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**OAK RIDGE
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LOCKHEED MARTIN

**CRADA Final Report
for
CRADA Number Y-1294-0251**

**CERAMIC COMPONENT MANUFACTURING PROCESS
DEVELOPMENT**

Sam Robinson
Lockheed Martin Energy Systems, Inc.
Bob Baker
Ceradyne, Inc.

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Abstract

Ceramic materials are well suited for applications where temperature, wear, and corrosion resistance are necessary. The toughness and wear resistance properties that make ceramics desirable, also make fabrication of parts difficult. The objective of this CRADA was to increase the grinding efficiency on Ceradyne Incorporated silicon nitride. This was to be accomplished through optimization of grinding wheel life and increasing silicon nitride material removal rates. Experiments were conducted to determine the relationship between grinding parameters, wheel wear, and material removal rates. Due to excessive, unexplained variation in the experimental results, a consistent relationship between the selected grinding parameters and wheel wear could not be established. Maximum material removal rates were limited by spindle and table drive power.

Additional experiments were conducted to evaluate high speed grinding. When compared to conventional grinding speeds, the material removal rates using high speed grinding (13,000 SFM) increased by a factor of five to ten with no degradation of fracture strength.

CRADA Objectives

This CRADA addressed increasing the efficiency of Ceradyne ceramic part grinding process. Objectives included maximizing the material removal rate, and extending the life of the grinding wheel.

Success in Meeting CRADA Objectives

The objective of maximizing the material removal rate was met. Under the constraint of using their Harig Model 618 grinders, Ceradyne is operating at or near the optimum material removal rate. Attempts to increase material removal rates will cause the grinder to stall under the increased load. The study indicated that material removal rates on Ceradyne silicon nitride can be increased by a factor of five to 10 by high speed grinding with no degradation of MOR fracture strengths. Implementation will require capital investments in high speed spindles and a safety analysis by the industrial partner. Attempts to extend grinding wheel life were inconclusive. Due to excessive, unexplained variation in the experimental data, the impact of grinding parameters on wheel wear could not be quantified.

DP Benefits

The CRADA allowed data and experience to be gathered on a ceramic material that has the potential for use by the DOE. A general purpose technique for measuring grinding wheel wear was developed. Experiments with high speed grinding indicate that the ceramic material removal process may be made more efficient at high wheel speeds.

Introduction

Ceramic materials are finding uses in areas where toughness, wear resistance, and good high temperature properties are needed. The desirable properties of toughness and wear resistance inherently causes the material to be difficult to grind during the part manufacturing process. This CRADA addressed issues aimed at increasing the efficiency of Ceradyne ceramic part grinding process. Goals include reducing labor by increasing the material removal rate, and reducing grinding wheel costs by extending the life of the grinding wheel. This must be accomplished without degrading the quality of the finished product.

Grinding ratio, GR, is defined as the volume of ceramic part material removed during grinding divided by the volume of grinding wheel worn away during the process. A high grinding ratio indicates low wheel use and a corresponding reduction in costs. The highest grinding ratio, however, may occur at a set of grinding parameters that result in a low material removal rate. In this case, an economic decision would be made to balance labor costs against wheel costs.

A set of experiments were planned to quantify the effects of grinding parameters on GR, material removal rate, grinding machine forces, and part quality. These tests involved surface grinding reaction bonded silicon nitride tiles.

Experimental Approach

The initial experimental matrix was developed with parameters centered on Ceradyne's current grinding parameters for silicon nitride which are listed in Table 1. Parameters varied in the experiment were grinding wheel grit, wheel hardness, wheel speed, depth of cut, cross feed, and table feed. Measurements of grinding forces, acoustic emission, and spindle horsepower were made during

Table I. Ceradyne Parameters - Surface Grinding Silicon Nitride

Grinding Wheel Specifications

Type	Resin	Resin	Resin
Grit	120	180	320
Outside Dia (Inch)	7	7	7
Width (Inch)	0.5	0.5	0.5
Speed (RPM)	2850/3450	2850/3450	2850/3450

Parameters for Cross-Feed Grinding

Depth of Cut (Inches)	0.0015	0.001	0.0005
Table Speed (IPM)	30	30	30

Cross-Feed Rate

0.333 Inches/Pass

grinding. Material removal rate was calculated from depth of cut, cross feed, table feed, and elapsed time. Wheel diameter measurements before and after grinding were used to calculate the

volume of wheel material removed for GR.

