt %	Density g/cc	wt %	Density g/cc
o	1.250	0.1	1.496	0.2	1.522
		0.4	1.450	0.4	1.546
		0.6	1.523	0.6	1.367
		0.8	1.390	0.8	1.572
		1.0	1.320	1.0	1.550

SURFACTANT C		SURFACTANT D		SURFACTANT E	
wt %	Density g/cc	wt %	Density g/cc	wt %	Density g/cc
0.2	0.549	0.5	1.492	0.05	1.477
0.4	0.524	1.0	1.454	0.1	1.488
0.6	0.550			0.3	1.538
0.8	0.548			0.5	1.421
1.0	0.667				

POWDER SURFACE MODIFICATIONS

The activities in this area were pursued at Princeton University under the supervision of Dr. Ilhan Aksay. The work was primarily focused towards surface modification of the silicon nitride powder in order to achieve highly loaded aqueous suspensions. Specifically the surface of the silicon nitride powder was coated with Al_2O_3 or SiO_2 using colloidal based approaches. Alumina and silica, in general, exhibit greater compatibility and improved in aqueous medium as compared to silicon nitride.

In the subsequent effort, a gelling substance was incorporated in the surface-modified silicon nitride powder suspensions in order to develop a robust, aqueous-based forming process similar to injection molding. The details of this research effort are documented in Appendix 1.

HIGH ENERGY ATTRITION MILLING

The activity was pursued at the National Institute of Standards and Technology (NIST) under the direction of Dr. Subhas Malghan. Primary efforts were directed towards the development of data for the selection of type and concentration of a polyelectrolyte dispersant for high density suspensions preparation through HEAM. In addition, surface chemical characterization of constituent materials (silicon nitride, yttria, and polyelectrolyte) were also completed. Five different methods were used to evaluate two potential surfactants (S_1 , S_2) as described below:

- Interface chemistry of powder in aqueous environment
- Effect of polyelectrolyte concentration
- Particle-size distribution
- Density of slip cast green body
- Adsorption isotherm

These data showed that both dispersants (S_1 , S_2) provide the same amount of dispersion at pH 9.0, while the concentrations to achieve the maximum dispersion were different due to difference in their molecular weights. Based on these evaluations, we have selected 300 and 640 ppm (with respect to powder weight) as the most suitable concentration for the two surfactants to achieve the highest green density. The green densities of SNE-03 powder without milling were 61-63% theoretical.

All three powder components have been fully characterized and representative samples for milling have been prepared. The

overall purpose of these milling tests was to achieve as high a solids loading as possible. Our final target was 80-85% solids by weight. Therefore, initial tests on 75% solids as the base level and 80% as the high level were planned, with the specific surface area of the milled powder at 8 and 12 m²/g. Further details of this effort are included in Appendix 2.

DENSITY GRADIENT CONTROL

A series of experiments was conducted to determine the effect of slip, mold, and casting characteristics on the green compact. The objective of the set of experiments was to evaluate and minimize density gradients in the cast components, and to examine how the control variables affect the performance variables. The control variables evaluated were the following:

Slip Variables

Surface area

Weight % solids

Mold Variable

Mold material

Casting Variables

Pressure profiles

Orientation of filtration surface (up or down).

The performance variables examined in these experiments were:

Density of the green compact

Cracking during drying

Density gradient after HIP (Total Indicated Runout).

As indicated by Figures 1 to 4, green densities and density gradients have been correlated with surface area and weight-percent solids of the slip. The casting rate of the slip has also been shown to have an effect on green densities and density gradients. The density gradient has been measured indirectly by observing the amount of bending (runout-TIR) in cast tensile rods after HIP. The higher the runout, the larger the density gradient. The orientation of the filtration surface was a significant variable only at low-weight-percent solids since with low-weight-percent solids there is enhanced segregation of the slip. This results in an increased density gradient if the component is cast with the filtration surface on top (segregation and casting rate act in concert to increase the density gradient), and reduced if the filtration surface is toward the bottom (density gradients due to segregation and casting rate are counteracting).

Process yield is reduced both by cracking during drying and by excessive bending during HIP'ing. These reject modes were found to depend upon green density in opposing fashions, with drying cracks more prevalent and bending less severe at higher green densities.

Figure 1: Green Density vs. Powder Surface Area

Figure 2: Green Density vs. Slurry Weight % Solids

Figure 3: Tensile Bar Runout vs. Powder Surface Area

Figure 4: Tensile Bar Runout vs. Slurry Weight % Solids

CASTING PROCESS SIMULATION AND MODELING

The modeling of the colloidal consolidation process (pressure casting) was extended beyond the historical models of Aksay and Schilling³ and Adcock and MacDowall⁴ to include a spatial variation in density.

The model accounts for green body density gradients through the use of a porosity constitutive relationship. Experimental data suggest that the porosity that develops at the casting front, Figure 5, can be represented as a function of casting rate as follows:

$$\varepsilon = \varepsilon^* + \Delta \varepsilon \tanh \left(\frac{d\xi}{dt} K \right) \quad (1)$$

The values ε^* and $\varepsilon^* + \Delta \varepsilon$ represent the low and high rate porosity levels, respectively. The governing equation for the casting process is given by

$$\frac{d\xi}{dt} = \frac{\chi}{1-\chi-\varepsilon} \frac{1}{\eta} \frac{P_{applied}}{\int_0^\varepsilon \alpha dx} \quad (2)$$

Where

- χ = solids volume fraction of slurry
- η = filtrate viscosity
- α = cake specific resistance

Equation (2) was solved numerically subject to the porosity constitutive equation (1) for representative conditions. Solutions provide cake thickness and density (porosity) distribution as a function of time.

