PROCESSING OF CERAMICS— SURFACE FINISHING STUDIES

Final Technical Report

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by

R. SEDLACEK and P. J. JORGENSEN

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13. ABSTRACT

The SRI expanded ring test was used to determine the tensile strength of high purity, dense alumina. The test materials were prepared in five different nominal grain sizes ranging from 10 to 50 μm. The blanks were diamond ground to final dimensions by a technique developed earlier in the program. It was found that in grinding this material extensive damage occurred which had not been observed in any other alumina body ground under identical conditions. The only difference in strength was found between groups of specimens having grain sizes equal to or smaller than 30 μm, and those whose grain size was equal to or larger than 40 μm. The vacuum strength of this material was the same as the strength in air regardless of grain size. In all other aluminas investigated previously in this program, the vacuum strengths were considerably higher than the strengths measured in air.

Griffith's theory was applied to test the data, assuming that the grain size determined the flaw length, and a good correlation between observed and calculated strength values was obtained for all aluminas tested with the exception of the high purity alumina. In this material the mechanical damage occurring during grinding extended beyond the first grain boundary in the smaller grain size samples, and thus the mechanical damage controls the strength. When the grain size is equal to or larger than the mechanically damaged region, the microstructure controls the strength.

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Effect of microstructure on strength						
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Vacuum strength						
Stress corrosion						

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FOREWORD

This work was performed by Stanford Research Institute under Contract N00019-70-C-0179 with the United States Department of the Navy. Work was administered under the direction of the Naval Air Systems Command. Mr. Charles F. Bersch was the project monitor. This report covers work conducted between 1 April 1970 and 31 July 1971. Personnel responsible for carrying out the research were Rudolf Sedlacek and Paul J. Jorgensen.

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ABSTRACT

The SRI expanded ring test was used to determine the tensile strength of high purity, dense alumina. The test materials were prepared in five different nominal grain sizes ranging from 10 to 50 μm. The blanks were diamond ground to final dimensions by a technique developed earlier in the program. It was found that in grinding this material extensive damage occurred which had not been observed in any other alumina body ground under identical conditions. The only difference in strength was found between groups of specimens having grain sizes equal to or smaller than 30 μm, and those whose grain size was equal to or larger than 40 μm. The vacuum strength of this material was the same as the strength in air regardless of grain size. In all other aluminas investigated previously in this program, the vacuum strengths were considerably higher than the strengths measured in air.

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I INTRODUCTION

Brittleness and a relatively low tensile strength are the main obstacles in the broader acceptance of ceramics as a structural material although they possess other properties which make them in many respects superior to metals. Brittleness is an intrinsic property of these materials, and it is doubtful that the problems associated with brittleness can be alleviated, except by proper design based on a thorough knowledge of the stress conditions which the ceramic will experience in service.

The tensile strength of ceramics presents a more complicated problem. In earlier literature, tensile strength data show such scatter that ceramics were considered to be unpredictable under stress. The reason for the data scatter has been since traced to the use of ill-suited test methods, and presently techniques exist that not only give reproducible results, but also are sensitive enough to detect various factors that affect tensile strength. This development, however, can only produce more meaningful data, it cannot improve strength. The theoretical strength of ceramics and brittle materials in general is believed to be approximately 1/10th of the value of the modulus of elasticity. The highest engineering strengths of polycrystalline oxide ceramics known to date are lower by about two orders of magnitude. The exact reason for this discrepancy is not known, but it may be assumed that it is due primarily to our lack of understanding the manufacturing process and the ensuing inadequate control of it.

The ceramic manufacturing process consists of several steps which are not independent of each other and therefore cannot be studied individually. The only step which can be at least partially isolated

is the final shaping of fired ceramics. This operation determines to a large degree the quality of the surface.

Since the formulation of Griffith's flaw theory, it has been generally accepted that the weakness of ceramics is due to the presence of submicroscopic cracks on the surface. At the crack tips, which may have dimensions on the order of a few lattice spacings, an applied external load can result in tensile stresses approaching theoretical thresholds. As a result, the crack propagates and failure ensues. This concept has been verified in glasses which can be prepared with flawless surfaces and which, under carefully controlled environment conditions, have strengths approaching the theoretical limits.

At the present state of the art, no such surface can be obtained on polycrystalline ceramics. It is a moot argument whether the as-fired surface has or does not have Griffith-type flaws. From a practical point of view, the fact is that the geometry of a sintered ceramic piece is such that it is not directly usable whenever even a minimum of dimensional accuracy is required. Because of the hardness of ceramics, the only practical and most expedient way of machining is grinding with bonded diamond. This operation then determines not only the geometry of the finished ceramic piece, but also the quality of its surface which in turn affects the strength. Assuming that in ceramics the surface conditions have the same bearing on tensile strength as they do in glasses, it is surprising that the relationship between grinding and strength of ceramics has not been investigated earlier.

The desirability of such study has been recognized and a program was initiated at the Stanford Research Institute by the Naval Air Systems Command under Contract NOw-66-0383-d, entitled "Processing of Ceramics--Surface Finishing Studies." In the first phase of this program,[1] an experimental grinding facility was established and project personnel

developed the skills required for dependable operation. Performance of the equipment was evaluated and several special fixtures were designed and built.

In the second phase of this program[2] under Contract N00019-67-C-0494, the tensile strength of alumina was evaluated as a function of the grinding process. The grinding variables whose effect on strength were studied included the rate of material removal, type of diamond used, the grit size of the diamond, type of matrix, length of spark-out, and wet versus dry grinding. Ground surfaces were studied by means of light microscopy, transmission- and scanning-electron microscopy, and profilometry.

In the third phase of this program[3] under Contract N00019-68-C-0388, the tensile strengths of four different aluminas were evaluated as functions of their microstructure, surface finish, and various postgrinding treatments.

In the fourth phase of this program[4] under Contract N00019-69-C-0229, a comparison of strengths was made between specimens having as-fired surfaces and those which were ground by conventional means. By measuring the strengths of ground specimens in air and under vacuum, the detrimental effect of atmospheric humidity on strength was demonstrated. Also, evidence was presented that microstructure strongly controls strength as well as surface finish attainable by grinding.

In the final phase of this program under Contract N00019-70-C-0179, a detailed study was made of the relationship between microstructure, strength, environmental factors, and surface finish. The material used in this study was a high purity alumina prepared in five lots, each having a specified grain size. In addition, the vacuum strengths of other aluminas used in previous phases were determined. Results obtained in all five phases of this program are discussed.

II SUMMARY

The tensile strength of a high purity dense alumina was determined in air and under vacuum using the SRI expanded ring test. The test materials were supplied by the manufacturer in five groups of rings having different nominal grain sizes, i.e., 10, 20, 30, 40, and 50 μm. The actual measured grain sizes varied considerably from the nominal values. The rings were ground to size by the technique developed earlier in this program. Two types of abrasive were used, i.e., 120-grit natural metal-bonded diamond and 100-grit synthetic resinoid bonded diamond. After grinding, the specimens were dye checked and, although no individual flaws were detected, all rings showed a considerable dye retention which has not been observed previously on any other test material used on this program. It was determined that the coloration is the result of extensive damage incurred in grinding.

The following tensile strength values were obtained on the high purity alumina specimens:

Nominal Grain Size (μm)	Average Strength (psi)
10	35,700
20	34,300
30	33,500
40	29,800
50	28,100

The above values comprise all strength measurements regardless of test conditions and diamond used in grinding, because statistical analyses show that there is no statistically significant difference

between the air and vacuum strengths, no between the strengths of rings ground with different types of diamond. The only difference found on the 0.01 significance level is between groups of specimens having grain sizes equal to or smaller than 30 μm and those having grain sizes equal to or greater than 40 μm.

The air and vacuum strengths of three other alumina bodies were compared and in each case the vacuum strength was shown to be considerably higher (up to 37%) than the air strength.

A study based on Griffith's theory of brittle fracture was made in which experimental strengths were compared with those calculated from Griffith's relationship. Good agreement between experimental values and those predicted on the basis of grain size was obtained for all aluminas with the exception of the high purity body. In the latter case, Griffith's relationship does not hold for those materials whose grain size is smaller than the depth of the damaged region.

Profilometric and microscopic evaluations of the ground and fracture surfaces were made. Both methods are found to be inadequate for the interpretation of ceramic surfaces.

III EXPERIMENTAL STUDIES

A. Materials

The principal material used in this study was a dense, high purity alumina (Lucalox) supplied by the Lamp Glass Department of the General Electric Company, Richmond Heights, Ohio. The material was prepared in the form of thick-walled rings. All rings originated in the same batch of raw material. They were divided into five groups, each having twenty pieces, and each group was sintered so as to obtain a nominal average grain size, i.e., 10, 20, 30, 40, and 50 μm. The rings having the 10- and 20- μm grain size were opaque, whereas the coarser grained materials were translucent. Upon receipt, all rings were dye-checked for flaws. Several rings of the 10-μm group showed deep flaws on the inside walls and on the adjacent parts of the end faces. No flaws were detected in the other rings. In addition to this material, a small lot of blanks of Al-600 alumina and a group of Al-300 specimens were used. The latter specimens were ground previously under Contract N00019-68-C-0388.[3] Both the Al-600 and the Al-300 aluminas are products of the Western Gold and Platinum Company, Belmont, California.

Segments of broken rings were polished and the average grain size was determined by the method of Hilliard.[5] In the high purity alumina, the following values were obtained: 11.0, 15.4, 25.4, 39.3, and 42.2 μm. The discrepancy between nominal and measured grain sizes is considerable. At first glance, one may doubt that any real difference exists between the last two groups, except that in some of the samples of the 42.2 μm group secondary grain growth occurred and grains in excess of 200 μm were observed. In the other groups the grain size distribution was reasonably

uniform. The average grain size of Al-600 was 8.0 μm. Micrographs of the polished sections, taken under oblique light, are shown in Figure 1.

