

TECHNICAL RESEARCH REPORT

Microstructural Effects on the Machining Performance of Dental Ceramics

by D.T. Le, L. Qi, G. Zhang, S.J. Ng

T.R. 97-36



*Sponsored by
the National Science Foundation
Engineering Research Center Program,
the University of Maryland,
Harvard University,
and Industry*

MICROSTRUCTURAL EFFECTS ON THE MACHINING PERFORMANCE OF DENTAL CERAMICS

D. T. Le, Lixun Qi and Guangming Zhang
Department of Mechanical Engineering & the Institute for Systems Research
University of Maryland
College Park, Maryland 20742

Stanley J. Ng
Naval Air Warfare Center Aircraft Division
Department of the Navy
Patuxent River, Maryland 20670

Abstract

Constraints of mechanical, thermal, and chemical properties are making ceramics the material choice for industrial and dental applications. The quality of a machined surface of ceramics is fundamentally dependent on the response of the material to the machining process. This paper presents a combined analytical and experimental study with focus on optimizing the machining performance of dental ceramics -- DICOR/MGC -- with three distinguished microstructures. The study starts from analyzing the microstructural characteristics to searching for the machining conditions that provide satisfactory performance in terms of acceptable flexural strength. Evidence gained from the cutting force measurements and evaluation of fracture strength degradation indicates that the control of micro-scale fracture formed on the machined surface, with microstructural characteristics being considered, is the key factor which dominates the machining performance.

1 INTRODUCTION

As industry needs continue to push the performance limits of metals, more and more research is being conducted to take advantage of the superior properties of advanced ceramics, such as higher strength-to-mass ratio, and higher resistance to severe environments such as heat, wear, and corrosion. These outstanding qualifications of ceramics have led to their uses in wide-ranging applications where performance and reliability must be ensured and cannot be compromised. Yet, for all the potential that ceramics offer, the question remains in how to best machine these materials. The material removal process in ceramics, unlike that of common metals and plastics, is characterized by micro-cracking and brittle fracture. Consequently, a machined ceramic part tends to possess micro-scale cracks and to fail at a reduced fracture strength.

1.1 NEW MACHINABLE CERAMICS FOR DENTAL RESTORATIONS

In this research, the type of ceramic material chosen for the study is DICOR/MGC, a machinable glass ceramic (MGC) developed at Corning Glass Works [Grossman, 1973]. The chemical makeup of DICOR/MGC consists of SiO_2 , K_2O , MgO , Al_2O_3 , and ZnO_2 . During the heat treatment process, micaceous crystals are formed to give DICOR/MGC its strength and machinability. This crystalline phase is composed of tetrasilicic fluoromica, $\text{K}_2\text{Mg}_5\text{Si}_8\text{O}_{20}\text{F}_4$, and makes up about 50% of the ceramic [Giordano, 1996]. The size and shape of these crystals, or mica flakes, are controlled by the heat treatment process, of which the most critical factor is the temperature of heat treatment. Under different temperatures, the size of these elongated, cylindrical grains may vary from 1 μm to 10 μm in platelet sizes. The difference in microstructure in terms of the size provides variation of mechanical properties of the material. Table 1 lists the three types of DICOR/MGC manufactured from three different heat treatment temperatures [National Institute of Standards and Technology, 1996]. As illustrated in Table 1, the DICOR/MGC material under the heat treatment temperature of 1000 $^\circ\text{C}$ has the smallest grain size among the three with average mica platelets of 1.1 μm in size, called as possessing fine microstructure in this study. On the other hand, the DICOR/MGC material under the heat treatment temperature of 1120 $^\circ\text{C}$ has the largest grain size with average mica platelets of 10.0 μm , called as possessing coarse microstructure in this study. Examining the difference in mechanical properties, the material with fine grain structure has the highest fracture strength ranging from 240 MPa to 280 MPa, and the so-called coarse material has the lowest fracture strength ranging from 115 MPa to 175 MPa [National Institute of Standards and Technology, 1996].