Figure 5: Geometry of Cake Growth

Results for density variations are shown in Figure 6 for the two cases of 60.6 and 68.9 w/o solids with $10 \text{ m}^2/\text{gm}$ surface area and 25 psi pressure. Notice that a low density region is predicted near the mold. This results from the very high growth rate that the cake initially experiences. With the parameters chosen here the low density region extends about 0.1 to 0.2 mm into the cake. The lowest density is about 5% less than the bulk value. These values are in general agreement with what has been observed in casting experiments. Figure 7 provides results which demonstrate that the density gradient at the mold can be controlled through manipulation of the pressure profile.

LOW DENSITY REGION PREDICTION

Figure 6: Prediction of Low Density Region Near Mold Interface

Figure 7: Density Variation for Constant and Ramped Pressure Profiles

EFFECT OF THE MOLD MATERIAL

Casting of aqueous silicon nitride slurries using plaster of Paris molds has typically resulted in a 0.1 - 1.0 mm high density skin, as well as a 1-3 mm low density cast layer at the surface of the component. It is believed that the low density layer is caused by floccing of the slurry in contact with the mold, and that the high density skin is caused by diffusion of mold material into the cast body. In order to eliminate these undesirable effects, a ceramic mold material was investigated. In preliminary work (Phase I) density gradients were reduced by up to 75% using this material. To examine the properties of this mold in relation to a standard plaster mold, pucks were cast using a constant pressure of 30 psi. The plot of thickness vs. time from the Kozeny-Carman model should be of the form:

$$L = k * t^m \quad (3)$$

where L is the thickness of the cast, t is the time, $m = .5$, and k is a constant which depends on parameters of the slurry and the applied pressure. Fitting the observed thickness vs. time data from the two pucks to the model (Figure 8), the estimated values for m were 0.485 for the plaster mold and 0.502 for the alternative mold material. This indicates that for the plaster mold we have a higher than expected initial casting rate, or a lower than expected final casting rate.

Figure 8: Effect of Mold Material on Casting Rate Profile

ESTABLISHING NDE LIMITS FOR DENSITY GRADIENT IDENTIFICATION

A Si_3N_4 step wedge was used as a tool to determine the detection limits of Microfocus X-ray on density gradients. The material used for the step wedge was dense (HIP'ed) NCX 5102. The wedge was machined with 10 steps that varied in thickness from 3.60 mm to 3.81 mm. These changes in thickness simulated density differences of 0.2, 0.3, 0.4, 0.5, 0.8 and 1%. The operating parameters of the X-ray unit were optimized in order to get the best contrast and resolution. Using film radiography, a 0.5% density difference was found to be the detection limit for Microfocus X-ray.

TENSILE BAR FABRICATION AND TESTING

As a demonstration of the mechanical properties associated with the above process, buttonhead tensile specimens were tested for RT fast fracture strength and flexure specimens were tested for 1370°C fast fracture strength and stress rupture life. A post-HIP crystallization heat treatment was used in the production of the specimens to accentuate the HT mechanical behavior. In Phase I it was established that the crystallization step enhances HT strength as well as creep resistance, although the RT strength suffers somewhat due to this process step. The results of the current tests confirmed past experience. The stress rupture tests were run at 300 MPa and 350 MPa. Exceptional creep resistance was found with 11 of 12 specimens surviving 200 hours at 1370°C. One specimen loaded to 350 MPa failed at 46 hours.

The NCX-5102 process developed in Phase I was found to yield material with 1 GPa level tensile strength (997 MPa mean for $n=320$). This is in contrast to 700 MPa level tensile strength of commercially available silicon nitride materials. A mean of 842 MPa was found for the tensile strength of 18 buttonhead specimens

produced in the current experiments. The corresponding 2-parameter Weibull values are 895 MPa for characteristic strength and 10.1 for Weibull modulus. Compared to commercially available material, the 842 MPa mean strength is excellent. Compared to NCX-5102, 842 MPa represents a 16% mean strength reduction. This disparity can be viewed to be primarily a result of the crystallization step, a step that was not used in the NCX-5102 process.

HIGH RELIABILITY COLLOIDAL PROCESSING OF GPS COMPOSITION

AQUEOUS PROCESSING AND HYDROLYSIS CONTROL OF MgO

Three methods were identified to minimize the problems associated with the aqueous processing of magnesia-containing slurries: temperature control, pH control, and surfactant addition. Since previous results had demonstrated the importance of maintaining temperature control, a set of experiments was designed to assess the importance of pH and surfactant addition while slurry temperature was maintained at a predetermined level. A baseline silicon nitride plus yttria slurry was prepared and then cast after making additions of the magnesium sintering aids. As seen in Table 2, the baseline slurry attains a high green density upon casting, regardless of pH. Addition of magnesium sintering aid Mg(a) with surfactant, however, resulted in slurries which were too thick for casting. The addition of Mg(c) sintering aid resulted in a decrease in green density for both pH levels. The combination of Mg(c) plus surfactant at pH B yielded a cast body within 2% of the density of the baseline slurry. Thus, a combination of pH control and surfactant addition significantly reduces the problems associated with the processing of magnesium containing slurries.