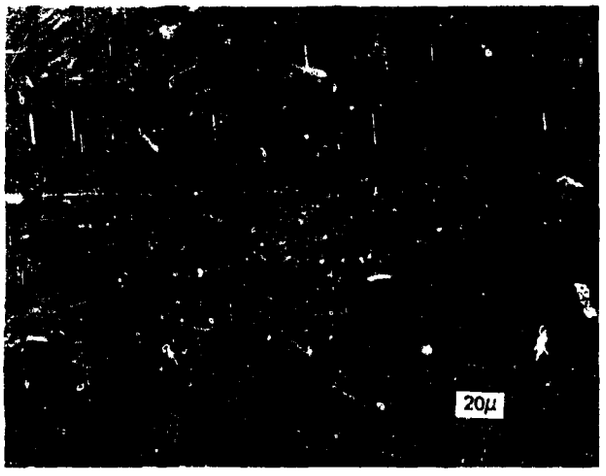
B. Grinding Procedure

Because of the limited number of available rings, it was essential to minimize breakage. Therefore the grinding technique developed in last year's work[4] was used. The rings were ground individually using existing mounting fixtures. First they were faced, the ID of all rings was then ground to final dimensions, and finally the OD was finished to size with the 320-grit diamond wheel (D320-N 100M 1/16) using the following settings:

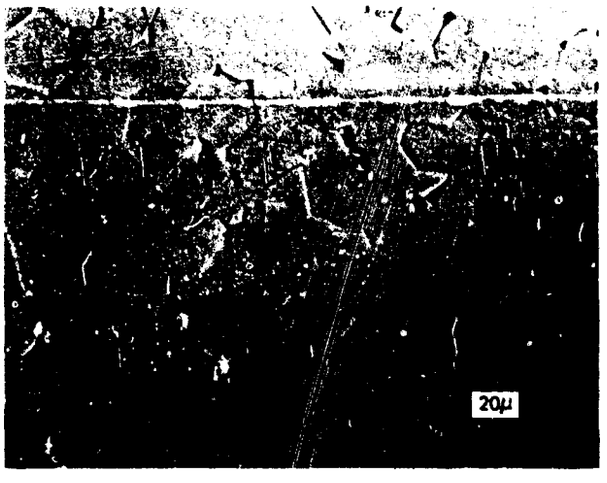
Surface speed	- 5700 sfpm
Continuous infeed	- 0.001 in./sec
Table travel	- 1 ft/min
Spark-out	- 1-1/2 min

Most of the ID grinding was done with the 120-grit wheel (D120-N-100M 1/16) under the above conditions. Five blanks of each group of high purity alumina rings were ground on the ID with the 100-grit synthetic diamond wheel (D100-R 50B76 1/16) under the same conditions. Ten out of the available eighteen Al-600 blanks were ground with the 120-grit wheel and the remaining eight were finish-ground (the last ten mils of the ID) with the 600-grit wheel (D600-N 75M 1/16) using a reduced infeed (0.0002 in./min).

The high purity alumina blanks were quite oversized so that approximately 80 to 100 mils had to be machined off both diameters and end faces The Al-600 blanks were undersized and their OD barely cleaned up. The Al-300 specimens were machined in a previous phase of the program[3] and were finish-ground with the 1200-grit diamond wheel (D1200-N 50M 1/16).



10 μm GRAIN SIZE



20 μm GRAIN SIZE

30 μm GRAIN SIZE

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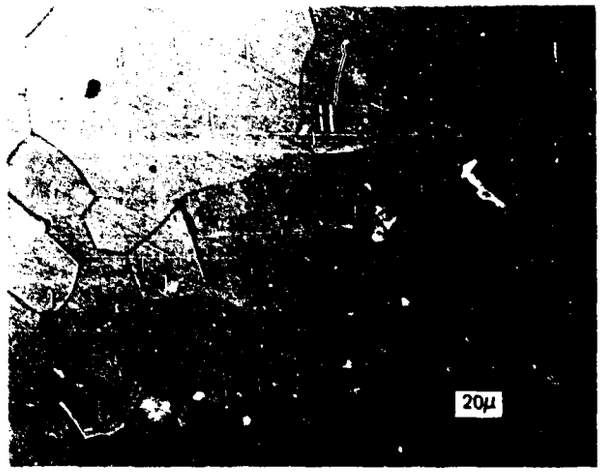
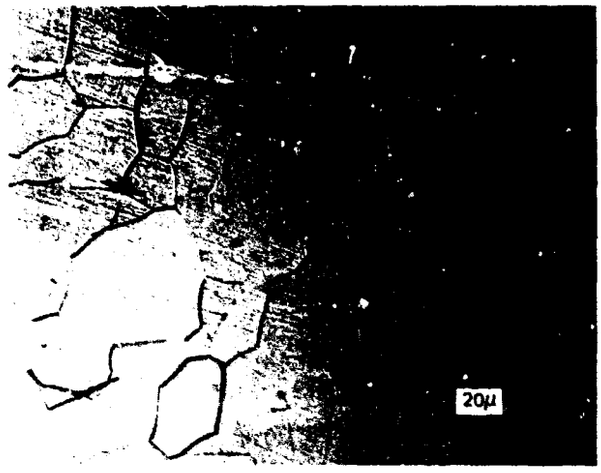
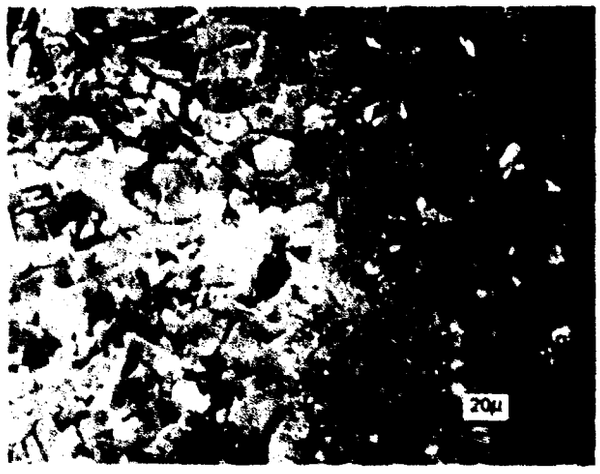
FIGURE 1 MICROSTRUCTURE OF TEST MATERIALS



40 μm GRAIN SIZE



50 μm GRAIN SIZE



Al-600

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FIGURE 1 MICROSTRUCTURE OF TEST MATERIALS (Concluded)

The final dimensions of all rings were:

ID - 2.000 ± 0.0005 in.

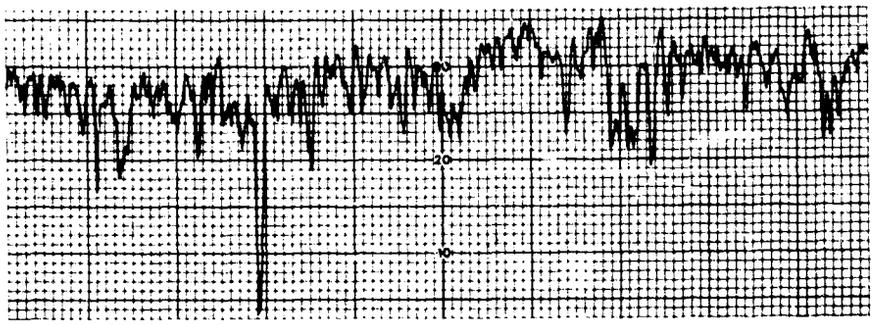
OD - 2.200 ± 0.0005 in.

Height - 0.300 ± 0.0005 in.

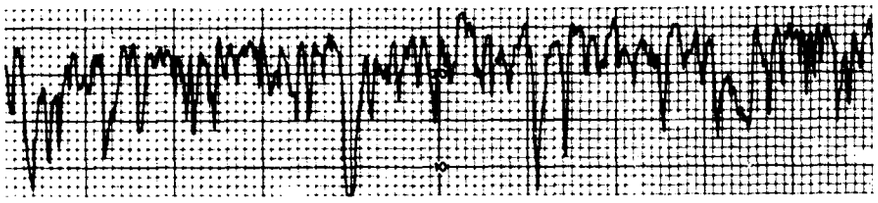
After grinding, the rings were degreased in trichloroethylene vapor washed with detergent, rinsed with hot water, and dried in an oven at 100°C. After cooling, the rings were immersed in a penetrating dye, rinsed in warm water, and examined for flaws. None were found, even the deep flaws on the inner walls of the 10-μm blanks were ground off. However, all of the high purity alumina rings retained a light pink color even after prolonged rinsing. The shade of pink varied from one ring to the next, and also between the groups having different grain sizes. No residual color was detected in the Al-600 rings, nor was any observed in the earlier phases of this program when Al-995 and Al-300 aluminas were used.

C. Surface Texture Evaluation

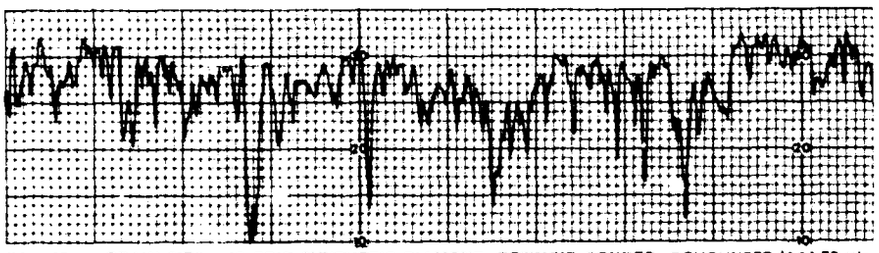
The texture of the ground surfaces was evaluated by means of profilometry and transmission- and scanning-electron microscopy. The profilometer used in this study was a Clevite/Brush 150 Surfanalyzer System. All measurements were made with a roughness-width cutoff of 0.030 in. and a stylus speed of 0.01 in/sec. Roughness values are expressed as arithmetical averages (AA) as defined by Standard B 46.1-1962 of the American Standards Association. Figure 2 shows profile traces obtained on the high purity body ground on the ID with two different wheels. On these graphs each horizontal division represents 1000 μin. and each vertical division represents 20 μin.