The assortment of wheels acquired for the experiments is shown in Table 2. Rustlick G-1066D in a concentration of 20:1 was used for coolant in all experiments. Ceradyne provided four inch square by 0.5 inch thick silicon nitride tiles.

A Harig Model 618 surface grinder was used. The grinder was instrumented with a package designed, fabricated, and installed by the Institute of Advanced Manufacturing Sciences (IAMS). Grinding forces were measured in three axes using an arrangement of Kistler load washers on the table. Acoustic emission, spindle vibration, and spindle power were also monitored. Data were collected on a personal computer using LabView software.

Table II. Wheels for the Experiment

<u>Grit</u>	<u>Hardness</u>
120	N
120	T
300	R
480	N
480	T

7 Inch Diameter
0.5 Inch Width
1.25 Inch Arbor
100 % Diamond Concentration
General Diamond, Inc.

The experimental process involved mounting a test specimen on the grinder, then measuring the grinding wheel diameter using the procedure described below. A predetermined volume of silicon nitride was then removed using parameters specified in the experimental plan. Data were collected during grinding. The wheel diameter was measured again to determine wear.

Grinding Wheel Diameter Measurements

An improved technique for accurately determining grinding ratios in a laboratory setting was developed. Grinding ratio is a calculated number that is based on direct or indirect measurement of the change in volumes of the grinding wheel and the workpiece. The simplest way to estimate the change in wheel volume is to directly measure the change in wheel diameter using a hand-held micrometer. However, the change in wheel diameter is usually too small (on the order of 20 micro-inches) to measure accurately by this method unless several cubic inches of test-specimen material are ground for each test. The method is not practical in a laboratory because it consumes large quantities of ceramic, grinding wheels, and labor.

Figure 1a. Grinder Set-up

A precision coordinate measuring machine (CMM) is a much better

Grind Step In
Measurement Block

Grind Ceramic
Test Specimen

Grind Step In
Measurement Block

To CMM.

Compare step height
before and after grinding
test part. The change in
height is the change
in test wheel diameter.

Figure 1b. Wheel Wear Measurement

instrument for making accurate and repeatable measurements of the wheel diameter. However, if the wheel is removed from the grinding spindle to perform the initial, before grinding, measurement, it must be trued when it is reinstalled on the spindle. This truing process invalidates the measurement. An improved technique was obviously needed for measuring changes in diameter without removing the wheel from the spindle.

Figure 2. Measurement Block

This was accomplished with the set up shown in Figures 1a and 1b. A 0.5-inch-wide, 6-inch-diameter wheel was mounted on the spindle along with the wheel being tested. All test wheels were 7 inches in diameter. Measurement blocks, shown in Figure 2, were fabricated of relatively soft aluminum oxide with a step height of approximately 0.5 inch. Before grinding a test specimen, a measurement block was mounted on the table of the grinder. A step was finish ground along one edge, with the test wheel grinding one step surface and the reference wheel grinding the other step surface. Next, the test grind on the silicon nitride was made. Because the test wheel had a larger diameter than the reference wheel, the reference wheel did no grinding. Another set of steps was finish ground along the other edge of the measurement block after the test grind. CMM measurements of the heights of the before-test-grind step and the after-test-grind step allowed a change in step height to be calculated. This change was the radius change of the test wheel.

Experimental Results

The initial experimental matrix is shown in Appendix A. When the matrix was used, the table and spindle on the Harig stalled if the depth of cut (DOC) exceeded 0.0015 inch. The matrix was redesigned such that the maximum DOC was 0.0015 inch. This redesigned matrix is in Appendix B.