Table 2: Green Densities Using Test Slurries

Sample	Green Density (g/cc)	
	pH = A	pH = B
Baseline ($\text{Si}_3\text{N}_4 + \text{Y}_2\text{O}_3$)	1.95	2.08
Baseline + Mg(a) + Surfactant	uncastable	uncastable
Baseline + Mg(c)	1.61	1.99
Baseline + Mg(c) + Surfactant	1.75	2.05

The results of these bench-top experiments were scaled up to a 35-kg-batch of slurry. As seen in Figure 9, the baseline silicon nitride plus yttria slurry attains a high green density. Addition of magnesium-containing sintering aids resulted in a decrease in green density relative to the baseline when no aging was allowed. There was an indication, however, that a kinetic phenomenon was occurring and thus the slurries were allowed to age under continuous agitation. After four hours of aging, the green densities of the magnesium-containing slurries recovered to within 10% of the baseline value. Further aging of the slurry had no additional beneficial effect. Thus a scaled-up process has been developed whereby magnesium-containing slurries can be cast to obtain bodies with acceptable green densities.

Figure 9: Cast Density vs. Aging Time of Test Slurries

GAS PRESSURE SINTERING (GPS) OPTIMIZATION

Sintering runs were used to examine the effect of the sintering bed and degradation of mechanical properties due to the reaction layer. Reduction in strength at the surface relative to the bulk was found to range from a low of 2% to a high of 30%, Table 3.

The mill batches also showed variation in material properties with the differences in properties attributed to processing conditions. In order to understand the batch-to-batch variability, an evaluation was completed which sought possible correlation of strength with ten separate chemical and physical characteristics (variables) of 5 batches of powder (3 IPA-milled, 2 water-milled). Of the ten, only one (variable A) showed a significant correlation with strength, Figure 10. The process was modified to consistently attain the lower levels of variable A.

Table 3: Flexure Strength of Surface and Bulk Samples

Bed	Location	Batch S004	Batch S008	Batch S009	Location Average	Bed Average
1	Surface	709	691	750	717	768
	Bulk	893	800	766	819	
2	Surface	798	663	539	667	733
	Bulk	846	778	772	799	
Batch Average		812	733	707		750

Figure 10: MOR Strength vs. Variable A Level

PROCESS SCALE UP AND VERIFICATION

The final batch (S012) of NCX 5400 was prepared according to the optimized process developed in the course of the program. Five techniques were used to facilitate the processing of S012:

- 1) Temperature Control
- 2) pH Control
- 3) Surfactant Additions
- 4) Form of MgO Addition
- 5) Si_3N_4 Powder Size Distribution

Approaches 1-4 address the solubility and, hence, dissolution of the magnesium sintering aid into Mg divalent cations. Controlling temperature and pH during milling seems to lower the magnesium ion concentration. The use of surfactants also inhibits the dissolution reactions of the sintering aid into the divalent cations. In addition, some chemical forms have an inherently lower solubility than MgO. Finally, controlling the size distribution of the Si_3N_4 powder leads to better powder packing.

In the early stages of the program, cast green densities of 1.2 - 1.4 g/cc and solids loadings of <60 weight percent were obtained. Using the 5 approaches outlined above, however, green densities above 2.0 g/cc were attained. Furthermore, the slurry could be concentrated to >70 w% and densified to >99% of theoretical density. A chronology of the progress is given in Table 4.

Table 4: Data Showing Batch Improvements in Casting Performance

Batch	Filtered	Concentrated Weight % Solids	Green Density (g/cc)	Fired Density % TD
S001	-	51	1.3	>99
S008	very fine	41	1.3	98.5
S010	medium	40	2.2	96
S012	very fine	72	2.0	>99

Batch S012 simultaneously achieved colloidal stability (as evidenced by the filtration step and concentration to 72 weight percent) and high green and fired density.

Cast tensile rods and CIP'ed tiles from S012 were air fired and then densified according to the GPS cycle. Buttonhead tensile specimens were machined following the machining procedure developed in the Processing Phase I program. Test data are summarized in Table 5.

Table 5: NCX 5400 Strength and Toughness Data

	Tensile Strength (MPa)	Flexure Strength (MPa)	K _{IC} MPa m ^{1/2}
Mean	625	773	8.28
S.D.	103	46	0.38
Characteristic Strength	667	775	
Weibull Modulus	7.0	20	

The tensile strength data were obtained from 27 buttonhead tensile specimens. Of the 27 rods tested, 13 failed from flaws located in the volume of the sample and 14 failed from surface flaws.

In Phase I of the program, it was demonstrated that the intrinsic flaw population of NCX 5102 was best described by a 3 parameter Weibull distribution. Since only a sampling of flaw origins for NCX 5400 was done, this grouping of the flaws was not performed in this case. The entire flaw population - both volume and surface - was therefore analyzed according to both 2- and 3-parameter Weibull statistics. As seen in Figure 11, both 2- and 3- parameter distributions describe the data reasonably well ($r^2 = 0.970$ and $r^2 = 0.979$, respectively).