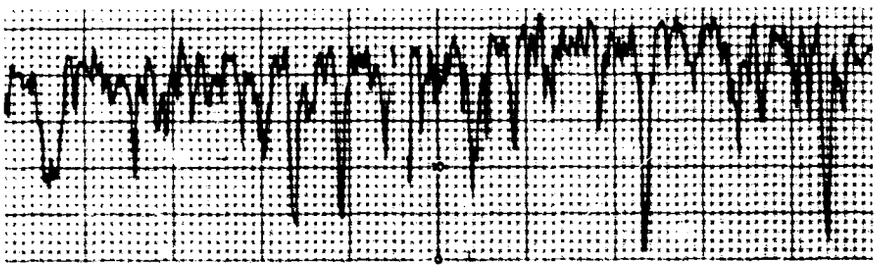
(f) 30 μm GRAIN SIZE, 100 GRIT SYNTHETIC DIAMOND, RESINOID BONDED ROUGHNESS (AA) 48 μin.



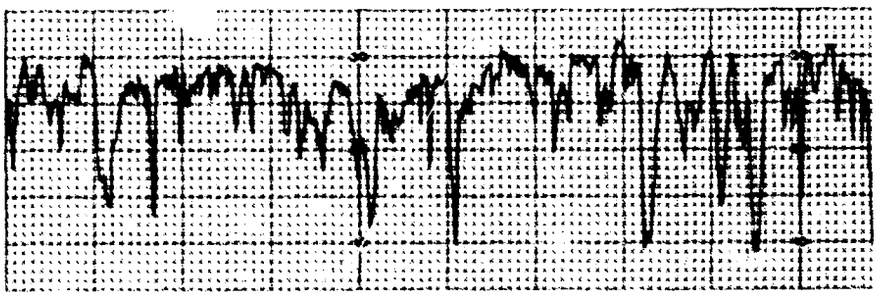
(g) 40 μm GRAIN SIZE, 120 GRIT NATURAL DIAMOND, METAL BONDED ROUGHNESS (AA) 50 μin.



(h) 40 μm GRAIN SIZE, 100 GRIT SYNTHETIC DIAMOND, RESINOID BONDED ROUGHNESS (AA) 52 μin.



(i) 80 μm GRAIN SIZE, 120 GRIT NATURAL DIAMOND, METAL BONDED ROUGHNESS (AA) 84 μin



(j) 80 μm GRAIN SIZE, 100 GRIT SYNTHETIC DIAMOND, RESINOID BONDED ROUGHNESS (AA) 88 μin.

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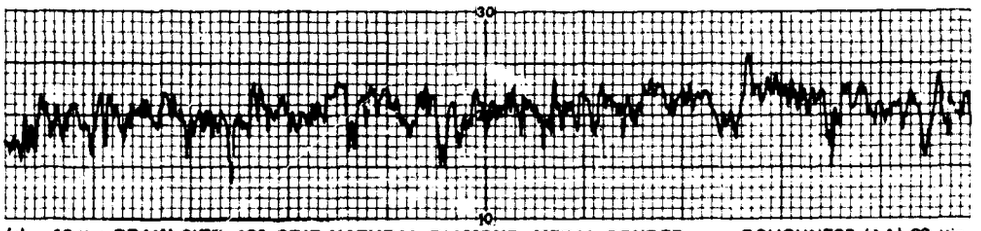
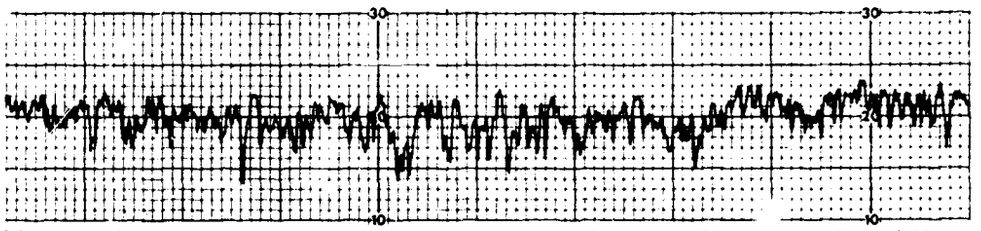
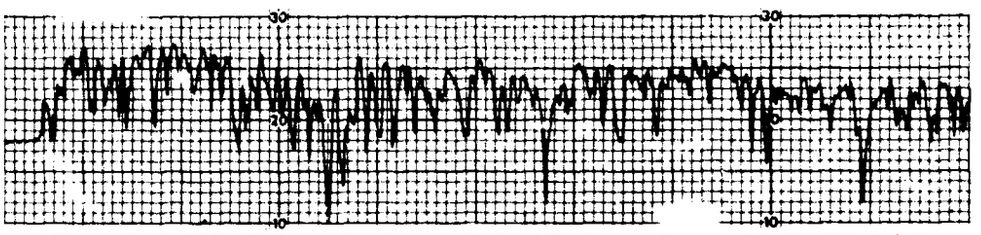
FIGURE 2 PROFILE TRACES OF HIGH PURITY ALUMINA HAVING DIFFERENT GRAIN SIZES AND GRINDING HISTORY



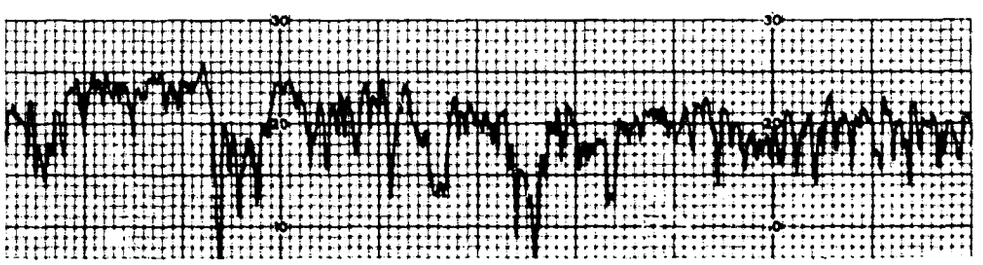
(a) 10 μm GRAIN SIZE, 120 GRIT NATURAL DIAMOND, METAL BONDED ROUGHNESS (AA) 28 μin.



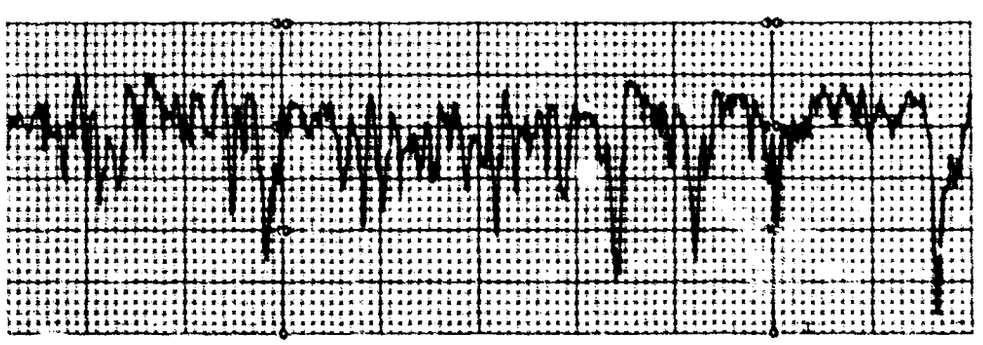
(b) 10 μm GRAIN SIZE, 100 GRIT SYNTHETIC DIAMOND, RESINOID BONDED ROUGHNESS (AA) 26 μin.



(c) 20 μm GRAIN SIZE, 120 GRIT NATURAL DIAMOND, METAL BONDED ROUGHNESS (AA) 36 μin.



(d) 20 μm GRAIN SIZE, 100 GRIT SYNTHETIC DIAMOND, RESINOID BONDED ROUGHNESS (AA) 40 μin.



(e) 0 μm GRAIN SIZE, 120 GRIT NATURAL DIAMOND, METAL BONDED ROUGHNESS (AA) 52 μin.

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FIGURE 2 PROFILE TRACES OF HIGH PURITY ALUMINA HAVING DIFFERENT GRAIN SIZES AND GRINDING HISTORY (Concluded)

The traces in Figure 3 were obtained on the outer walls of rings having an average grain size of 10 and 50 μm. These surfaces were ground with a metal-bonded wheel having 100-grit diamond. The OD of these rings was later finished with the 320-grit wheel. In Figure 3 each horizontal division is 500 μin. and each vertical division represents 20 μin.

In Figure 4 the profile traces of Al-600 alumina are shown.

