

THESIS REPORT

Master's Degree

Assessment of Surface Integrity of Mechined Ceramics Using Image Processing

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Abstract

Title of Thesis: Assessment of Surface Integrity of Machined Ceramics
using Image Processing

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Recent manufacturing advancements have allowed designers to create ceramic materials with tightly controlled microstructures and compositions. Some advantageous properties of these materials are their lightweight, high strength, and resistance to heat and corrosion. However, advanced ceramics pose additional reliability problems. Specifically, the brittle nature of advanced ceramics often leads ceramic components to sudden catastrophic fracture during service.

To address the problem of inadequate reliability caused by microcracks formed on the surface, direct contact techniques -- such as stylus profilometry, scanning tunneling microscopy, and atomic force microscopy -- have been traditionally used to assess the surface integrity. Some problems associated with these assessment techniques are the lack of achievable resolution and the introduction of additional damage during the measurement process. In this thesis, DICOR/MGC, a dental restorative ceramic, is selected for this study. Specimens are prepared by a milling

process using a designed set of machining parameters to examine their roles on surface integrity. A surface assessment system incorporating environmental scanning electron microscopy and image processing techniques is used to quantify the surface integrity in terms of roughness average and cavity density. By utilizing the image data, the developed technique allows assessment of surface integrity in a unique nondestructive manner. The unique contribution of the thesis is the establishment of penetration depth and parameters of the developed image system to explore the potential of assessing surface cracking. Analysis of the internal stress distribution using finite element method provides deep insights of the micro-scale mechanism of material removal during machining. Significant findings are directing the development of a crack controlled machining technology.

**ASSESSMENT OF SURFACE INTEGRITY OF MACHINED
CERAMICS USING IMAGE PROCESSING**

by

Lenox Selvakumar Job

Thesis submitted to the Faculty of the Graduate School
of The University of Maryland in partial fulfillment
of the requirement for the degree for
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Advisory Committee:

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Professor Nariman Farvardin**

Dedication

To my parents, brother, and sister.

Acknowledgments

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Nomenclature

A	Atomic Weight
α	Significance Level
α	Tilt Angle
β	Skewness
BSSZ	Berger-Seltzer Equation
BTH	Bethe Equation
dE	Differential Energy Element
dS	Differential Displacement Element
Δd	Distance between points a and b for the untilted surface
Δd_0	Distance between points a and b for the tilted surface
Δz	Height of an object
Δz	Parallax
E	Instantaneous Energy
E _i	Electron Energy after Scattering Event
E ₀	Initial Beam Energy
F _n	Tangential Milling Force (Axial Direction)
F _t	Normal Milling Force
GR1	Gray level intensity of point a for the untilted surface
GR2	Gray level intensity of point b for the untilted surface
J	Mean Ionization Energy
k	Number of Variables
λ	Mean Free Path
λ_a	Mean Free Path for Scattering Event a
λ_{Total}	Total Mean Free Path
M	Magnification

n	Number of Replicated Runs
n	Total Number of Electrons
ne	Total Electron Counter
$Q(>\phi_o)$	Probability of Scattering Event with Angle Greater than ϕ_o
ρ	Density
R_a	Roughness Average
R_B	Bethe Range
R_{KO}	Kanaya-Okayama Range
R_{KO}	Kanaya-Okayama Range
R_q	Root Mean Square Roughness
RTF	Rutherford Cross Section
s	Standard Deviation
$SA1$	Sampling Area 1
$SA2$	Sampling Area 2
$SA3$	Sampling Area 3
SS	Sum of Squares
$\bar{x}$	Mean Value of Escape Depth
x_i	Specified Depth Thickness Histogram Bin
Z	Atomic Number