The strength performance of NCX 5400 as compared to other silicon nitride materials is displayed in Figure 12 in which the tensile strength is plotted versus the flexure strength. Materials with superior volume properties lie in the upper half of the graph; materials with superior surface properties lie in the lower half. NCX 5400 is seen to be equivalent in tension to other gas-pressure sintered materials such as SN253 and NGK. Since the objective of this program was to demonstrate reliable volume properties, this can be assessed by observing how near to the upper half of the graph a material falls. NCX 5400 is seen to be the 3rd best material in this regard following NCX 5102 and SN253.

Figure 11: Weibull Plots of NCX 5400 Tensile Strength Data

Figure 12: Tensile vs. Flexure Strength of NCX5400 and Other Silicon Nitride Material

COMPLEX SHAPE DEMONSTRATION

Consistent with the objective of this task, complex-shape-forming capability of the NCX 5102 slurry was evaluated. Using the standard operating procedure (SOP) of the closed loop process, a 15 kg batch of NCX 5102 slurry was milled, filtered, and subsequently concentrated to a solids loading 72 w%. Shape-forming capability of this slurry was evaluated by pressure casting a complex shape vane (Figure 13) designed for an APU. Castings were performed using a standard mold being utilized currently by NAC (Norton Advanced Ceramics) for prototype production of these components. In excess of 30 components were pressure-cast and HIP'ed. The overall process yield for acceptable components was established to be greater than 85%.

Net-shape castings were produced crack free with NCX 5102 slurry with and without binder addition. The quality of the NCX 5102 castings was found to be equivalent to that produced by the prototype process involving conventional powder processing and agglomerated powder.

Vanes were examined for presence of cracks and surface pits both in the green and dense (HIP'ed) states. Frequency of cracks was found to be equivalent compared to the conventional process. However, presence of surface pits in the HIP'ed component were minimized due to the use of non-agglomerated powder in the closed-loop NCX 5102 process.

The mechanical properties of the densified component could not be established using standard test specimens, due to the small size of the vane. Tile produced from two closed-loop-processed slurry batches (representative of the cast components) provided MOR bars for fast fracture strength and fracture toughness testing. Mean strength and toughness values resulted as 1009 MPa, 1011 MPa and $7.12 \text{ MPa}\sqrt{\text{m}}$, $6.87 \text{ MPa}\sqrt{\text{m}}$, respectively. These data are consistent with the superior intrinsic tensile strength of NCX 5102 established in Phase I of this program.

Figure 13: Vane Formed Using NCX 5102 Slurry

CONCLUSIONS

Work in the HIP composition (Task 1) centered on the optimum slurry and forming condition for high green density and minimal green density variation. Experiments established that casting rates increase with weight % solids and magnitude of applied pressure.

A casting model was developed which accounts for green body density gradients through the use of a porosity-constitutive relationship. Computer solutions provide cake thickness and density (porosity) distribution as a function of time.

A high solids loading (48 v%) suspension was developed by high energy attrition milling (HEAM). Also, surfactant evaluation was completed to achieve optimal slurry.

A major milestone demonstrated superior mechanical properties of the HIP composition processed for minimal density gradients. Tensile specimens were used for RT fast fracture strength and flexure specimens for stress rupture life. The crystallization treatment was used in the process to accentuate the HT mechanical behavior. Results were dramatic: 6/6 specimens survived 200 hours at 1370°C under 300 MPa and 5/6 specimens under 350 MPa survived. This superior HT performance was coupled with an excellent RT mean tensile strength of 842 MPa.

The shape forming capability of closed-loop-processed slurry was demonstrated through the casting of APU rotor vanes. These possessed superior surface quality relative to vanes produced using standard processing with agglomerated powder. Also, GPa level strength data of flexure specimens from associated slurry batches suggest that the vanes have superior strength as well.

In the GPS composition processing (Task 2), work focused on the role of pH and surfactant on green density. Kinetics of aqueous-based, magnesium-containing slurries showed that short-term slurry aging (4 hours) enhances green density to levels comparable to that of baseline $\text{Si}_3\text{N}_4 - \text{Y}_2\text{O}_3$ slurry. Thus, a suitable approach for full scale colloidal processing of the GPS composition was established. Strength data for the experimental material (NCX 5400) suggest that it ranks very high relative to other silicon nitride materials in terms of tensile/flexure strength ratio, a measure of volume quality. This high quality was derived from the closed-loop colloidal process employed in the program.

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APPENDIX 1: AQUEOUS INJECTION MOLDING OF Si_3N_4 BY THERMAL GELATION

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INTRODUCTION

Processing silicon nitride monoliths using injection molding is limited by the surface chemistry of the silicon nitride particles which, in turn, prevents the use of environmentally benign surfactants in aqueous suspensions.^{1,2} Our goal in this project has been to develop methods for shape forming Si_3N_4 monoliths using hyperloaded aqueous suspensions. We have determined that coating the Si_3N_4 particles with an oxide layer provides a method with strong potential for successfully gelcasting silicon nitride. Adjunct to this work has been a study on the use of surfactants to passivate the surface of magnesium oxide particles in aqueous suspension.