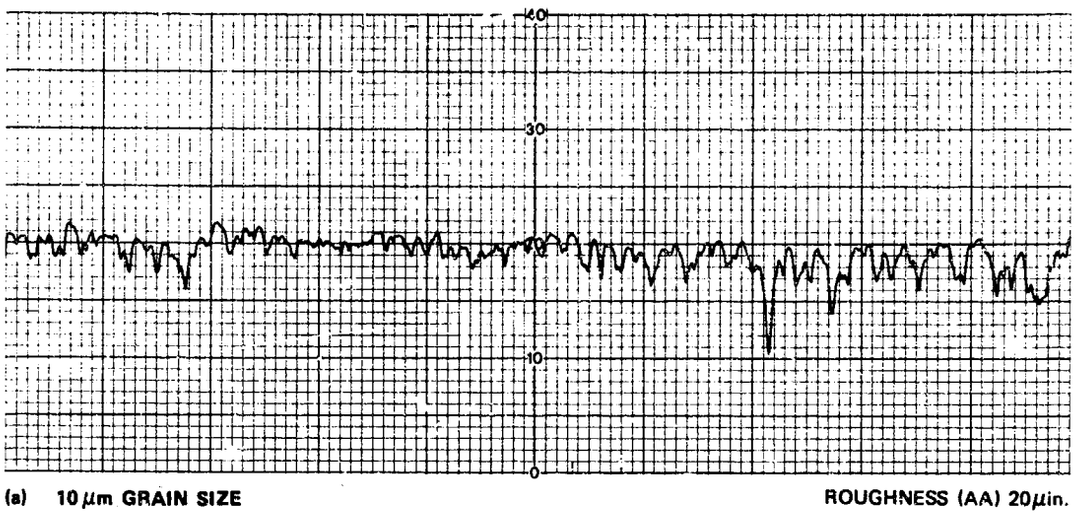
For ease of comparison, the roughness values from Figures 2 and 3 are summarized in Table 1. Included are values obtained on the OD ground with the 320-grit wheel although the corresponding profile traces are not shown.

Table 1

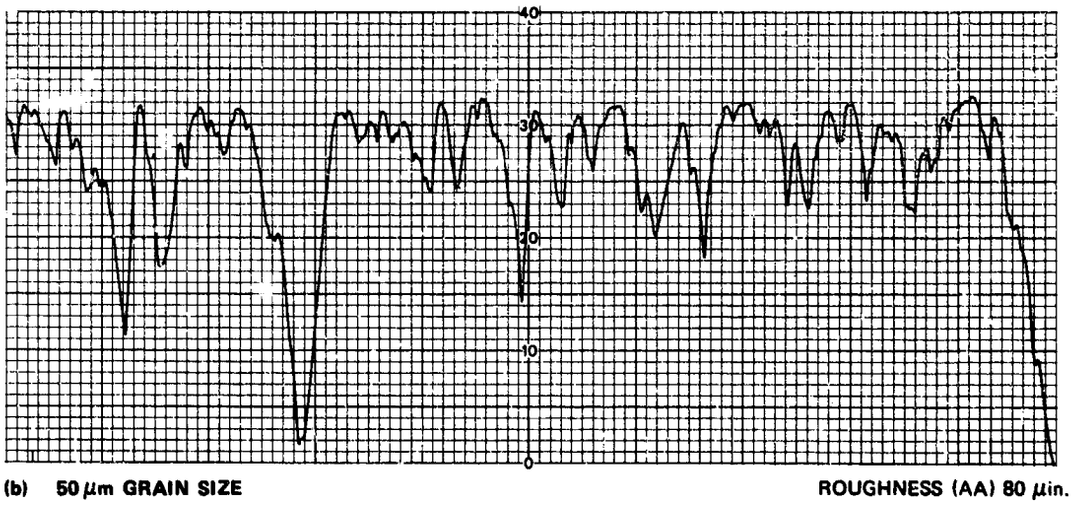
RELATIONSHIP BETWEEN GRAIN SIZE, DIAMOND GRIT, AND SURFACE ROUGHNESS (AA)

Grain Size (μm)	Grinding Conditions			
	IDS	IDN	ODF	ODC
	AA Surface Roughness (μin.)			
10	25	26	20	20
20	42	39	36	
30	42	50	29	
40	47	52	33	
50	65	62	42	80

IDS - Inside diameter 100-grit synthetic diamond.
IDN - Inside diameter 120-grit natural diamond.
ODF - Outside diameter 320-grit natural diamond.
ODC - Outside diameter 100-grit natural diamond.



(a) 10 μm GRAIN SIZE ROUGHNESS (AA) 20 μin.



(b) 50 μm GRAIN SIZE ROUGHNESS (AA) 80 μin.

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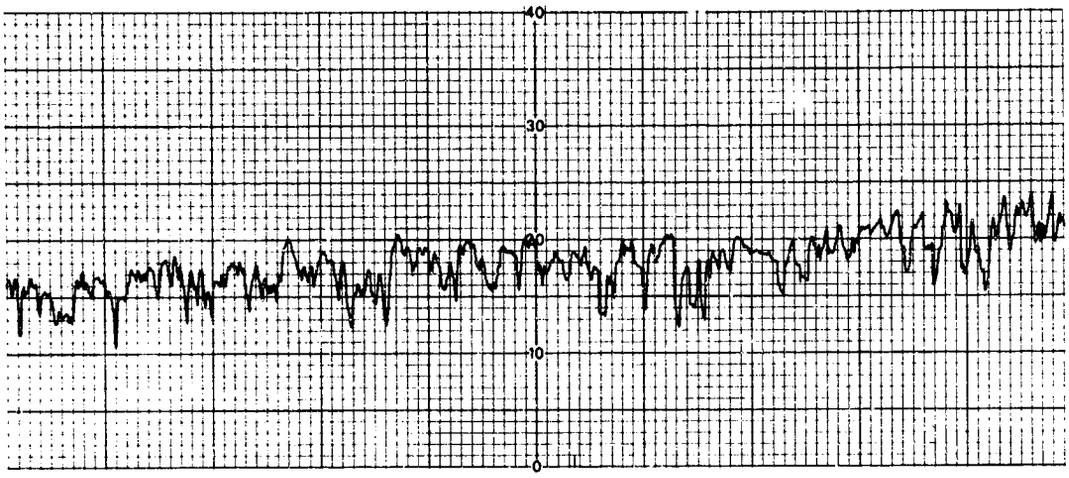
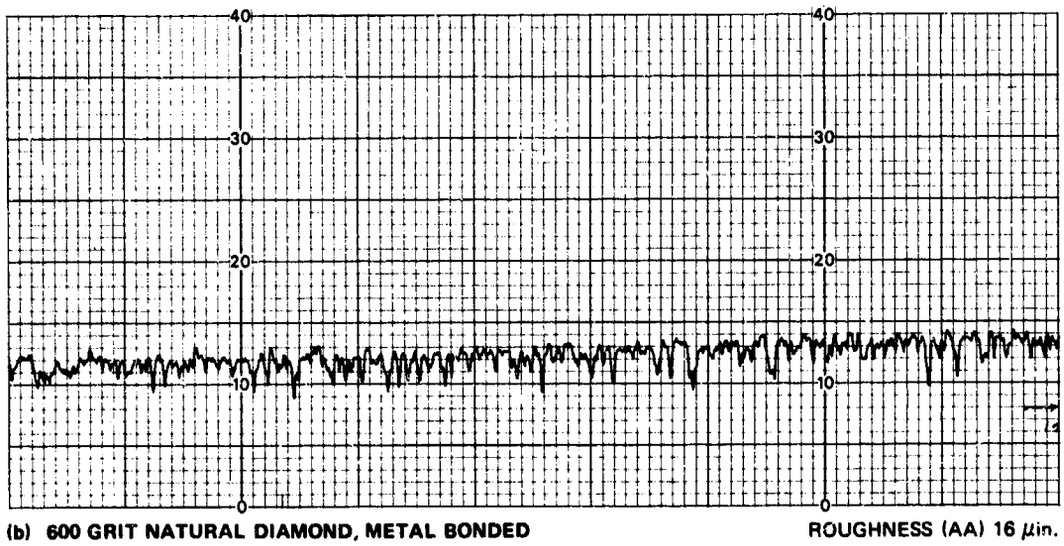
FIGURE 3 PROFILE TRACES OF HIGH PURITY ALUMINA GROUND WITH 100 GRIT METAL BONDED DIAMOND



(a) 120 GRIT NATURAL DIAMOND, METAL BONDED — ROUGHNESS (AA) 28 μin.



(b) 600 GRIT NATURAL DIAMOND, METAL BONDED — ROUGHNESS (AA) 16 μin.

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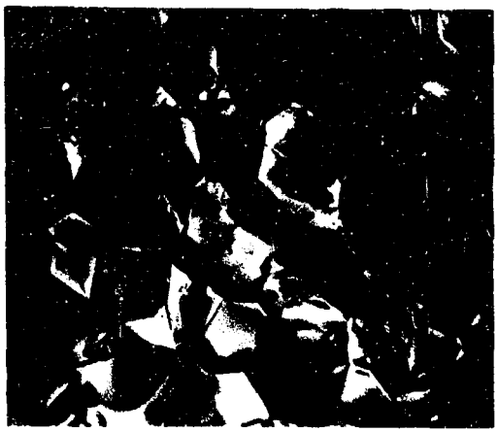
FIGURE 4 PROFILE TRACES OF Al-600 ALUMINA

It can be seen from Table 1 that the relationship between grain size and surface finish is not very systematic. The 10-μm grain size material has the smoothest finish and the 50-μm grain size material is the roughest. However, the surface finish of all materials having intermediate grain sizes is practically the same. Also, the roughness values (AA) obtained with the 320-grit wheel on the OD are always lower than those obtained on the ID with either wheel.

In Figure 3, the dissimilarity between profile traces obtained on the OD of the 10-μm and the 50-μm grain size materials ground with the 100-grit wheel is very obvious. However, there is another important difference between these two materials. In the 10-μm material, the OD finishes obtained with the two different wheels are identical (20 μin.). In the 50-μm material, the use of the coarser wheel caused almost a two-fold increase in roughness (42 vs. 80 μin.). It appears that, in some materials, the attainable surface finish is highly dependent on grinding conditions, whereas in other materials the surface finish is not affected by the grinding treatment within reasonable limits. The latter phenomenon has been observed in an earlier study[2] in which the use of a broad variety of grinding conditions failed to produce a significant change in strength. In Figure 4 the surface finish of A1-600 alumina is shown. It is seen that the 600-grit diamond produced the finest finish of all. However, the roughness value obtained with the 120 grit (28 μin.) wheel is higher than that obtained previously[3] (16-20 μin.) with the same wheel on another lot of A1-600.