Chapter 1

Introduction

1.1 Advanced Ceramic Materials

Traditional ceramic materials have been used extensively for many years. By heating ceramic raw materials which had been molded into finished shapes, designers manufactured products such as dishes, tiles, brick, pottery, and enamels. However, recent advancements in material processing have allowed scientists to manufacture ceramic materials with tightly controlled microstructures and compositions. These materials, classified as modern ceramics or advanced structural ceramics, are noted for their unique mechanical and chemical properties [1]. In general, many of the advanced structural ceramics are used in applications in which high strength at elevated temperatures, corrosion and wear resistance, and low weight are essential. Alumina (Al_2O_3), for example, is noted for its versatility. Some of the applications for alumina include its use in prosthetics, artificial teeth and bones, and as insulators in spark plugs. High temperature components, used to contain molten metals, are also fabricated from this ceramic. Other ceramics noted for high temperature applications in the automotive and gas - turbine industry are silicon carbide (SiC) and silicon nitride (Si_3N_4), respectively. Furthermore, boron carbide (B_4C), noted for its high strength and low weight, is used to fabricate armor, while sialon ($\text{Si}_3\text{Al}_3\text{O}_3\text{N}_5$) is used in tool inserts required to machine cast irons and nickel-base superalloys. Finally, an application of another ceramic -- urania (UO_2) -- is as a nuclear reactor fuel [2].

1.2 Processing of Ceramics

The basic methodology of manufacturing advanced ceramic components consists of three steps -- powder processing, shaping, and densification. The first step, powder processing, consists of crushing the raw materials to obtain correctly sized powders. This is achieved by ball or hammer milling and can be performed in a wet or dry environment. Next, additives, which assist with the shaping and densification processes, are mixed with powder particles that have been ground to the appropriate size.

The second step -- shaping of the powder material -- can be accomplished by casting, plastic forming, or pressing. Slip casting is one of the casting techniques commonly used in the shaping process. This technique involves pouring a solution containing ceramic particles and a liquid into a porous mold. After, some of the liquid is absorbed, and the mold is inverted, the remaining fluid flows out allowing the ceramic particles to dry. The shaped component is removed after the mold is opened. Another shaping mechanism -- plastic forming -- can be accomplished by injecting molding, jiggering, or extrusion. These techniques involve active contact with the liquid - ceramic solution. The third shaping process -- pressing -- can be used to shape as well as densify the ceramic - liquid suspension. Different pressing techniques, such as dry pressing, wet pressing, isostatic pressing, hot pressing, and hot isostatic pressing, consist of applying pressure to the liquid - ceramic solution under different conditions. Hot pressing, for example, consists of the simultaneous application of heat and temperature.

The final step in manufacturing ceramics involves densification of the shaped component. This is done by drying the pre-shaped part and then firing or sintering the

component. Sintering, a process in which the component is heated to high temperatures, is used to strengthen the ceramic material and to reduce its porosity [3]. Overall, the manufacturing process can be summarized as shown in Figure 1.1.