The matrix included replications of parameters in runs 2, 6, 12, and 19. Analysis of replications indicated significant scatter in the wheel wear data. Approximately 50 experimental runs were made to identify the source of the scatter, and reduce the contribution of measurement error. A critical factor was the selection of CMM measurement points on the steps of the measurement block. This was due to the fact that the wear on the test wheel was uneven, which created non-flat step surfaces on the measurement block. Initially, measurements were taken at the discrete points shown in Figure 3a. After several experiments, the scheme shown in Figure 3b which uses 2-dimensional profile scans was developed to minimize

measurement error. The profile of the step was measured in three different planes for both the before-grinding and after-grinding side of the block. These scans accurately depicted the irregular wear patterns that occurred on the face of the grinding wheel. The CMM software computed a straight line that represented the mean height for each scanned profile. The difference in height between these lines was computed. The result was three sets of heights that were averaged to determine the step height.

Figure 3a. CMM Point Measurements

A summary of eight typical grinding runs is shown in Figure 4. All eight runs were made using the same parameters. The top graph is the CMM data of the difference in before and after step height for the eight runs. This change corresponds directly to the change in wheel radius. The middle graph is a plot of the grinding ratios calculated from the data in the top graph. To verify the repeatability of the CMM measurement, duplicate measurements were made on each run. The top and middle graph indicate each measurement is repeatable. To further verify the measurement process, an additional measurement block was set aside to be used as a measurement standard. This standard

was checked in conjunction with each of the eight runs. The bottom graph shows eight repeated CMM measurements on the control standard.

The variation in wheel wear in replicated silicon nitride grinding runs was on the order of 100%. The source of variation in the grinding process has not been found. This variation made it impossible to determine the relationship between parameters and the grinding ratio.

The performance of a high speed wheel was evaluated. The wheel used in this experiment was rated at 7600 RPM. Appendix C contains the grinding parameters and results. This study

indicates that material removal rates on Ceradyne silicon nitride can be increased by a factor of five to 10 with no degradation of MOR fracture strengths.

Figure 3b. CMM Line Scans

Figure 4. Results of Eight Runs

Conclusions and Recommendations

Under the constraint of using Harig Model 618 grinders, Ceradyne is operating at or near optimum conditions. Attempts to increase material removal rates will cause the grinder to stall under the increased load. Variations in the grinding process were evidenced in scatter in the wheel wear measurement data. This scatter masked the impact of grinding parameters on wheel wear. It is conceivable that variations in the composition of the wheel itself are a major contribution to the scatter observed in the wheel wear data. Analysis of the wheel was outside the scope of this study.

A technique was developed to characterize grinding wheel wear. This technique is an improvement over other wheel wear methods because an accurate wheel diameter measurement can be made without removing the wheel from the grinder.

During the high speed grinding test at approximately 13,000 sfpm, material removal rates were increased by a factor of five to 10 over conventional speed grinding. There was no degradation of fracture strength of the MOR bars fabricated by high speed grinding when compared with bars fabricated using conventional speeds. There should be additional experimentation to confirm this result. The high speed work was on silicon nitride. It could be expanded to include titanium diboride, silicon carbide, and boron carbide tiles and silicon carbide rollers. Implementing high speed grinding at Ceradyne would require investments in high speed spindles. There are safety issues related to high speed operation that would also need to be evaluated. The investments and risks must be weighed against the increased material removal rates to determine if high speed grinding would be cost effective.

Inventions

There were no inventions made or reported.

Commercialization Possibilities

There were no commercialization possibilities that resulted from this CRADA.

Future Collaboration

There are no plans for future collaboration.

Appendix A

CERADYNE CRADA
PROCESS PLAN for SiN -- PHASE I
(12/294)

Objective: Optimize process parameters for conventional surface grinding for typical Ceradyne operations. Ceradyne provides their baseline parameters currently being used in their shop. Our optimization plan is to perform a series of grinds using parameters chosen by a statistical design that is centered around the Ceradyne baseline. Key parameters of grit size, depth of grind, and surface feet per minute (wheel speed) will be varied to obtain optimization data. Optimization goals are to increase material removal rates (Q') and/or grinding ratios (GR) while maintaining adequate strength and wieble modulus.