An extensive study of the ground high purity alumina surfaces was made by means of transmission- and scanning-electron microscopy. In both instances the results are the same (Figures 5 and 6). Regardless of grain size, the topography of the surfaces suggests that they are primarily the



10 μm GRAIN SIZE

20 μm GRAIN SIZE

30 μm GRAIN SIZE

40 μm GRAIN SIZE

50 μm GRAIN SIZE

FIGURE 5 TRANSMISSION ELECTRON MICROGRAPHS OF GROUND HIGH PURITY ALUMINA SURFACES (2000X)

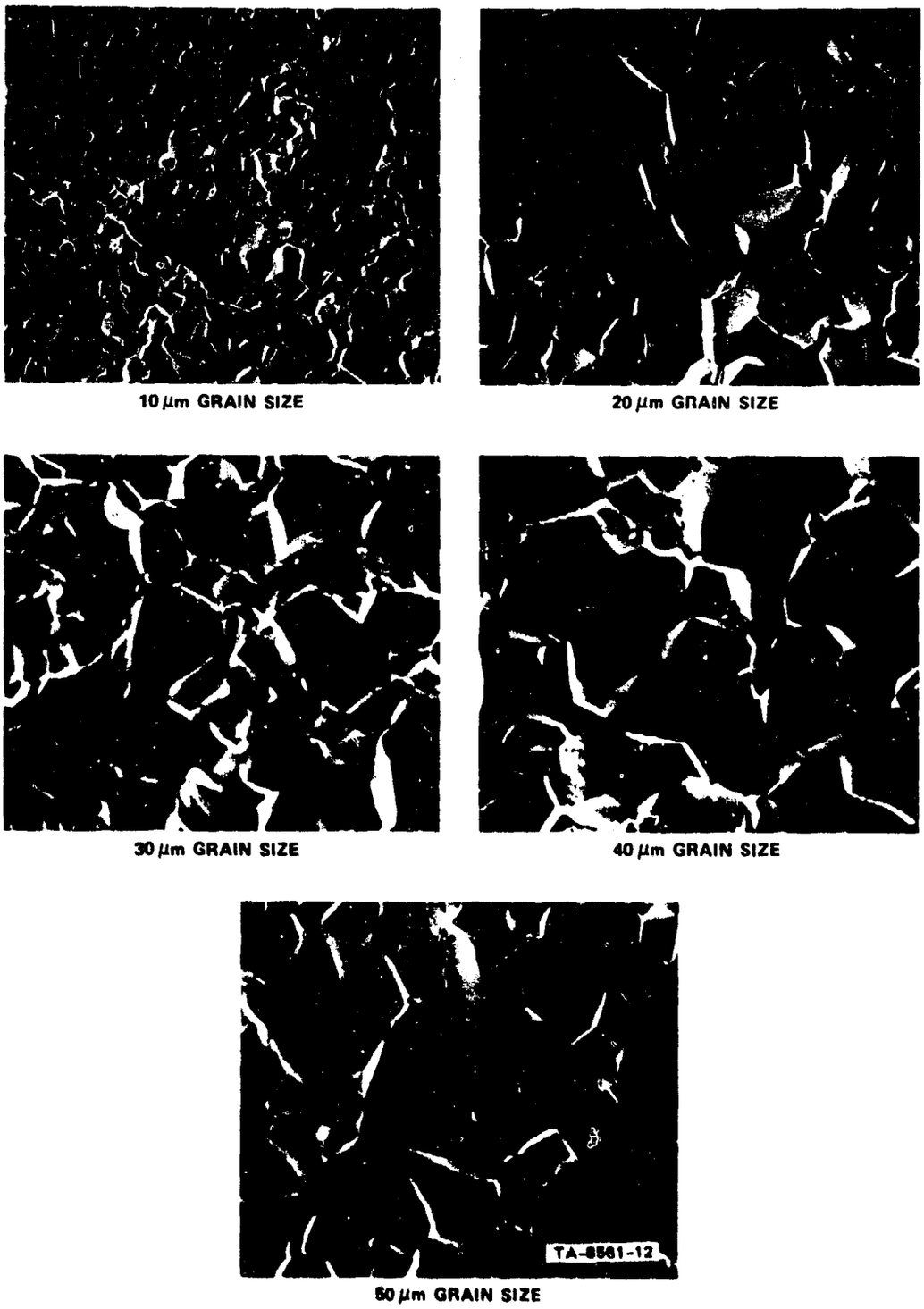


FIGURE 6 SCANNING ELECTRON MICROGRAPHS OF HIGH PURITY ALUMINA FRACTURE SURFACES (560X)

result of intergranular fracture. Moreover, fracture surfaces of the broken rings show hardly a trace of transgranular fracture. This finding is in disagreement with those of other workers[6] who found in this material as much as 31% of transgranular fracture.

In an attempt to get a better understanding of the reasons for the appearance of the ground surfaces, a study of the swarf was undertaken. One ring of each grain size of the high purity alumina was ground using tap water, which was not recirculated, as coolant. The swarf was collected and examined under the microscope. It was found that the largest particle measured about 7 μm across. Obviously the swarf does not contain entire grains. A possible explanation for the smaller particle size of the swarf is that after a grain is removed from the surface, it is subsequently crushed as it passes between the ceramic ring and the grinding wheel. Whether or not this helps the grinding process was not determined.

Another facet of the study of the high purity alumina was aimed at clarifying the reason why ground surfaces retained some of the penetrating dye even after prolonged rinsing. This study was feasible only on fracture surfaces of the more opaque materials (10-μm and 20-μm grain size). The others, being translucent, did not provide sufficient contrast to permit measurement of the extent of the damaged region. On the fracture surfaces of the fine grain materials, dye was found to penetrate beneath all surfaces of the rings and to extend inward to an average depth of 27 μm. Ground surfaces of this alumina thus show evidence of damage due to the grinding process. A typical example of dye penetration is shown in Figure 7.

An interesting observation can be made by comparing the measured depth of dye penetration with the profile traces of Figure 2. The depth of 27 μm (1080 μin.) corresponds to 54 vertical divisions on the graph. No such spread between peak and valley can be found in the profile traces.

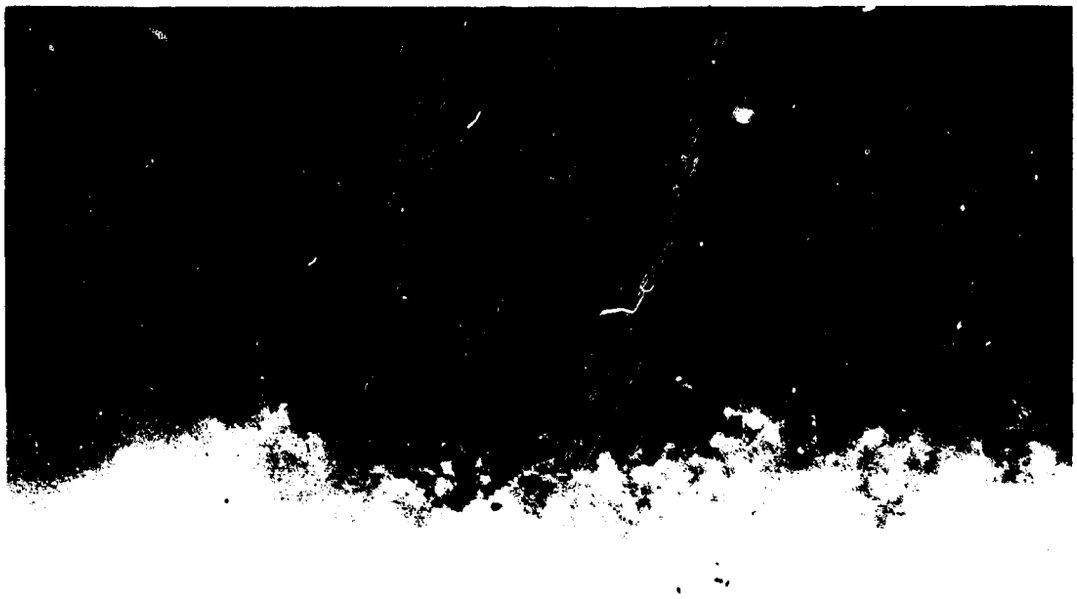


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FIGURE 7 SURFACE DAMAGE IN HIGH PURITY ALUMINA (300X)

Therefore, it is apparent that profilometry is an inadequate tool for interpreting the true conditions of a ceramic surface.

D. Testing Procedure

Throughout the program, the tensile strength determinations were made by the SRI expanded ring test. The description of the test method can be found in the open literature[7] and will not be repeated here.

All strength measurements were obtained at a stress rate of 3000 psi/sec. The vacuum strength determinations were made at pressures between 5×10^{-7} to 1×10^{-6} torr. The testing facility is shown in Figure 8.