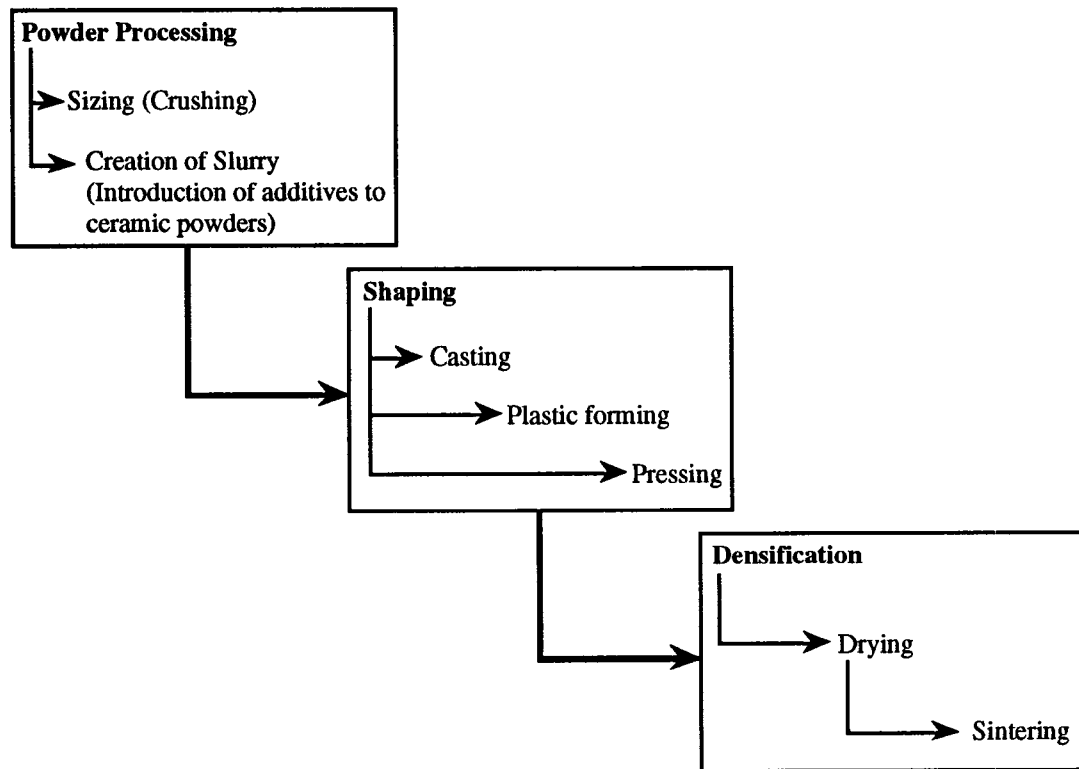


Figure 1.1 Manufacturing of Advanced Ceramics

1.3 Scope of Thesis

The primary focus of this thesis is characterization of machined surface defects using an innovative image processing technique - stereophotogrammetry. Overall, an image processing system was developed to acquire and manipulate image data from an environmental scanning microscope. Specifically, the surface texture was reconstructed from electron micrographs using image processing and mathematical software. Next, the roughness average was computed from a specified area of the

surface. To generate different surface textures, a set of samples were machined using different milling conditions. The cutting forces were monitored to correlate machining conditions to surface finish. An analytical investigation of stress distributions during milling was also performed to investigate stress distributions during machining. Specifically, a finite element method (F.E.M.) analysis using the measured cutting forces as input parameters was performed.

1.4 Organization of Thesis

The ultimate goal of this thesis is to assess surface integrity of machined ceramic surfaces using environmental scanning microscopy and image processing. The assessment process provides basis for the development of new and innovative machining technologies. Overall, the thesis work is performed in four stages. In the first stage, a detailed literature survey is conducted to examine the advantages and disadvantages of advanced ceramics and to examine conventional surface integrity assessment techniques. Chapter 1 provides a brief overview of some of the applications of advanced ceramics. Furthermore, processing techniques used to manufacture ceramics are also described in the chapter. In Chapter 2, pertinent background information is provided. To begin, the physical and mechanical properties of advanced ceramics are described in this chapter. Next, two primary techniques used to machine advanced ceramics -- grinding and milling -- are discussed. Then, issues of concern when designing with ceramics are discussed. In addition, findings from a literature survey on assessment of surface finish are also presented in Chapter 2. The advantages and disadvantages of different contacting and noncontacting surface integrity assessment techniques are examined. In addition, optical and non-optical surface evaluation systems are also investigated.

The second stage of the thesis work relates to the image acquisition system. Chapter 3 describes the surface integrity assessment system used to evaluate the surface roughness, and detect surface and subsurface damage. A description of the different modules of the environmental scanning electron microscope based system is provided. Fundamental concepts regarding electron interactions with materials are also examined in the chapter. In Chapter 4, a feasibility analysis to determine the applicability of the environmental scanning electron microscope based surface integrity assessment system is discussed. In particular, results from an analytical investigation of depth of penetration of electrons into a flat bulk surface are described. Overall, the role of different parameters on depth of penetration is examined.