Table 1. Some mechanical properties of DICOR/MGC

| Nomenclature | Heat Treatment Temperature ($^\circ\text{C}$) | Mica Platelet Size (μm) | Fracture Strength (MPa) |
|--------------|---|--------------------------------------|-------------------------|
| Fine | 1000 | 1.1 | 240-260 |
| Medium | 1060 | 3.7 | 210-245 |
| Coarse | 1120 | 10.0 | 115-175 |

1.2 PERFORMANCE OPTIMIZATION

It has been well recognized that grinding is the machining operation to process ceramics in industry [Groenou, 1979]. In applications of dental restorative fabrication on the clinic site, however, grinding is not a rational candidate for meeting the need to achieve complexity in geometrical shapes. Milling operations are in general accepted because most of dental ceramics are machinable.

Figure 1 intuitively demonstrates a three-stage process of dental restorative fabrication. The bulk material is manufactured and supplied to dentists by industry. The CAD/CAM system converts the bulk material to a dental restorative with the required size and shape. The third stage is a post process, such as finishing and polishing with diamond burs, to further improve the esthetics and reliability of the dental restorative. As illustrated in Fig. 1, one of the critical issues to ensure the quality of a dental restorative is to minimize the degradation of material properties during the fabrication process. Such degradation can be caused by several facts, such as the formation of cracks on the machined surface, and the presence of residual stress left after machining. The implication is that before machining, a dental ceramic is characterized by certain mechanical properties, such as its inherent microstructure and fracture strength. However, after the CAD/CAM processes, the mechanical properties of the material are altered in an uncertain, yet permanent fashion, resulting in a restoration with weakened properties, such as low fracture strength. In general, the higher the fracture strength of the material, the more difficult a machining operation can be implemented. The presence of a large cutting force, such as the case of machining the material with fine microstructure at the current study, may induce more micro-cracks on the machined surfaces, thus leading a dramatic loss of fracture strength. On the other hand, the material with coarse microstructure may suffer less in terms of the degradation because the stress distribution produced during machining may not be severe enough to induce micro-cracks on the machined surface. The machining performance optimization calls for a balance between high fracture strength of the material before machining and the high loss of fracture strength of the material after machining. The research to seek such the balance characterizes the joint effort between the material and machining communities to provide the material with high machinability to be processed at a careful selection of machining conditions, thus achieving the optimal performance of a dental restorative.

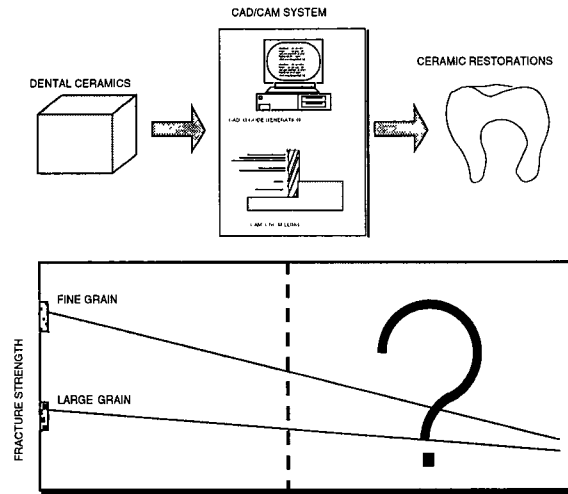


Figure 1. Sequence of CAD/CAM systems on dental ceramics and the degradation in mechanical properties after machining

This paper is organized as follows. In section 2, an experimental procedure consisting of four steps is presented. Details of the implementation of each step are described in section 3. Results of this study obtained from data analyses are provided in section 4. Section 5 gives the conclusions of this study.

2. EXPERIMENTAL PROCEDURE

In this research, the three types of dental ceramic material used are DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-Coarse, as specified in Table 1. The procedure listed below is followed to carry out the experimental investigation :

1) Microstructural Evaluation before Machining: Each material, prepared in both raw (non-polished) and polished form, to be examined using the Environmental Scanning Electron Microscope(ESEM) before machining. All of the prepared specimen have been ultrasonically cleaning, and etched to optimize the conditions for machining tests. To minimize possible variations caused by the initial status of specimens, a unified polishing process is enforced to prepare all the specimens. Half of the polished specimens will be used for machining tests. And the other half of the specimens are not being machined and reserved for the purpose of comparison.