Plan: A statistically designed test matrix will be developed around the baseline parameters supplied by Ceradyne:

Baseline Parameters for Crossfeed Grinding:

Material: Ceradyne _____ Sintered Reaction Bonded Silicon Nitride
Grinding Wheel: 180 grit diamond/resin bonded, 100 concentration, 7" diam., 0.5" wheel width running at 2850 or 3450 rpm.
Depth of Grind: 0.001"
Table Speed: 30 (ft/min)
Coolant: Rustlik
Crossfeed: 0.333 inch

Test Matrix:

<u>Run #</u>	<u>Wheel Speed</u>	<u>Depth of Grind</u>	<u>Grit Size</u>	<u>Hardness</u>
<u>Block 1</u>				
1	4500 (rpm)	0.0025 (inch)	480	N
2	2850	0.0025	120	T
3	3650	0.0020	300	R
4	2850	0.0015	480	N
5	2850	0.0015	480	T
6	2850	0.0025	120	N
7	4500	0.0025	480	T
8	4500	0.0015	120	N
9	3675	0.0020	300	R
10	4500	0.0015	120	T
<u>Block 2</u>				
11	2850	0.0015	120	N
12	4500	0.0025	120	T
13	3675	0.0020	300	R
14	4500	0.0015	480	N
15	2850	0.0025	480	T
16	3675	0.0020	300	R
17	4500	0.0025	120	N
18	2850	0.0015	120	T
19	4500	0.0015	480	T
20	2850	0.0025	480	N
<u>Block 3</u>				
21	3675	0.0020	300	R

22	3675	0.0030	300	T
23	3675	0.0020	300	R+2
24	5325	0.0020	300	T
25	* 3675	0.0020	300	R
26	3675	0.0020	120	T
27	3675	0.0010	300	T
28	3675	0.0020	480	T
29	3675	0.0020	300	R-2
30	2025	0.0020	300	T

All runs to be performed with the following parameters held constant:

Table Speed: 30 ft/min
Coolant: Rustlik
Crossfeed: 0.333 inch

Billets supplied by Ceradyne are 3" x 3"x by 3/8" minimum.

Plan is to mount billet on Harig machine such that top surface will be ground. With freshly trued and dressed wheel and parameters determined by the test matrix, remove 0.028" of material (volume removed = 0.25 in³). All force data and wheel wear data will be recorded. Q' and grinding ratio will be calculated. Surface finish will be measured.

Grind will be repeated using parameters for the next run until all runs are completed (Approximately 1.5 billets will be consumed)

Ceradyne and MMES will consult and agree upon the best set of parameters to perform a strength and wieble modulus evaluation. These parameters will be used to prepare the experimental side for a minimum of 20 bars from the remaining 1/2 billet.

The bars will be completed and broken. Evaluation of strength and wieble will be made.

Results will be reported

[SiNtst1]

Appendix B

CERADYNE CRADA - PHASE 1

EXPERIMENTAL PLANS FOR OPTIMIZATION OF SURFACE GRINDING PROCESS

Goal:

Find settings of process factors that maximize material removal rates, minimize wheel wear, and maintain adequate strength and wieble modulus.

Strategy:

Step 1. Analysis of the data from the screening expeirment will allow us to :

- (a) determine which factors have a statistically significant affect on the measured responses,
- (b) rank the responses in order of importance,
- (c) determine if all factors tend to yield linear responses or if at least one factor yields a curvilinear response,
- (d) determine experimental error (how well a given run can be replicated).

The suggested screening experiment requires 20 runs.

Step 2. Run an optimization experiment to determine optimum setting of the three (or at most four) factors found to be most significant in Step 1. The optimization experiment will allow for estimation of interaction and curvilinear effects.

For three factors, the optimization experiment will require approximately 18 runs.

Experimental Factors:

<u>Factor:</u>	<u>No. Levels</u>	<u>Levels</u>
Cross Feed	3	0.15, 0.25, 0.35 in./pass
Table Feed	3	10, 20, 30 ft./min.
Wheel Speed	3	3000, 4000, 5000 rpm
Depth of Cut	3	0.0005, 0.0010, 0.0015 in./pass
Wheel Grit Size	3	120, 300, 480
Wheel Hardness	3	N, R, T (N=soft, R=medium, T=hard)

Constant Factors:

<u>Factor</u>	<u>Level</u>
Part Material	Ceradyne Sintered reaction bonded silicon nitride
Machine	Bridgeport Harig CNC II
Wheel Vendor	General Diamond Wheel
Wheel Diameter	7 in.
Wheel Bond Type	Resin
Wheel Grit Type	Diamond
Wheel Grit Conc.	100 (25% diamond per cm ³)
Wheel Width	.5 in.
Coolant Type	Rustlick
Wheel Truing	All wheels will be trued between runs

Measured Responses:

Response:

Determined By:

Grinding Ratio (GR)

Calculated from volume of material removed
(see below)

Material Removal Rate (Q')

Calculated value from feed rates and wheel width
 $Q' = (\text{cross feed} * \text{table feed} * \text{depth of cut}) / (\text{wheel width})$

Surface Finish

Visual, stylus

$GR = (\text{volume of part material removed}) / (\text{volume of wheel material removed})$

Volume of wheel material removed will be determined from CMM measurements taken on a block of aluminum oxide before and after each cut.

Proposed Test Matrix for Crossfeed Grinding Experiment

Run No.	Cross Feed (in.)	Table Feed (ft./min.)	Wheel Speed (rpm)	Depth of Cut (in.)	Wheel Grit Size	Wheel Hardness
<u>Block 1</u>						
1	0.250	60	3000	0.0015	480	N
2	0.175	40	4000	0.0010	300	R
3	0.250	20	3000	0.0005	480	T
4	0.100	20	5000	0.0005	120	T
5	0.100	60	5000	0.0015	120	N
6	0.175	40	4000	0.0010	300	R
7	0.100	20	3000	0.0015	480	N
8	0.250	60	5000	0.0005	120	T
9	0.100	60	3000	0.0005	480	T
10	0.250	20	5000	0.0015	120	N
<u>Block 2</u>						
11	0.250	20	3000	0.0015	120	T
12	0.175	40	4000	0.0010	300	R
13	0.250	60	5000	0.0015	480	T
14	0.100	20	3000	0.0005	120	N
15	0.100	60	5000	0.0005	480	N
16	0.250	60	3000	0.0005	120	N
17	0.100	20	5000	0.0015	480	T
18	0.100	60	3000	0.0015	120	T
19	0.175	40	4000	0.0010	300	R
20	0.250	20	5000	0.0005	480	N

Appendix C

HIGH SPEED WHEEL VERSUS CERADYNE BASELINE

FRACTURE STRENGTH (ksi)					
Wheel Speed	Table Speed (fpm)	Depth of Cut (in.)			Summary Statistic
		0.001	0.0008	0.0005	
HIGH SPEED WHEEL	60	4	4	4	No. of MOR Bars
		100.13	98.62	98.43	Mean
		4.51	2.67	5.31	Std. Dev.
		25.68	40.73	21.45	Weibull Slope
	30	4	4	4	No. of MOR Bars
		100.33	101.28	98.99	Mean
		4.32	2.31	3.85	Std. Dev.
		25.94	50.6	29.06	Weibull Slope
	All HIGH SPEED SAMPLES COMBINED			24	No. of MOR Bars
				99.63	Mean
		Samples machined and tested at ORNL		3.66	Std. Dev.
				33.39	Weibull Slope
CERADYNE BASELINE	ALL CERADYNE BASELINE SAMPLES Samples machined at Ceradyne and tested at ORNL			30	No. of MOR Bars
				78.62	Mean
				10.81	Std. Dev.
				8.29	Weibull Slope

GRINDING CONDITIONS

FACTOR	CERADYNE	ORNL
Machine	Harig	Harig CNC II
Material	Reaction bond silicon nitride	Reaction bond silicon nitride
Wheel OD	7 in.	7 in.
Wheel bond type	Resin	Resin
Wheel width	1/2 in.	1/2 in.
Wheel grit type	Diamond	Diamond
Wheel grit size	320	320
Wheel grit concentration	100	100
Coolant	Rustlick	Rustlick
Step over	0.333 in./pass	none
DOC per pass	0.0001 in.	0.0005, 0.0008, 0.001 in.
Total DOC	unknown	0.004 in.
Table speed	30 fpm	30, 60 fpm
Wheel speed	2850/3450 rpm	7,000 rpm

Comparison of High Speed Wheel Results with Ceradyne Baseline

Average Fracture Strength with 1-Standard Deviation Error Bars

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