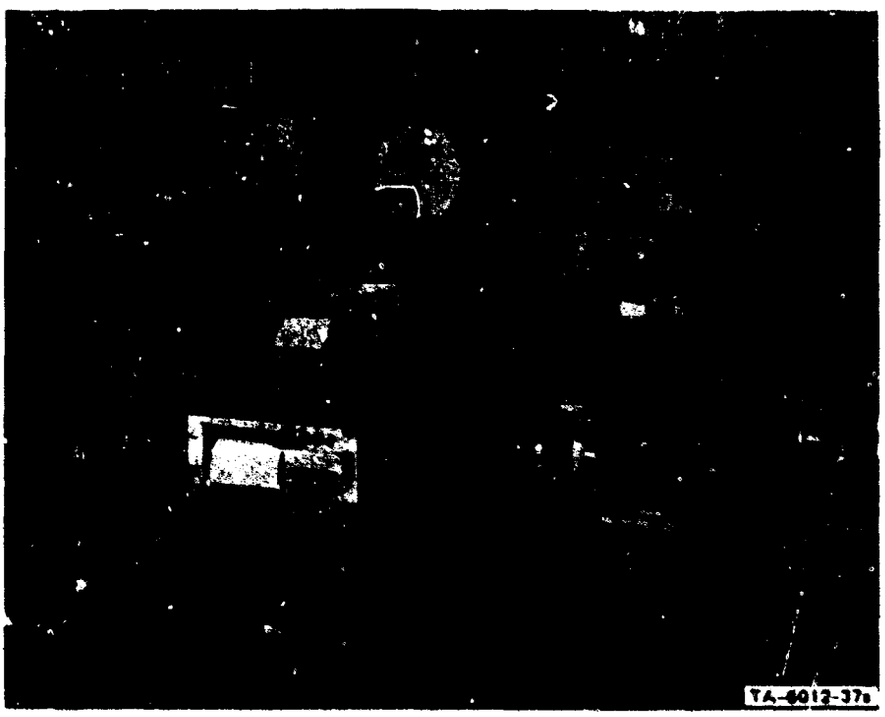

FIGURE 8 MECHANICAL PROPERTIES TESTING FACILITY

IV RESULTS AND DISCUSSION

A. Experimental

The objective of this study was to evaluate the relationship between strength and microstructure as typified by grain size, surface finish, and environmental conditions. To eliminate the uncertainties related to the effect of chemical compositions on strength, a high purity alumina was chosen as the main test material because it can be prepared in a wide range of grain sizes, each of which can be closely controlled. The Al-600 and Al-300 aluminas were used to a limited extent mainly to complete the already existing knowledge of their behavior under tensile stress. All test data were subjected to various statistical analyses in order to assign proper weights to various trends indicated by empirical results. In the case of the high purity alumina, the variable which was expected to have the greatest influence on strength was the grain size. Thus, the tensile strength values are presented as a function of grain size only, regardless of environmental conditions and grinding history. This information is summarized in Table 2. Individual strength values are found in Appendix A.

The data in Table 2 can be subdivided so as to show the effect on strength of the other variables studied, namely test conditions (vacuum versus air) and the difference between grinding wheels (synthetic versus natural diamond). In Table 3 under the heading "Conditions" the following symbols are used: A - air, V - vacuum, S - synthetic diamond, and N - natural diamond.

Data in Tables 2 and 3 show that under all conditions (grinding and environment) the strength of this material decreases with increasing

Table 2

TENSILE STRENGTH OF A HIGH PURITY ALUMINA
AS A FUNCTION OF GRAIN SIZE

Nominal Grain Size (μm)	Actual Grain Size (μm)	Tensile Strength (psi)	Std Dev (± psi)	Coeff of Var (%)
10	11.0	35,700	2,830	7.9
20	15.4	34,300	3,050	8.9
30	25.4	33,500	3,240	9.7
40	39.3	29,800	3,240	10.9
50	42.2	28,100	3,080	11.0

Table 3

TENSILE STRENGTH OF HIGH PURITY ALUMINA
AS A FUNCTION OF GRAIN SIZE, GRINDING TECHNIQUE,
AND TEST CONDITIONS

Grain Size (μm)	Conditions	Tensile Strength (psi)	Std Dev (± psi)	Coeff of Var (%)
10	AN	34,600	2,250	6.5
	AS	34,800	3,900	11.2
	VN	36,800	2,500	6.8
20	AN	32,400	2,100	6.5
	AS	35,200	1,590	4.5
	VN	34,900	3,820	11.0
30	AN	31,300	3,050	9.7
	AS	33,800	3,270	9.7
	VN	34,500	3,250	9.4
40	AN	29,800	3,560	11.9
	AS	28,300	3,230	11.4
	VN	30,600	2,990	9.8
50	AN	26,500	1,720	6.5
	AS	29,500	1,740	5.9
	VN	28,200	3,720	13.2

grain size. However, the data scatter is large, making it difficult to assess objectively the importance of the differences in strength. For this reason, several statistical analyses were applied to test data. The details of these analyses are given in Appendix B. The following are conclusions derived by the use of the nonparametric Kolmogorov-Smirnov[8] test:

(1) In high purity alumina ground with 120-grit natural diamond and tested in air (condition AN, Table 3), the difference in strength as a function of grain size is statistically significant only between groups having grain sizes equal to or smaller than 30 μm and those with grain sizes equal to or greater than 40 μm.

(2) In high purity alumina ground with 100-grit synthetic diamond and tested in air (condition AS), a difference in strength was found on the 0.01 significance level between groups having grain sizes smaller than or equal to 30 μm and those having grain sizes equal to or greater than 40 μm.

(3) For results obtained under vacuum (condition VN), the same conclusion holds, i.e., there is a difference in strength on the 0.01 significance level between groups having grain sizes smaller than or equal to 30 μm and greater than or equal to 40 μm.

(4) In comparing air and vacuum strengths, no statistically significant difference was found in any grain size.

The results in Table 4 clearly show that the air strength of Al-600, Al-995, and Al-300 aluminas are very much different from the vacuum strengths, and statistically the difference is significant on the 0.01 level. The data for Al-600 represent the combined strength values of specimens ground with two different wheels. However, the analysis shown in Appendix B proves that the grinding history did not significantly influence strength either in air or under vacuum. Previous work[2] also indicated that grinding has a relatively mild effect on strength.

Table 4

COMPARISON OF AIR AND VACUUM STRENGTHS
OF VARIOUS ALUMINAS

Type of Al_2O_3	Air Strength (psi)	Standard Deviation (± psi)	Vacuum Strength (psi)	Standard Deviation (± psi)	Increase in Strength (%)
Al-300	28,600*	1,740	32,100	2,480	12.2
Al-995	30,800†	1,000	35,800†	1,600	16.2
Al-600	33,900	2,460	46,400	3,290	36.9

* Data obtained of specimens previously[3] ground with 1200-grit diamond.
† Data obtained previously.[4]

An interesting relationship between air strength of the various test materials and the increase in strength under vacuum can be clearly seen in Table 4 where air strength and relative increase in strength under vacuum followed in the order Al-300, Al-995, and Al-600. This is also the order of decreasing grain size and improved surface finish.[3] However, the orderly sequence ends here, since we find no logical way of including in this picture the effect of chemical composition.

If we assume that the increase is strength under vacuum is caused by the absence of a stress-induced chemical reaction, we should also investigate the nature of the reactants. One is without doubt water from the air, and the other can be any of the oxide constituents of the ceramic body. Silica is the most likely candidate because the detrimental effect of water on the strength of glass is well known. Consequently, one might expect that a higher silica content will be accompanied by a greater difference between the air and vacuum strengths. However, experimental results do not bear out this assumption. The difference in strength is

the highest in Al-600 alumina containing 2.0% SiO_2 and the lowest in the high purity alumina whose silica content is practically nil. On the other hand, in Al-300 and Al-995 aluminas containing 1.25 and 0.40% SiO_2 respectively, the pattern is reversed.

The second highest impurity in conventional aluminas, and the only one present in any significant amount in the high purity alumina, is magnesia. The MgO concentration in the test materials used is 0.02% in Al-300, 0.50% in Al-995, and 0.75% in Al-600. The MgO content in the high purity material is approximately 0.1%. If magnesia is the constituent sensitive to stress corrosion, then there is a systematic, although not linear, relationship in conventional aluminas between the MgO content, grain size, and difference between air and vacuum strengths. The high purity material, having a higher MgO content than Al-300, has the same strength in air as under vacuum and does not follow the above pattern. This fact is surprising, although not unique. In unrelated works, the same phenomenon was observed in lead zirconate-titanate and in quartzite rock.

It appears that each material has pecularities, which are reflected in strength, response to environmental conditions, and in the grinding behavior.[9] The last factor is most pronounced in the high purity material. No other alumina body used in the entire program showed any evidence of damage induced by grinding. The tendency of the high purity alumina for pullouts, i.e., intergranular fracture, has been observed by other workers.[10] In comparison with lower purity aluminas, the former appears to be more brittle and to have a lower impact resistance. The reason is not clear, but it is evident that, in grinding, this material shatters along the grain boundaries. This damage then controls the strength, and its effect largely overrides that of grain size and stress corrosion.

B. Theoretical

Griffith's theory[11,12] for brittle fracture of solids is based on the assumption that flaws, i.e., small cracks, exist in the material and that these flaws are responsible for the existence of low strengths. Griffith analyzed the elastic stress distribution around an elliptically shaped crack in a uniform tensile stress field following the analysis of Inglis[13] and obtained the following expression

$$S = \left(\frac{2\gamma E}{\pi L}\right)^{1/2} \qquad (1)$$

where

S = the critical applied stress required for failure

γ = the fracture surface energy

E = Young's modulus

L = the half length of the elliptical shaped crack.

Orowan[14] postulated that the presence of a grain boundary could stop a cleavage crack from penetrating the next grain, and he used this concept to explain the strength versus grain size relationship of steels at low temperatures.

If it is assumed that grinding introduces in the alumina grains cleavage cracks that penetrate to the first grain boundary and that these cleavage cracks occur under all conditions of grinding, then the microstructure becomes the most important variable in determining the strength of a particular alumina body. To test this hypothesis, we calculated the tensile strength by Griffith's equation, using a value for Young's modulus of 55×10^6 psi. Wiederhorn[15] measured γ, the fracture surface energy of Al_2O_3 single crystals, using a double cantilever beam technique, and obtained values of 7300 erg/cm^2 and 6000 erg/cm^2 for $\{10\bar{1}0\}$ and $\{\bar{1}012\}$

type planes, respectively. Thus a value of 7000 erg/cm^2 was used for calculating the strength of ground polycrystalline aluminas. The crack size was evaluated by counting the number of grain boundaries intersected by random straight lines of known length, and the average linear intercept was converted to an average grain size by multiplying by a factor of 1.5.[16] The critical flaw which determines the strength should be equal to the maximum grain size. To obtain maximum grain sizes, we have assumed that normal grain growth has occurred, as defined by Hillert,[17] and that the maximum grain size is twice the average grain size. Thus the crack depth resulting from grinding is assumed to be two times the average grain size.