2) Machining Experiments: The method of using A 2^3 fractional factorial

experiments is applied to study the main effects, as well as effects of interactions, of the three key machining parameters, spindle speed, feed, and depth of cut, on the machining performance for each of the three types of microstructural characteristics of the DICOR material. Force signals generated in the feed and transverse directions from the material removal process is recorded and analyzed using a PC computer system powered by LabVIEW software. An empirical model, stating the main effects, and interaction effects relating the cutting force to the machining condition is developed for each of the three types of microstructural characteristics of the DICOR material. .

3) Fracture Strength Evaluation of the Specimens before and after Machining:

Bi-flexural strength tests are performed on the all the specimens before and after machining. Comparison between these results presents the strength degradation associated with individual machining conditions. A second empirical model is developed to characterize the main effects, and interaction effects related to the strength and the machining conditions.

4) Data Analysis: Special efforts are towards to study the interplay between the cutting force generation during machining and the applied machining conditions, thus leading to the identification of correlation between the cutting force generation and the strength degradation.

3. DESCRIPTION OF EXPERIMENTAL INVESTIGATION

3.1 MICROSTRUCTURAL ANALYSIS

Microstructure plays a significant role in governing the machining behavior of ceramics, in terms of its susceptibility to machining-induced surface and subsurface cracking. Therefore, a clear understanding of the microstructural aspect of each material, such as the grain size, orientation, and boundary relations are necessary to interpret machining results. Figure 3 illustrates a computerized image processing system used in this study [Zhang, 1994]. As illustrated, the process begins with a prepared specimen, either raw(non-polished), or polished. The specimens are 28 mm long bars with rectangular cross-section measured as 6 mm wide and 4 mm high x 28 mm. The specimen is placed into the environmental scanning electron microscope (ESEM) where

microstructural images at magnification of 2500x can be clearly observed. The captured images of 640 pixels x 640 pixels are saved in a digital format, stored in the computer system, and can be retrieved for microstructural analyses.

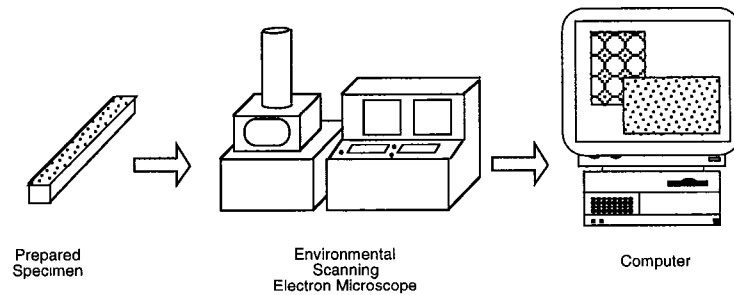


Figure 2. ESEM/computer-based system for microstructural analysis


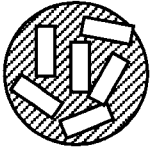
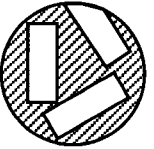
Figure 3 presents SEM micrographs obtained from the performed microstructural analyses. The two columns represent the information before and after machining. The three rows represent the three types of microstructural characteristics, namely, DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials, respectively.

| | RAW | POLISHED |
|--------------|-----|----------|
| Fine DICOR | | |
| Medium DICOR | | |
| Coarse DICOR | | |

Figure 3. Microstructures of Dental Ceramics

As shown in Figure 3, the polished specimens produce better results than those of the non-polished specimen in terms of grain size, grain shape and grain boundaries. For example, the grains of the DICOR material are characterized by elongated rod-like structures of varying sizes, depending on whether the material is fine, medium, or coarse. As expected, the micrographs show that fine and coarse DICOR contain the smallest and largest grains, respectively, while the medium DICOR is somewhere in between. The visual evidence also reveals that these grains are suspended in a glassy phase, as shown most dramatically by the medium DICOR micrograph under the "polished" column in Figure 3. Thus, for DICOR, its microstructural features are characterized by grains of similar shapes but of different sizes, and by the existence of a two-phase structure. Table 2 is a summary of the microstructural characteristics observed for the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials.