Shape forming requires an effective method for controlling the liquid to solid transition; for this reason, we have emphasized the use of thermal casting (e.g., gelcasting) for processing hyperloaded suspensions. Our initial studies involved the gelcasting of alumina suspensions, which we use as a model system for determining the general process parameters important to gelcasting. Attempts to apply our results to the gelcasting of Si_3N_4 suspensions were disappointing; we thereupon determined that gelcasting was more likely to succeed when the surfaces of the Si_3N_4 particles were converted to an oxide. The use of an oxide surface should simplify the processing through the presence of a well understood surface. In addition to improving the processability, coating the surface of the Si_3N_4 particles is known to be an effective method for introducing additives into the particle suspension and thence into the final monolith.^{3,4}

GELCASTING HYPERLOADED ALUMINA SUSPENSIONS

Figure 1-1 is a schematic of the gelcasting process used to cast alumina suspensions from stabilized suspensions. Based on the success of using agarose solutions to gel high-solids-content alumina suspensions, a similar system was used to process aqueous silicon nitride suspensions. The dispersants used were S1, S2, and S3, all of which act to disperse alumina powders in water. The gelling polymer was agarose, also soluble in water. S1 and S2 are commonly used dispersants whose behavior in aqueous suspensions is understood. Dispersant S3 is biogenic in origin, environmentally benign, and burns out with little residue.⁵⁻⁷

To successfully cast an alumina suspension, two approaches can be used to minimize the suspension viscosity: (i) a dispersant is used to prepare a low-viscosity suspension which is then gelled by the addition of the gelling solution (as shown in

Figure 1-1), and (ii) a single polymeric or monomeric species is used that first stabilizes the suspension and then is itself gelled. In the first procedure, the key difficulty is the incompatibility of the dispersant with the gelling solution. In the second, the identification of an appropriate moiety is the necessary first step. Our work in this project has been concerned with finding appropriate gelling agents.

Figure 1-1: A schematic of the gel-casting method of forming as developed for alumina suspensions.

We have demonstrated that alumina suspensions can be cast using agarose solutions, which reversibly gels at $\sim 40^{\circ}\text{C}$ as the temperature of the solution is lowered.^{5,6} However, when a suspension of S1 - stabilized alumina particles is added to agarose solution above 45°C , the dispersant S1 was found to induce phase separation in the agarose solution resulting in (i) low packing densities ($<55\%$ by volume) in the green body, and (ii) increased sintering temperatures, Figure 1-2. The agarose/S3 system approaches the performance of colloidally cast, high-solids-content suspensions. Surface modification using short, highly charged molecules imparts the necessary anionic surface to the particles while maintaining miscibility with agarose gelling solution.

To eliminate the phase separation behavior, either of two strategies can be used: (i) decrease the molecular weight of the dispersant, or (ii) modify the agarose through the addition of anionic side groups. Dispersant S3 is seen to be an effective dispersant for alumina in aqueous suspensions and its use results in higher densitites at lower temperature (Figure 1-2). Suspensions stabilized with S3 are fluid at solid loadings above 55 volume %, but the reasons behind this observation are not well understood. We do know that dispersant S3 strongly adsorbs onto the alumina surface and imparts a strong negative surface charge, Figure 1-3.

Figure 1-2: Sintering behavior of alumina processed by
 (i) stabilization with surfactant S3 and agarose gelation,
 (ii) agarose/S2 gelation, (iii) dry pressing, and
 (iv) colloidal casting of 60 vol % suspensions.

Figure 1-3: The surface charge of α -alumina particles prepared with 0.5% (dry weight basis) surfactant S3 at different pH's.

PROCESSING OF HYPERLOADED Si_3N_4 SUSPENSIONS

Dispersion Studies on Uncoated Si_3N_4

The use of gelcasting to process alumina provided necessary but not sufficient guidelines for using the same process with silicon nitride powders in aqueous suspensions. Past studies indicate that surface coating is required to achieve high solids loading in such systems.⁸⁻¹⁶ For example, a solids loading of 47.5 vol% is reported for aqueous Si_3N_4 suspensions containing Dolapix PC33 polyelectrolyte (an anionic polyacrylate similar to S1) as the dispersant and $\text{Y}_3\text{Al}_5\text{O}_{12}$ as an additive.¹⁶ However, nonaqueous

systems are far more successful in preparing highly concentrated suspensions.^{17,18} In Figure 1-4 the results of using polyguluronic acid dispersant (S4) in water and a fluorinated polyester in paraffin are compared; the non-aqueous system is clearly seen to be the more successful in preparing high density green bodies.

Figure 1-4: Comparison between aqueous and non-aqueous processing of silicon nitride suspensions.

Bergstrom¹ has described the surface of silicon nitride in non-aqueous suspensions as consisting primarily of silanol groups with a few secondary amine groups. He notes that the surface is amphoteric and used different probe molecules in non-aqueous solvents to demonstrate that highly acidic or basic molecules had the highest amount adsorbed on the surface, confirming the amphoteric nature of the silicon nitride surface in such systems. In an attempt to replicate Bergstrom's results in aqueous suspensions, a number of similar probe molecules were used to prepare aqueous silicon nitride suspensions. It is apparent from the results shown in Table 1-1 that the solid volume fractions were not improved relative to loadings achieved by simple electrostatic stabilization of Si_3N_4 in water. Although the amphoteric surface of silicon nitride tightly adsorbs either strongly acidic or basic molecules in non-aqueous suspensions, the deleterious effect of water is apparent in the values for solids loadings.

Table 1-1: Aqueous silicon nitride suspensions using substituted dispersants.