The third stage consists of the machining experiments. In Chapter 5, the parameters used in the milling experiments are described. In addition, the cutting forces measured during the machining of DICOR/MGC dental ceramic material are also provided in the chapter. Furthermore, results from an experimental analysis of surface integrity are discussed in Chapter 5. Next, in Chapter 6, the measured cutting force data, described in Chapter 5, are used as input parameters for a finite element method analysis. This analytical investigation provides insight into the stress distributions which occur during machining.

The final stage of the thesis work consists of discussing the significance of research. Chapter 7 provides a discussing relating the significance of the cutting force measurements, finite element method analysis, and surface integrity assessment. Furthermore, conclusions and recommendations for future work are also provided.

Chapter 2

Background

2.1 Properties of Advanced Ceramics

As previously stated, advanced ceramics exhibit unique mechanical properties, many of which are directly attributable to their microstructure. In general, these materials can be characterized as complex compounds containing both metallic and nonmetallic elements joined by covalent and ionic bonds. However, depending on the particular elements and their microstructures, properties among the different advanced ceramics vary. An advantage directly attributable to the bonding mechanism is the high melting temperature of these materials. In general, to date, the engineering materials with covalent bonds have the highest melting temperatures. Furthermore, those ceramics containing transition metals tend to have high melting temperatures. A second advantage of advanced ceramics is their light weight. This is attributable to the lower densities of most of these materials when compared to conventional engineering materials such as metals. However, depending on the elements involved, certain advanced ceramics such as tungsten carbide (WC), due to the high atomic numbers of their elements, have greater density values than their metallic counterparts. In turn, these ceramics are heavier, not lighter, than their metallic counterparts. Another important advantage of engineering ceramics is their superior compressive strengths when compared to metals.

Thermal shock, which can initiate cracks in ceramic materials, results from a build up of residual stresses due to a repeated cycles of large changes in temperatures in high temperature environments. Certain ceramics such as silicon carbide (SiC)

exhibit excellent thermal conductivity and thermal shock resistance. On the other hand, other ceramics such as alumina (Al_2O_3) exhibit low thermal conductivities allowing for their use as insulators. Finally, another important property of advanced ceramics -- their wide range of electrical conductivities -- is attributable to the combination of elements in the materials. For some materials, the electrical conductivity of a given ceramic can be increased by mixing a semiconducting or conducting material with a nonconducting material. This allows ceramics to exhibit semiconducting characteristics while retaining their other unique physical properties [1].

2.1.1 Material Selection

The variety of potential industrial applications of advanced ceramic materials provides designers with opportunities of constructing lightweight, high strength, heat resistant components. Applications of advanced ceramics include their use as insulators, nuclear fuels, light weight armor components, and high temperature engine components. In addition, in terms of biomedical applications, advanced ceramics have been used in prosthetic devices, orthopedic implants, and as dental restorative material. In particular, a growing area of research is the use of advanced ceramics for dental restorations. Advanced ceramics provide a unique opportunity of developing dental restorations which closely resemble natural teeth in terms of chemical durability, biocompatibility, strength and color. However, the brittle behavior of ceramics makes them susceptible to fracture, resulting in failure of the restoration. In general, machined components with smooth surface finishes are desired. Recently, the development of machinable ceramics for use in dental computer aided design (CAD) / computer aided manufacturing (CAM) applications has resulted in materials which closely resemble the properties of human enamel. In this thesis, the machinability of

DICOR/MGC ceramic material is assessed by means of a 2 level 3 factor factorial design experiment [4, 5].

2.1.2 Composition and Properties

The glass-ceramic selected for the machining experiments was DICOR/MGC. The chemical formula for this tetrasilicic mica glass ceramic -- which contains the elements potassium (K), magnesium (Mg), fluorine (F), silicon (Si), and Oxygen (O) -- is $K_2O-MgF_2-MgO-SiO_2$. In terms of the microstructure, DICOR/MGC is composed of uniformly dispersed randomly oriented mica crystals, whose average diameter is about 1 μm . Overall, the unique microstructure of DICOR/MGC enhances its machinability by blunting propagating cracks [4]. Some of the physical properties of this ceramic are listed in Table 2.1 [4].