Table 2. Microstructural features of DICOR/MGC

| DICOR/MGC | | |
|---|---|--|
| Fine | Medium | Coarse |
| Small sized elongated rod-like grains embedded in a glassy phase | Medium sized elongated rod-like grains embedded in a glassy phase | Large sized elongated rod-like grains embedded in a glassy phase |
|  |  |  |

3.2. MACHINING EXPERIMENTS

Machining experiments, in the form of end milling, are conducted on the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials. A 2-level, 3-variable, factorial design of experimentation is illustrated in Figure 4 [DeVor, 1992]. The three machining variables are spindle speed, feed rate, and depth of cut. The upper and lower levels for each of the three machining variables are listed below:

| | | Spindle Speed (rpm) | Feedrate (mm/min) | Depth of Cut (mm) |
|-------------|-----|------------------------|----------------------|----------------------|
| Upper Level | (+) | 900 | 30 | 0.19 |
| Lower Level | (-) | 600 | 20 | 0.12 |

Table 3 lists a design matrix and Fig. 5 depicts the set of eight tests for each of the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials. Therefore, a total of 24 machining tests are performed in this study.

Table 3. Design Matrix

| TEST | VARIABLE | | | |
|------|---------------|-----------|--------------|---------------------|
| | Spindle Speed | Feed Rate | Depth of Cut | Surface Preparation |
| 1 | - | - | - | - |
| 2 | + | - | - | + |
| 3 | - | + | - | + |
| 4 | + | + | - | - |
| 5 | - | - | + | + |
| 6 | + | - | + | - |
| 7 | - | + | + | - |
| 8 | + | + | + | + |

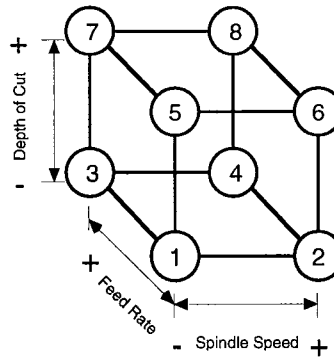


Figure 4. Geometrical representation of the design matrix

The experimental setup for the machining experiments is shown in Figure 5. The rectangular bars are secured to a metal fixture by first placing them in slots which have been machined at evenly spaced locations, and second applying epoxy around the specimens to form a strong bond to the fixture. The machining is performed using a 6.35 mm-diameter end mill with one pass over each specimen. Note that the cutting force is decomposed into its orthogonal feed and transverse components. For clarity, the feed force refers to the force generated in the direction of the path that the end mill travels in its machining operation, while the transverse force is the force generated in the direction that is 90 degrees from the feed direction. During machining, these two force components are measured simultaneously by a dynamometer. Tables 4a, 4b, and 4c list the data obtained from recording the two cutting force components during the 24 machining tests.

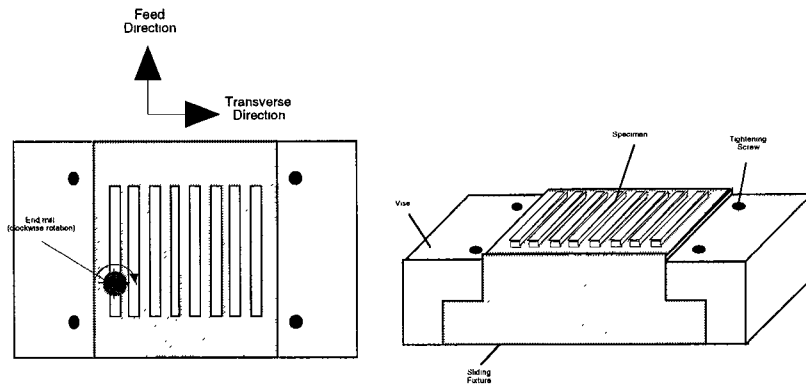


Figure 5. Vise-fixture system for machining

Table 4. Cutting Force Measurement Data

a) Fine DICOR/MGC

| Test # | Machining Conditions | | | Feed Force [N] | Transverse Force [N] |
|--------|----------------------|-----------|--------------|----------------|----------------------|
| | Spindle Speed | Feed Rate | Depth of Cut | | |
| 1 | - | - | - | 15.59 | 3.05 |
| 2 | + | - | - | 2.19 | 0.86 |
| 3 | - | + | - | 3.34 | 2.26 |
| 4 | + | + | - | 11.98 | 3.46 |
| 5 | - | - | + | 1.38 | 2.56 |
| 6 | + | - | + | 6.95 | 4.95 |
| 7 | - | + | + | 7.44 | 7.37 |
| 8 | + | + | + | 3.12 | 3.19 |