Surfactant	Vol %	pH	drops NH_4
none	44	10.0	as required
S4	43	10.1	30
S5	44	10.3	51
S6*	39	10.1	33
S3	30	9.5	36
S7	44	10.1	76
S8	42.8	6.6	2
Agarose	41	10.1	as required

Dispersion Studies on Coated Si_3N_4

Although the above work was not an exhaustive survey of potential surfactants, the failure of the probe molecules in water indicates that the hydration of the silicon nitride surface prevents effective electrosteric stabilization with polymeric agents. It is known that the surface of the silicon nitride can be readily reduced using either weak organic acids or by heat treatment in an reducing atmosphere, thereby increasing its hydrophobicity.^{1,2} On the other hand, the surface can be oxidized using either oxide coatings or heat treating in an oxidizing atmosphere. Since coating offers the dual advantages of providing a surface that is like that of silica or alumina and a method by which other metal oxides may be added to the powder to improve sintering and/or densification, we chose to coat the surface of as-received Si_3N_4 powders with either aluminum or silicon oxide.

Initially, as-received silicon nitride powders were coated using a method based on the procedure of Tewari.⁴ Yttrium nitrate was dissolved in a stabilized aqueous suspension of Si_3N_4 to coat the particles and the nitrate layer was then converted to oxide, Figure 1-5. TEM imaging has shown the presence of an amorphous layer on the surface of particles, but, as shown in Table 1-2, the resulting loadings in aqueous suspension were not high. Samples of powders coated with metal nitrates performed no better in suspension than powders that had been electrostatically stabilized without dispersant. The coating is not complete nor fully converted to oxide as shown by high-resolution TEM analysis.

Figure 1-5: Schematic of coating silicon nitride powders using metal nitrates.

Table 1-2: Summary of suspensions made using agarose as a gelling agent

Dispersant	pH	Coating	Vol % agarose added	Vol % Si_3N_4
None	10.1	None	1.1	40.0
1.1 Wt % S3	8.0	None	2.0	35.5
None	9.5	Yes	2.0	34.9
1 Wt % S4	5	Yes	2.0	38.8
0.8 Wt % S3	8.5	Yes	2.0	34.0

To improve the quality of the oxide layer on the Si_3N_4 particles, we modified Tewari's procedure using aluminum sec-butoxide for alumina coatings and tetraethoxysilane (TEOS) for silica coatings. As shown in Figure 1-6, alumina was successfully deposited on the dispersed silicon nitride particles.

Figure 1-6: TEM Images of alumina-coated silicon nitride particle.

In Figure 1-7, the change in the ξ -potential of Si_3N_4 suspensions containing increasing amounts of TEOS indicates that the silicon nitride particles begin to resemble silicon oxide particles in suspension. An increasing amount of TEOS added tends to make the silicon nitride act more like silica until 0.09 grams is reached.

Figure 1-7: Electrophoretic mobility of silica-coated silicon nitride.

Diffuse reflectance IR spectroscopy of the silica-coated powder further indicates the existence of a silica layer on the silicon nitride particles, Figure 1-8. The presence of the shoulder near 1000 cm^{-1} indicates a modification of the surface of the powder. The decrease in hydroxyl content after calcining is apparent above 3000 cm^{-1} ; the absence of free silica indicates that the TEOS condensed on the surface of the silicon nitride and did not form silica particles in the original solution.

Figure 1-8: FTIR reflectance measurements on as-received, TEOS-coated, and TEOS-coated and calcined powders.

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APPENDIX 2: HIGH ENERGY AGITATION MILLING OF SILICON NITRIDE POWDER

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INTRODUCTION

One of the key issues in the production of high-density, ceramic green bodies has been the development of slips with high-solids content. High energy agitation milling (HEAM) has been shown to be an effective and technically superior method in the preparation of high-solids-content slips. Since a large number of variables can affect the properties of a milled slip, an experimental program was developed to investigate the critical variables that influence the production of silicon nitride slips containing greater than 75% solids by weight (%wt). The milling process and resultant green bodies were studied to gain better understanding of the overall process. The physical aspects of milling have been coupled with continuing investigations into the surface chemistry of silicon nitride in various milling environments to develop a more complete view of the physico-chemical processes that are involved in the production of high solids content slips.

The production of ceramic green bodies with a high density (>67% of theoretical density) from a highly loaded slip is of considerable importance. Of further importance is the consistent production of high-solids-content slips with specific physical properties that will enhance the production of parts with desired mechanical properties. The particle size distribution (PSD), specific surface area (SSA), and interface chemistry parameters of the powder were measured for all tests that were conducted to determine the ultimate loading of the slip and density of the green ceramic. The tailoring of a particular size distribution can be greatly enhanced by the selection of surfactants type and concentration, and milling parameters. Previous work in our laboratory has shown that the choice of feed rate, mill speed, media size, media loading, solids loading, type of media, and duration of milling will affect the physical properties of the milled powder. The present work has been focused on using these data as the basis and build upon the study of two dispersants to obtain high solids loading for a specific set of milling parameters, and to evaluate the resulting slips and green ceramics. In addition to this, we have attempted to produce a slip with narrowly defined physical properties by tracking the median particle size measurements as a function of milling time. Correlation of the apparent spherical diameter with the specific surface area measured on milled powder has allowed the milling to be terminated and the resulting slip to be cast based on a pre-determined specification of the median particle diameter and SSA.