The calculated strengths are compared to the experimentally determined strengths in Table 5 for various alumina bodies. The average grain sizes measured on these materials are also listed. A common method of presenting this type of data is to plot the strength as a function of the reciprocal of the square root of the grain size. This plot is shown in Figure 9.

Table 5

TENSILE STRENGTH OF VARIOUS ALUMINAS

Material	Grain Size (μm)	Calculated Strength (psi)	Experimental Strength* (psi)
Al-300	32.0	26.0×10^3	27.8×10^3
Al-995	18.3	31.1×10^3	31.4×10^3
AD-995	12.6	37.5×10^3	35.3×10^3
Al-600	10.5	41.1×10^3	39.6×10^3

* All data were obtained previously[3] on materials ground with 120-grit diamond.

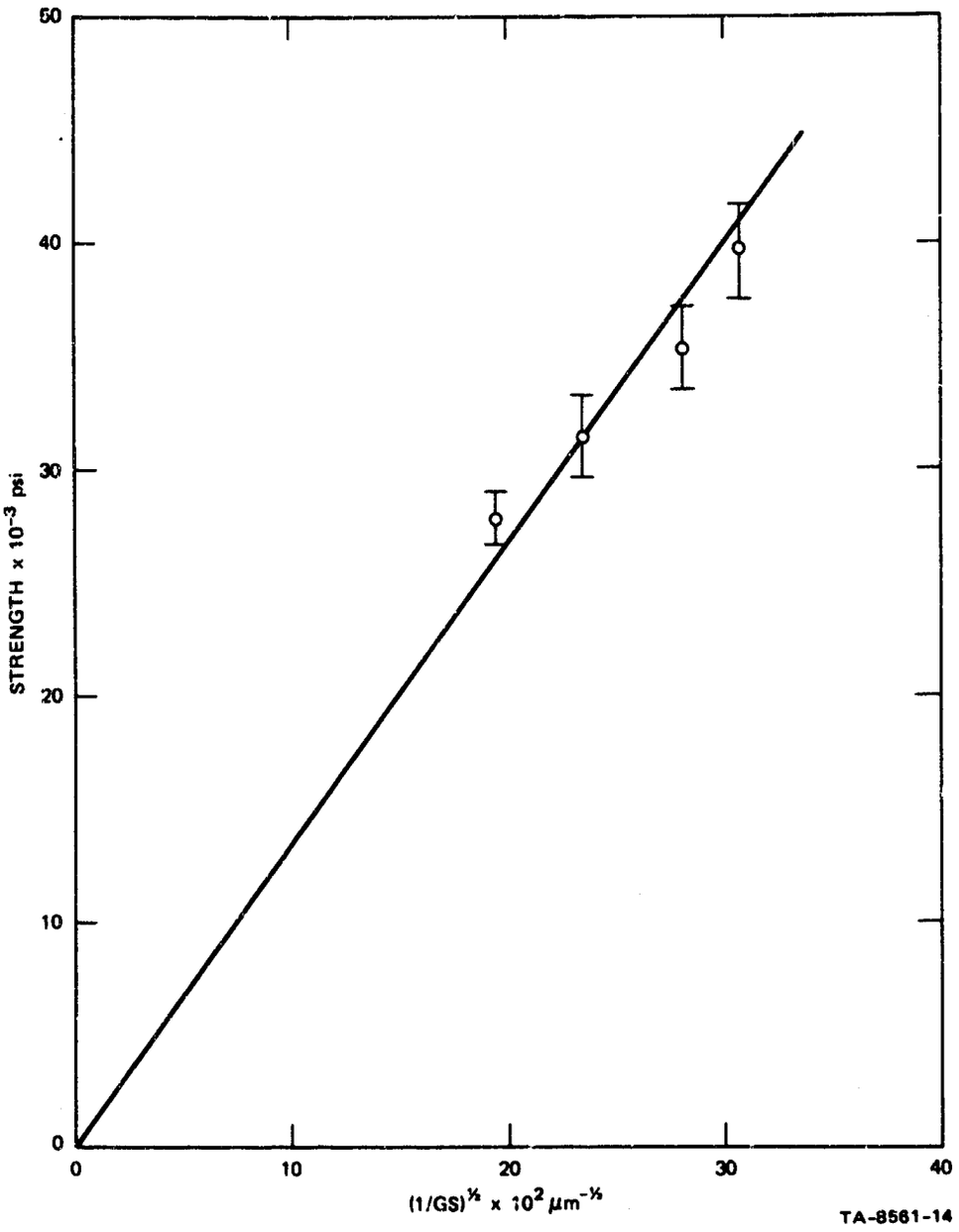

FIGURE 9 PLOT OF RELATIONSHIP BETWEEN STRENGTH AND GRAIN SIZE FOR DIFFERENT ALUMINAS

The agreement between the calculated and experimental values is extremely good and lends credibility to the concept that grinding of alumina causes cleavage cracks that terminate at the first grain boundary. Since the data in Table 5 were obtained on aluminas of varying purity, we attempted in this phase of the program a correlation based on Griffith's equation of calculated strength and the experimental strengths in a high purity alumina. Early in the program it was observed that this alumina exhibited predominant intergranular fracture while the aluminas listed in Table 5 exhibited predominant transgranular fracture. The change in fracture mode should result in a different fracture surface energy while not affecting Young's modulus. In support of the latter assumption, Young's modulus measured for the high purity alumina is 57×10^6 psi.[18] We have assumed that the grain boundary fracture surface energy of the high purity alumina is the same as the fracture surface energy for single crystal alumina modified by a geometrical factor of two to account for the tortuous nature of the crack path. The factor of two is based on a consideration of circular and spherical crosssection. Table 6 compares the calculated strengths with the measured strengths. The agreement between calculated and measured strengths for the first three grain sizes in Table 6 is exceedingly poor, but the calculated strengths of the last two grain sizes agree well with the measured strengths.

All of the alumina rings used in this study were dye checked using a red penetrant dye after completion of the grinding. The aluminas listed in Table 5 did not exhibit any dye retention, whereas the high purity alumina rings had a pink surface. Microscopic examination of the fracture surfaces revealed cracks with dye penetrating to an average depth of 27 μm irrespective of the grain size. This phenomenon was readily observed in the 11- and 15.3-μm grain size samples and was observed with difficulty in the 25.4-μm samples, but could not be observed in the large grain size samples. The decreased detectability of

Table 6

CALCULATED AND EXPERIMENTAL STRENTHS OF HIGH PURITY ALUMINA

Grain Size (μm)	Calculated Strength (psi)	Experimental Strength (psi)
11.0	56.8×10^3	35.7×10^3
15.4	48.0×10^3	34.3×10^3
25.4	37.3×10^3	33.5×10^3
39.3	30.1×10^3	29.8×10^3
42.2	29.0×10^3	28.1×10^3

the dye penetration was due to the increased transparency of the material with increasing grain size.

The largest crack or flaw in the surface of the ceramic rings will control the observed strength. Thus, if the above described grinding treatment produces flaws with an average depth of 27 μm, and if we assume a crack size distribution similar to the distribution of grain sizes, we then expect the mechanically produced flaws to control the tensile strength of high purity alumina specimens having grain sizes smaller than 27 μm. For grain sizes larger than 27 μm, we expect the grain size to control the tensile strength through the Griffith equation relationship. Figure 10 shows that the high purity alumina follows these concepts. The sloping line in Figure 10 was calculated from Griffith's equation, and the plateau was based on the observed mechanical damage in the samples. The agreement between the experimental points and the calculated lines is very good; furthermore, there is no statistical difference between the first three grain size samples listed in Table 6, and this finding supports the concept of a plateau in strength due to a uniform mechanical damage.