b) Medium DICOR

| Test # | Machining Conditions | | | Feed Force [N] | Transverse Force [N] |
|--------|----------------------|-----------|--------------|----------------|----------------------|
| | Spindle Speed | Feed Rate | Depth of Cut | | |
| 1 | - | - | - | 0.53 | 0.39 |
| 2 | + | - | - | 2.34 | 3.30 |
| 3 | - | + | - | 2.64 | 3.94 |
| 4 | + | + | - | 4.86 | 2.94 |
| 5 | - | - | + | 5.18 | 6.15 |
| 6 | + | - | + | 4.40 | 1.70 |
| 7 | - | + | + | 0.88 | 3.34 |
| 8 | + | + | + | 3.18 | 5.31 |

c) Coarse DICOR

| Test # | Machining Conditions | | | Feed Force [N] | Transverse Force [N] |
|--------|----------------------|-----------|--------------|----------------|----------------------|
| | Spindle Speed | Feed Rate | Depth of Cut | | |
| 1 | - | - | - | 0.30 | 0.65 |
| 2 | + | - | - | 1.83 | 1.63 |
| 3 | - | + | - | 2.85 | 3.35 |
| 4 | + | + | - | 2.53 | 1.28 |
| 5 | - | - | + | 2.94 | 4.33 |
| 6 | + | - | + | 2.04 | 0.88 |
| 7 | - | + | + | 1.47 | 1.89 |
| 8 | + | + | + | 4.54 | 6.70 |

3.3. FRACTURE STRENGTH MEASUREMENTS

The specimens after being machined are released from the metal fixture. As illustrated in Figure 6, four-point bending tests are used to evaluate the fracture strength of those specimens after being machined, and those reserved specimens without being machined. The four-point bending tests are performed in parallel to block possible variations introduced to the measurement data. Table 5 presents the data obtained from these measurements. The three columns are used to distinguish the measurements from the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials, respectively.

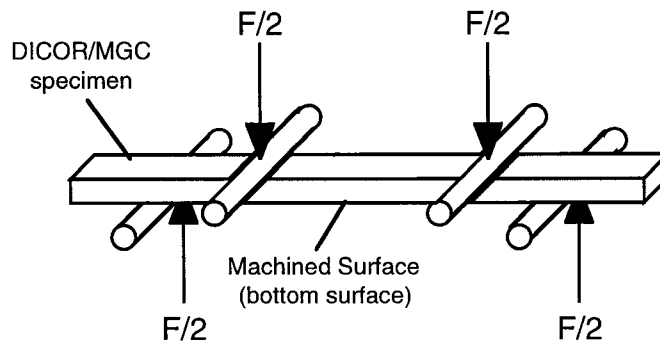


Figure 6. Four-Point Bending Tests to Evaluate Fracture Strength

Table 5. Fracture Strength Measurement Data

| Fine DICOR/MGC | | | | Medium DICOR/MGC | | | |
|----------------|------------------|-----------------|------------|------------------|------------------|-----------------|------------|
| Test # | Before Machining | After Machining | Difference | Test # | Before Machining | After Machining | Difference |
| 1 | 260 | 47 | 213 | 1 | 225 | 41 | 184 |
| 2 | 260 | 49 | 211 | 2 | 225 | 89 | 136 |
| 3 | 260 | 34 | 226 | 3 | 225 | 66 | 159 |
| 4 | 260 | 47 | 213 | 4 | 225 | 34 | 191 |
| 5 | 260 | 44 | 216 | 5 | 225 | 75 | 150 |
| 6 | 260 | 45 | 215 | 6 | 225 | 66 | 159 |
| 7 | 260 | 41 | 219 | 7 | 225 | 62 | 163 |
| 8 | 260 | 31 | 229 | 8 | 225 | 62 | 163 |

| Coarse DICOR/MGC | | | |
|------------------|------------------|-----------------|------------|
| Test # | Before Machining | After Machining | Difference |
| 1 | 145 | 90 | 55 |
| 2 | 145 | 87 | 58 |
| 3 | 145 | 86 | 59 |
| 4 | 145 | 93 | 52 |
| 5 | 145 | 91 | 54 |
| 6 | 145 | 83 | 62 |
| 7 | 145 | 97 | 48 |
| 8 | 145 | 93 | 52 |