OBJECTIVE

The objective of this investigation was the development of high density, fully dispersed, Si_3N_4 aqueous slips by the application of surface and colloid chemical characterization techniques to slips produced by HEAM. The SSA, PSD, and electrokinetic sonic amplitude (ESA) measurements were used to provide a detailed description of the complex physico-chemical interactions of the powders during the milling and casting processes.

EXPERIMENTAL

MATERIALS AND EQUIPMENT

A horizontally mounted high energy agitation ball mill (Model LME-1, Netzsch Industries) loaded at 75% by mill volume with 2 mm hot pressed Si_3N_4 media (Tosch Industries) was used during the experiments. The slurry was prepared with a pre-blended mixture of SN-E-3, SN-E-5, and SN-E-10 (Ube Industries). The powder was mixed in a borosilicate beaker with distilled water using a constant velocity, high-shear mixer (Pioneer Mfg. Co.). A 4% by weight of Y_2O_3 (5600, Molycorp) was added as a sintering aid. The pH of the slurry was measured and adjusted as necessary with reagent grade HNO_3 or $\text{NH}_4(\text{OH})$. The slurry from the mill discharge was pumped through a 9-mm-ID (0.375 in.) urethane tubing (Nalgene) by a variable-speed peristaltic pump (Masterflex-Cole Palmer Co.) and was returned directly to the beaker.

The slurry was sampled at various pre-determined intervals during the milling process and dilute sub-samples were analyzed for their PSD using a laser light scattering instrument (LA-900, Horiba Instruments Company). The remaining sample not used for sizing was dried for SSA determination using an automated volumetric type BET instrument (Autosorb-1, Quantachrome Corp).

Surfactant concentrations were chosen based on residual total carbon analysis (Rosemont DC-80, Rosemont Instruments), Figures 2-1 and 2-2, from dilute slurries prepared at a specific pH and surfactant concentration. An ammonium salt of polyacrylic acid (PAA), Darvan 821A, (R. T. Vanderbilt Co.), and an ammonium salt of polymethacrylic acid (PMA), Daxad 32, (W. R. Grace) were used as dispersants.

MILLING PROCEDURE

A 2-kg-batch of the silicon nitride powder mixture was subdivided into 1 kg, 500 g, and two 250-g-samples for ease of addition. Five hundred cm^3 of distilled water was mixed with ammonium hydroxide to increase the pH to 9.3. Next, the dispersant was added. Initially, 1 kg of the pre-blended powder was added while constantly stirring to produce a slurry of 66 wt% solids. The milling was begun with the mill rotational speed at an arbitrary low value until the slurry had made one pass through the mill. At this time the rotor speed was increased quickly to a predetermined set point, which was 2800 rpm in most tests. As the milled slurry thinned, due to particle size reduction and/or deagglomeration, the smaller sub-samples were added as quickly as

possible without causing a substantial decrease in either pumping rate or rotor speed. The Y_2O_3 was added either in a dry form or as an aged slurry in water after 20 minutes of silicon nitride powder milling. The aged slurry form of Y_2O_3 was prepared by adding the required amount of distilled water to the pre-weighed Y_2O_3 and shaking overnight. As the solids content to the slurry increased, more time was required between the additions of the additional silicon nitride. Extreme care was needed during the final additions as the viscosity of the slip could increase quickly, especially when the solids loading exceeded 75 wt%. Samples of the milled slurry were taken at predetermined intervals during each milling test and were either analyzed immediately or placed in a sample shaker overnight for PSD measurement. Sub-samples were also dried for SSA analysis. Milling was conducted as quickly as possible in spite of the above mentioned constraint. When the desired median particle size was reached, milling was stopped and the slurry was removed for shipment to Saint-Gobain/Norton Industrial Ceramics. At NIST, the slip was cast into 12.7 mm (0.5 in.) and 25.4 mm (1.0 in.) diameter pucks and 76.2 X 12.7 mm (3x0.5") bars.

RESULTS AND DISCUSSIONS

DISPERSANTS EVALUATION

The two dispersants, Darvan 821 A and Daxad 32, were evaluated using ESA, particle size distribution, adsorption isotherms, and slip casting of the resulting suspensions. These studies were conducted using Ube SNE-3 as the model powder at 2% by volume in aqueous environment. The goal of this activity was to decide on which of these dispersants gives improved dispersion, and the most appropriate concentration of the dispersant to provide an optimum dispersion. The results of these tests indicated the following:

1. Very little adsorption of these dispersants took place at pH 9.0. At lower pH values, significantly more of the added dispersant adsorbed on the silicon nitride. Based on these and other results, we concluded that in alkaline pH range of 9.0, the primary stabilization mechanism is electrosteric. The results of adsorption isotherms at three pH values for the two dispersants are presented in Figures 2-1 and 2-2. According to these data, even at pH 3.5 only a small fraction of the dispersant added to the slip gets adsorbed on the particles.
2. The degree of dispersion was about the same irrespective of which one of these dispersants was used. An example of the degree of dispersion from the two dispersants is shown in Figure 2-3. At the appropriate concentration levels, both dispersants yield the same particle size distribution. Below these concentration levels the particle size distribution was coarser, and higher concentrations did not provide enhanced dispersion.
3. The optimum concentration for attaining the maximum dispersion or the finest particle size distribution for these dispersants was different. The respective optimum concentrations for Daxad 32 and

Darvan 821A were in the range of 300 and 640 ppm based on the weight of the powder suspension. The optimum concentration was determined from ESA and green density data. The ESA response of the powder-dispersant system as a function of the dispersant concentration is shown in Figure 2-4, while the green density data are presented in Table 2-1. Slips were prepared from 30% weight suspensions. The optimum concentration data obtained from ESA results is strictly true only when the adsorption of dispersants is governed strictly by the electrostatic interactions. We know at this time that adsorption in the alkaline pH range is not entirely due to electrostatic interactions. Therefore, these data provide only an indication of the optimum concentration for maximum dispersion. However, these data in conjunction with the green-density data in Table 2-1 confirm that the range of concentrations is approximately correct.