Table 2.1 Properties of DICOR/MGC Ceramics

Property	Value
Density [g/cm^3]	2.8
Translucency	0.44
Modulus of Elasticity [MPa]	$6.8(10^4)$
Thermal Conductivity [$W / m \text{ } ^\circ K$]	1.6
Thermal Diffusivity [mm^2 / s]	0.79
Specific Heat	74.1
Coefficient of Expansion	6.4

2.2 Machining of Advanced Ceramics

Due to their enhanced strength and hardness characteristics, advanced ceramics pose machining related problems. In particular, low material removal rates and accelerated tool wear are two primary issues of concern. Currently, machining of structural ceramics, at present, is primarily accomplished by grinding and milling. Grinding is extensively used since the forces generated during this machining process are comparatively lower than the those generated by the other machining methods. This reduction in forces are directly attributable to the machine tool -- the grinding wheel -- which consists of a thin layer of bonded abrasive particles. Furthermore, the lower forces also result in a corresponding decrease in wheel wear and workpiece damage. On the other hand, milling is used in applications involving detailed geometries, i.e., intricate workpieces can be machined more rapidly using milling than grinding [3, 6].

2.2.1 Machining Techniques

2.2.1.1 Grinding

Grinding can be considered as a chip removal machining process with the abrasive grit particles serving as cutting tools. Material of a specified depth of cut (d) is removed as the workpiece (moving at tablespeed (v_w)) is fed into the grinding wheel rotating at speed (v_s). As indicated in Figure 2.1, downgrinding occurs if the wheel and workpiece move in the same relative directions. On the other hand, an upgrinding process results when the wheel and workpiece move in opposite directions

relative to each other. Furthermore, the location of the grits on the wheel and the geometries of individual particles are randomly distributed [7].

Overall, as shown in Figure 2.2, the grinding system is comprised of four different components: the machine tool, the grinding wheel, the grinding fluid (coolant), and the workpiece. Of these four elements, workpiece properties can vary significantly among the different materials. Each of three other components plays a unique and critical role in the grinding process [7].

The machine tool contains the spindle, table, and feed mechanisms. In general, machine tools used for the grinding of ceramics are stiffer (more rigid) than their counterparts used in the grinding of conventional engineering materials. This is required because of the increased hardness of ceramic materials. Furthermore, high rigidity in the machine tool is also needed to avoid excessive vibration which can be detrimental to the quality of surface finish.

The grinding wheel, the cutting tool of this machining process, consists of grit particles embedded in a bonding medium. Generally, wheels used in the machining of modern ceramics contain synthetic diamond particles. These particles are typically attached to the wheel with resin bonds. Although other bonds such as vitreous and metals bonds exist, resin bonds, which contain the particles in pliable matrix, are extensively used because of their durability in withstanding shock loads and their high strength [7].