4. DATA ANALYSIS AND DISCUSSION OF RESULTS

4.1. RELATION BETWEEN CUTTING FORCE AND STRENGTH DEGRADATION

Examining the cutting force data presented in Tables 4a, 4b, 4c, the three maximum cutting force components of feed force are 16 Newtons, 6 Newtons, and 5 Newtons for the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials, respectively. These three maximum force components were measured when machining the DICOR/MGC-Fine at low spindle speed, low feedrate, and low depth of cut, machining DICOR/MGC-Medium at high spindle speed, high feedrate, and low depth of cut, and machining the DICOR/MGC-coarse at high spindle speed, high feedrate, and high depth of cut. Using the information and examining the data listed in Table 5, the corresponding fracture strengths of these specimens after machining can be identified as 47 MPa for the DICOR/MGC-Fine, 75 MPa for the DICOR/MGC-Medium, and 93 MPa for the DICOR/MGC-coarse, respectively. Figure 7a is the plot of these observation together with the fracture strengths measured from those specimens without being machined. Therefore, the plot clearly depicts the magnitudes of strength degradation for the three cases. The largest drop in fracture strength is 213 MPa

associated with the DICOR/MGC-Fine, and the least drop is 47 MPa. Here the conclusion is the larger the cutting force component in the feed direction is generated, the severe degradation of the fracture strength can be anticipated.

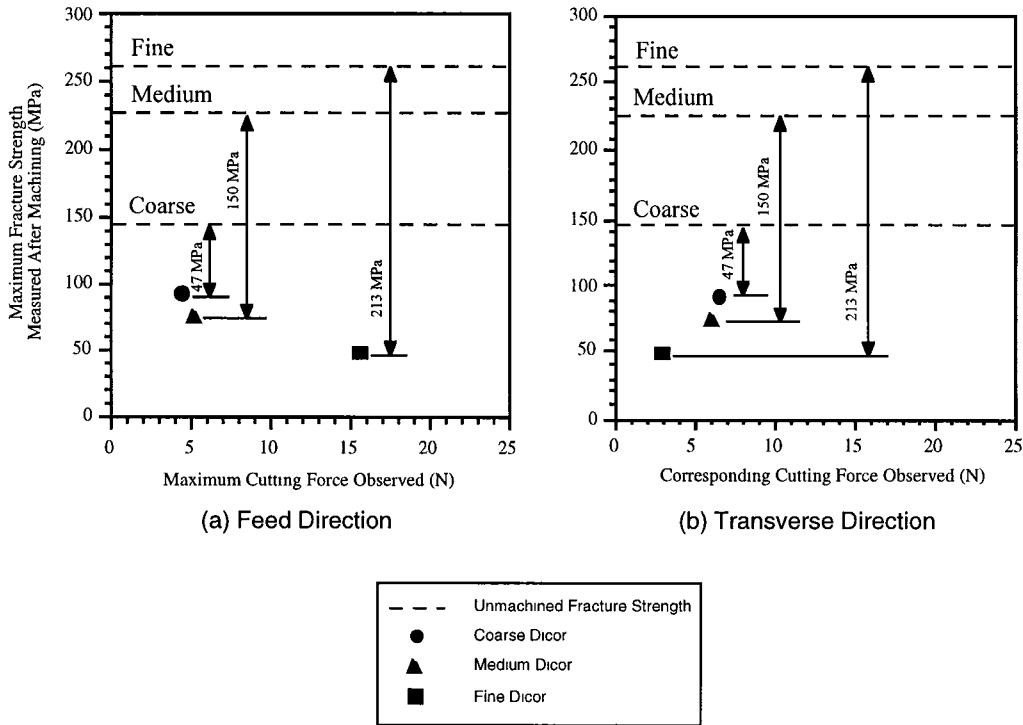


Figure 7. Fracture strength degradation of three types of DICOR/MGC for the a) feed and b) transverse directions