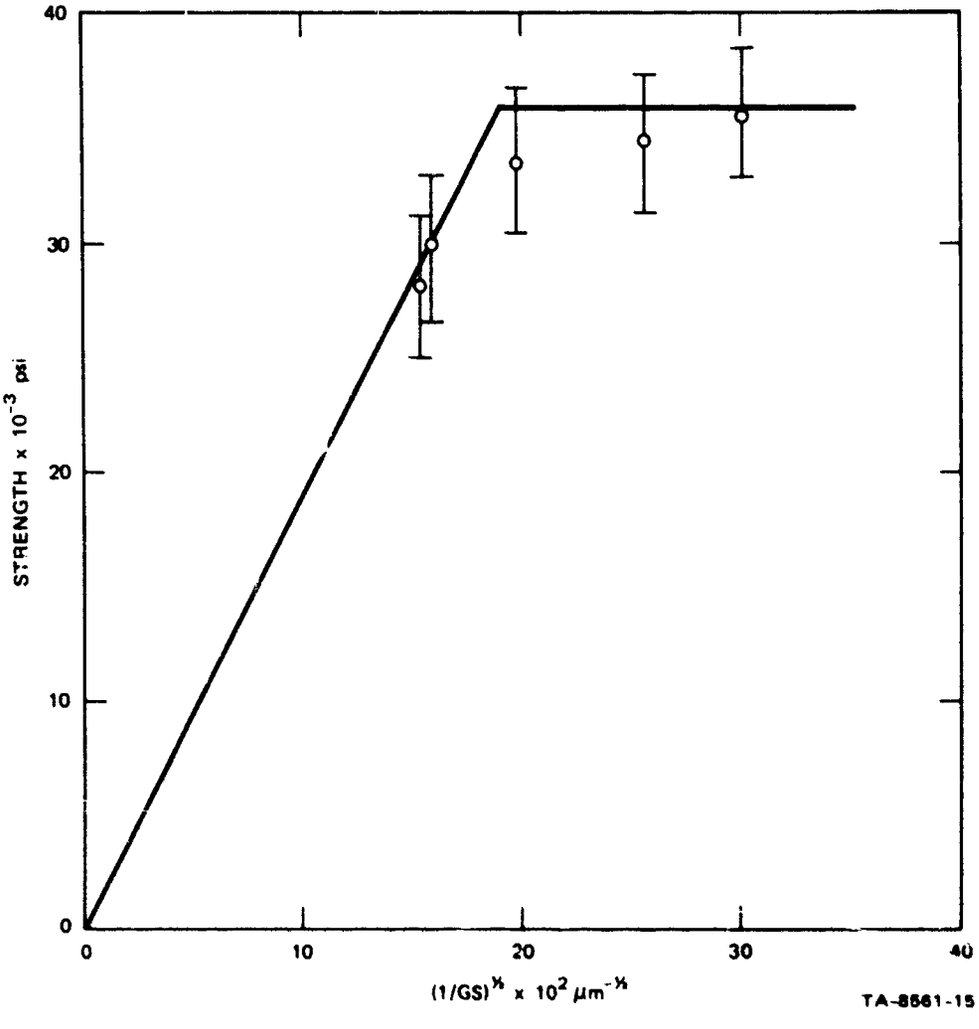

FIGURE 10 PLOT OF RELATIONSHIP BETWEEN STRENGTH AND GRAIN SIZE FOR HIGH PURITY ALUMINA

Thus the tensile strengths after grinding of the alumina materials studied, with the exception of the high purity alumina, are controlled by the microstructure, i.e., grain size. If mechanical damage extends beyond one grain diameter for small grain samples, this damage controls the tensile strength until the grain size becomes larger than the depth of the mechanical damage, after which the microstructure or grain size controls the tensile strength.

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Appendix A

INT)UAL STRENGTH VALUES FROM TABLES 1 TO 6

Table A-1

TENSILE STRENGTH OF HIGH PURITY ALUMINA
(Tested in Air, Ground with 120-Grit Natural Diamond)

Grain Size (μm)	Strength (psi)	Grain Size (μm)	Strength (psi)	Grain Size (μm)	Strength (psi)
10	34,800	20	34,500	30	30,400
	33,000		34,300		32,100
	36,200		30,100		26,600
	37,300		32,600		34,800
	31,800		30,300		32 500
Average	34,600		32,400		31,300
Std Dev ±	2,250		2,100		3,050
40	36,000	50	27,800		
	27,500		27,500		
	28,700		27,300		
	27,400		23,600		
	29,500		26,100		
Average	29,800		26,500		
Std Dev ±	3,560		1,720		

Table A-2

TENSILE STRENGTH OF HIGH PURIETY ALUMINA
(Tested in Air, Ground with 100-Grit Synthetic Diamond)

Grain Size (μm)	Strength (psi)	Grain Size (μm)	Strength (psi)	Grain Size (μm)	Strength (psi)
10	35,400	20	37,200	30	36,600
	37,000		34,300		34,800
	28,100		33,600		28,500
	38,000		35,700		36,100
	35,600				33,000
Average	34,800		35,200		33,800
Std Dev ±	3,900		1,590		3,270
40	27,800	50	28,400		
	26,100		28,400		
	24,600		28,500		
	32,400		30,000		
	30,800		32,400		
Average	28,300		29,500		
Std Dev ±	3,230		1,740		

Table A-3

TENSILE STRENGTH OF HIGH PURITY ALUMINA
(Tested Under Vacuum, Ground with 120-Grit Natural Diamond)

Grain Size (μm)	Strength (psi)	Grain Size (μm)	Strength (psi)	Grain Size (μm)	Strength (psi)
10	33,600	20	37,300	30	34,600
	40,500		38,500		35,800
	34,700		37,200		27,300
	35,700		27,300		35,200
	33,800		31,900		36,400
	37,300		36,800		37,300
	38,500		38,300		38,100
	39,600		31,600		32,200
	37,600		35,200		33,800
Average	36,800		34,900		34,500
Std Dev ±	2,500		3,820		3,250
40	34,300	50	27,200		
	30,600		33,000		
	29,400		20,000		
	24,600		31,600		
	28,500		26,300		
	31,800		29,600		
	30,000		29,700		
	33,700		28,000		
	32,700		28,400		
Average	30,600		28,200		
Std Dev ±	2,990		3,720		

Table A-4

COMPARISON OF AIR AND VACUUM STRENGTHS OF A1-300 ALUMINA

Air Strength* (psi)	Vacuum Strength (psi)
29,400	29,600
27,000	28,600
27,800	34,600
29,800	34,500
28,400	29,500
28,400	29,300
30,200	33,100
25,800	33,500
28,800	32,800
25,600	28,100
31,000	36,400
27,000	32,800
31,600	33,200
29,000	33,200
Average 28,600	Average 32,100
Std Dev ± 1,140	Std Dev ± 2,480

* Data generated under Contract No. N00019-68-0388.

Table A-5

COMPARISON OF AIR AND VACUUM STRENGTHS OF Al-995 ALUMINA

Air Strength* (psi)	Vacuum Strength* (psi)
31,500	35,400
31,000	34,000
32,200	37,700
30,800	37,700
29,200	34,400
30,800	
31,100	Average 35,800
30,200	Std Dev ± 1,600
31,100	
31,300	
30,100	
29,200	
32,200	
29,200	
32,000	
Average 30,800	
Std Dev ± 1,000	

* Data generated under Contract No. N00019-69-C-0229.

Table A-6

COMPARISON OF AIR AND VACUUM STRENGTHS OF Al-600 ALUMINA

Air Strength (psi) (120-grit diamond)		Vacuum Strength (psi) (120-grit diamond)	
	31,700		42,400
	32,700		43,000
	37,200		46,500
	31,200		47,200
	34,100		49,500
Average	33,400	Average	45,700
Std Dev ±	2,200	Std Dev ±	2,490
(600-grit diamond)		(600-grit diamond)	
	31,600		48,200
	31,600		48,800
	33,900		40,800
	37,900		50,800
Average	34,500	Average	47,200
Std Dev ±	2,250	Std Dev ±	3,790

Appendix B

STATISTICAL EVALUATION OF TEST DATA

Appendix B

STATISTICAL EVALUATION OF TEST DATA

Several statistical analyses were applied to test data, such as Student's t-test and F-test, but the results were not fully trusted, although they appeared logical. For these tests, the sample distribution must be known, and this is a time-consuming process particularly if the sample size is small and the scatter large. Therefore, the non-parametric test of Kolmogorov-Smirnov[8], was used, since it requires no assumption as to sample distribution. In the following tables comparisons are made between pairs of samples, and the differences between them are evaluated on various levels of significance. These levels are primarily determined by the sample size. In each box of the tables there appears a pair of numbers. The upper number is the value computed from the sample, the lower is the table value corresponding to a given significance level. Whenever the upper value exceeds the lower, the null hypothesis that both samples come from the same parent population must be rejected, i.e., there is a significant difference between the two samples.

Table B-1

DIFFERENCE IN STRENGTH OF HIGH PURITY ALUMINA
AS A FUNCTION OF GRAIN SIZE
(0.05 significance level)

Grain Size (μm)	10	20	30	40	50
10		0.29 0.45	0.32 0.44	0.74 0.44	0.84 0.44
20	0.29 0.45		0.23 0.45	0.52 0.45	0.79 0.45
30	0.32 0.44	0.23 0.45		0.53 0.44	0.68 0.44
40	0.74 0.44	0.52 0.45	0.53 0.44		0.32 0.44

Table B-2

DIFFERENCE IN STRENGTH OF HIGH PURITY ALUMINA
AS A FUNCTION OF GRINDING ENVIRONMENT

	10-μm grain size		20-μm grain size		30-μm grain size		40-μm grain size		50-μm grain size	
	SA	NV	SA	NV	SA	NV	SA	NV	SA	NV
NA	0.60 0.80	0.67 0.69	0.60 0.80	0.67 0.69	0.60 0.67	0.58 0.69	0.40 0.67	0.47 0.69	1.00 0.67	0.67 0.69
SA		0.33 0.75		0.33 0.75		0.22 0.69		0.49 0.69		0.44 0.69

A = Air Strength.
V = Vacuum Strength
N = Natural Diamond
S = Synthetic Diamond.

Table B-3

DIFFERENCE IN STRENGTH OF A1-600 ALUMINA AS A FUNCTION OF GRINDING AND ENVIRONMENT
(0.01 significance level)

	AC	AD	VC	VD
AC		0.35 0.80	1.00 0.80	1.00 0.80
AD	0.35 0.80		1.00 0.80	1.00 0.75
VC	1.00 0.80	1.00 0.80		0.55 0.80
VD	1.00 0.80	1.00 0.75	0.55 0.80	

A = Air Strength
V = Vacuum Strength.
C = Ground with 120-Grit Diamond.
D = Ground with 600-Grit Diamond

Table B-4

DIFFERENCE BETWEEN VACUUM AND AIR STRENGTHS OF A1-600 ALUMINA REGARDLESS OF GRINDING CONDITIONS
(0.01 significance level)

	Vacuum
Air	1.00 0.67

Table B-5

DIFFERENCE BETWEEN VACUUM AND AIR STRENGTHS
OF Al-300 ALUMINA
(0.01 significance level)

	Vacuum
Air	0.64 0.583

Table B-6

DIFFERENCE BETWEEN VACUUM AND AIR STRENGTHS
OF Al-995 ALUMINA
(0.01 significance level)

	Vacuum
Air	1.00 0.733