Table 2-1: Summary of green density data for two dispersants at different concentrations.

Dispersant Conc. ppm	Green density, % Th.	
	Darvan 821A	Daxad 32*
240	62.3	-
640	63.0	-
800	63.4	-
150	-	62.4
300	-	62.0
500	-	62.0
1000	-	61.0

*Repeatability not as good as that for 821A

MILLING OF SILICON NITRIDE POWDER

Based on these data, we carried out the milling tests using Daxad 32 and Darvan 821A as dispersants at appropriate concentrations (300 ppm and 650 ppm for Daxad 32 and Darvan 821A, respectively) based on the weight of solids in the slip. A schematic of the experimental procedure shown in Figure 2-5 was used to gradually build the solids loading in the mill. As shown in this illustration, we start with approximately 65% by weight solids in the slip before any milling is initiated. The remaining powder is added to the slip during milling as the slip continues to deagglomerate. Therefore, depending on the amount of the powder to be added, longer milling time is needed to achieve higher solids loading. A number of variables were studied to achieve as high a solids loading as possible. The test results are summarized in Table 2-2. One of the early problems was related to more than desired decrease in the particle size distribution or higher than desired specific surface area of the final powder. This result was related to the requirement of longer milling times to add the extra powder to increase the solids loading to 80% by weight.

Figure 2-1. Adsorption isotherms of Darvan 821A at three different pH values.

Figure 2-2. Adsorption isotherms of Daxad 32 at three pH values.

Figure 2-3. Comparison of particle size distribution at pH 9.0 of SNE-3 using optimized concentrations of Daxad 32 and Darvan 821A.

Figure 2-4. Titration of Darvan 821A and Daxad 32 at pH 4.0 in presence of SNE-3. Interception of slopes at the break point indicates approximate optimum concentrations.

Figure 2-5. Schematic of slip preparation, sampling, and milling procedure. Note the addition points for powders.

Table 2-2. Summary of experimental parameters and the resulting powder characteristics.

Test #	Final Solids % wt	Rotor Speed, rpm	Green Density, g/cm ³	Dispersant, ppm	Size Data D ₂₅ D ₅₀ D ₇₅	SSA m ² /g
60	74.8	2800	-	Daxad-32, 300	0.40 0.58 0.81	10.6
62	75	2800	2.28	Daxad-32, 300	0.40 0.58 0.82	10.1
63	75	2800	2.22	Daxad-32, 300	0.40 0.57 0.81	9.8
64	80	2800	2.22	Daxad-32, 300	0.39 0.38 0.79	11.3
65	75.3	2800	2.27	Daxad-32, 440	0.36 0.50 0.72	11.4
67	75.8 77.5	2800	2.22	Daxad-32, 300	0.43 0.63 0.87	8.4
68	75.3	2800 1700		Daxad-32, 300	0.59 0.88 1.24	9.7
69	75.6 77.5	2800 2520	1.95	Daxad-32, 1000	0.40 0.59 0.83	10.9
70	75.8	2800 2765	2.13	Darvan-821A 650	0.40 0.54 0.78	9.8
72	80.0 74.9	1850 1650	2.04	Darvab-821A 350	0.50 0.79 0.94	7.4

EVALUATION OF MILLED SLIPS

The resulting slips were evaluated by the measurement of PSD, SSA, ESA, and green density. A summary of the properties of these slips is shown in Table 2-2. One special feature of these results is the high green density which is an evidence of a higher degree of dispersion of the slip milled in the HEAM. The slips were slip cast or CIPed followed by HIPing at St. Gobain-Norton. The flexural strength data from two of the tests are shown in Table 2-3. These values are smaller than those reported for this material by the St. Gobain Industrial Ceramics. The primary reason for the lower strength values is that HIP parameters may be different for green ceramics prepared by HEAM.

Table 2-3. Flexure strength data of HEAM milled slips.

Test #	Flexural Strength, MPa	
	NIST	Norton
1	690	-
2	603	-
56	-	725 (CIP'ed)
59	-	691

SUMMARY AND CONCLUSIONS

The dispersant optimization tests conducted at low concentrations indicated that both Darvan 821A and Daxad 32 provide the same amount of dispersion. However, the optimum concentration to obtain the best dispersion was different for these dispersants due to the differences in their chemical compositions.

Using these results, milling tests were conducted using HEAM on a silicon nitride powder provided by Saint-Gobain/Norton Industrial Ceramics. In a number of tests, depending on the milling parameters, high solids concentration, as high as 80% by weight, was obtained.

The slips were evaluated at NIST and Saint-Gobain/Norton Industrial Ceramics. These slips produced high density green compacts. However, the HIP'ed densities and mechanical properties were not as high as those obtained from the conventionally milled slips. This difference is attributed to the fact that the HIP parameters used for these samples may not be the best for this purpose.

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