The second observation is related to the data analysis of the cutting force components in the transversal direction. Figure 7b is the plot of the cutting force components in the transversal direction corresponding to the three maximum cutting force components of feed. They are 3 Newtons, 6 Newtons, and 6 Newtons for the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials, respectively. The variation among the three values is 3 Newtons, which is rather smaller than the variation among the three cutting force components in the feed direction, which is 11 Newtons. From comparing the two variations, it is evident that the dramatic drop of the fracture strength degradation of the DICOR/MGC-Fine material is mainly due to the presence of a significant large value of the cutting force component in the feed direction, which is 16 Newtons. This observation is important because it indicates the possible existence of a threshold stress value, above which cracking will be initiated and propagated in a much faster rate during machining. It is important to point out that using the magnitude of cutting force components in the transversal direction may not be as

sensitive as using the component in the feed direction in evaluating the fracture strength degradation.

The third observation from this study is "how to assess the machinability among the DICOR/MGC-Fine, DICOR/MGC-Medium, and DICOR/MGC-coarse materials?" The data presented in this paper could lead readers to think that the DICOR/MGC-Coarse material possesses the best machinability because the fracture strength of the material in its final stage, or after being machined, remains at the highest level if comparing with those associated with the DICOR/MGC-Fine and DICOR/MGC-Median materials. However, the machining conditions explored in this study are very limited. Further investigations to look at the machining performance under machining conditions different from what has been used are imperative. In addition, interplay between the grain size and the machining condition applied is so complicated that DICOR/MGC materials with various grain sizes, especially between 5 μm to 10 μm in platelet sizes, are needed to further search those machining conditions, under which higher strength levels could be reached.

5. CONCLUSIONS

This paper presents a research effort to perform a combined analytical and experimental study. The study is focused on identifying fracture strength degradation caused by micro-crack fracture induced during the machining of dental ceramics. Results obtained from this study clearly indicate that microstructural characteristics of dental ceramics have dominant effects on their mechanical properties, thus leading to variation of the response of the material to the machining process. To optimize the clinical performance of dental ceramics, a balance has to be made between the effort in material synthesis and the effort in identifying appropriate machining conditions to achieve the minimized strength degradation with a sufficiently high strength sustained after machining.

Acknowledgment

The authors acknowledge the support from the University Research Board, the Mechanical Engineering Department, and the Institute for Systems Research under NSFD

CDR-88003012 grant and NIDR grant P01-DE01976. Special thanks are due to Bill Lawson and Dave Stenbakken for their valuable contribution in performing microstructural analyses. The authors would also like to thank Dr. Dianne Rekow at the University of Medicine and Dentistry of New Jersey and Dr. Irene Peterson at the National Institute of Standards and Technology at Gaithersburg for her assistance in completing the fracture strength evaluation. The support from the Northeast Consortium for Engineering Education (NCEE) of the Department of Defense is deeply appreciated.

References

- Devor R., Chang T., and Sutherland J., Statistical Quality Design and Control, Macmillan Publishing, 1992.
- Giordano, R. A., Dental Ceramic Restorative Systems, Compendum, Vol. 17, No. 8, 1996.
- Groenou, A. B. V. and Veldkamp, J. D. B., "Grinding brittle materials," Machining Hard Materials, Society of Manufacturing Engineers, 1982.
- Grossman, D. G.: Tetrasilicic mica glass-ceramic material, Washington, D.C.: US Patent Office, US Patent No. 3,732,087, 1973
- Labview Software Manual, National Instruments, Inc., 1994.
- Lawn B. R., Fracture of Brittle Solids, Cambridge University Press, Cambridge, 1993.
- National Institute of Standards and Technology, Machinable Ceramics Monthly Report, 1996.
- NIH Image Version 1.58: Image Processing and Visualization Software, National Institutes of Health, 1994.
- Zhang, G. M., Ko, W. F. , and Ng, S. J., " Submerged Precision Machining of Ceramic Material", Proceedings of 1995 Joint ASME Applied Mechanics & Materials Conference, AMD-Vol. 208, pp. 65-79., 1994.
- Zhang, G. M., Ng, S. J., & Le, D. T., " Characterization of Surface Cracking Formed during the Machining of Ceramic Material", Proceedings of 1995 ASME International Mechanical Engineering Congress and Exposition, MED-Vol., 2-1, pp. 415-429, 1995.