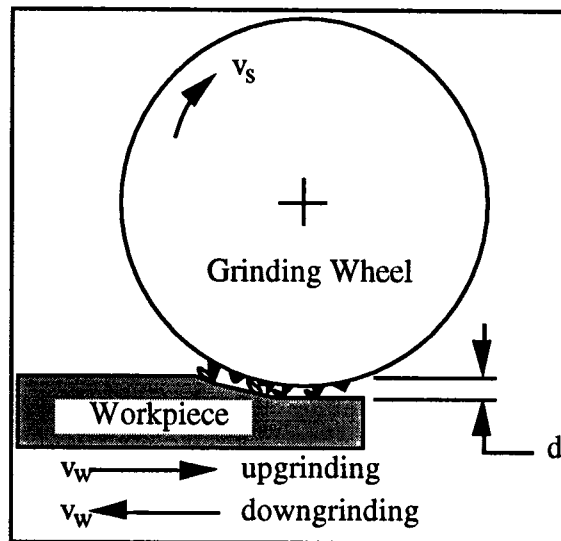


Figure 2.1 Grinding Modes

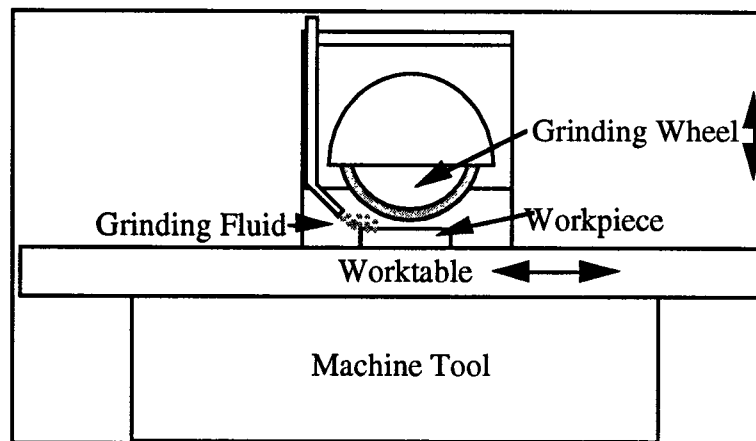


Figure 2.2 Grinding System

The primary roles of the grinding fluid are lubrication and removal of heat. In terms of lubrication effects, grinding fluids reduce the friction and force between the tool and workpiece, resulting in an improvement in tool life. The fluid also removes the debris remaining on the grinding wheel and or workpiece. Furthermore, the

grinding fluid assists in the control of temperature in the grinding zone. In practice, due to the high speed associated with grinding, the grinding fluid usually has to be applied to the workpiece - grinding wheel interface at a high pressure to ensure proper lubrication [7].

2.2.1.2 Milling

As previously stated, milling is used to machine components requiring intricate geometries. Depending on the machining requirements, different milling configurations; such as slab milling, face milling, and end milling, can be utilized. For the experiments performed for this thesis, end milling was used. In this machining mode, the cutter rotates with a fixed speed N while the workpiece is moved. Furthermore, end milling can be classified as either up milling or down milling. In up milling, the workpiece moves in a direction opposite to the rotation of the cutter. On the other hand, in down milling, the workpiece moves in the same direction as the rotation of the cutter (see Figure 2.3 [3]).

Overall, column - and - knee type machines are extensively used for most milling operations. For end milling, a vertical - spindle column - and - knee type machine is used. As shown in Figure 2.4, the primary components of this machine are the worktable, saddle, knee, and head. The workpiece to be machined is attached to the worktable, which moves horizontally (left and right). In turn, the worktable is supported by the saddle, which also moves horizontally (in and out). The vertical movement of the table (up and down) is controlled by the knee. This component allows the user to control the depth of cut. Finally, the head houses the spindle and different cutters [3].

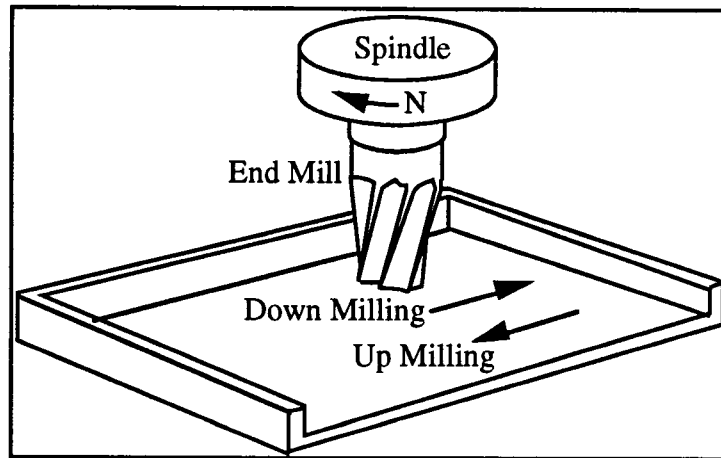


Figure 2.3 End Milling Configurations

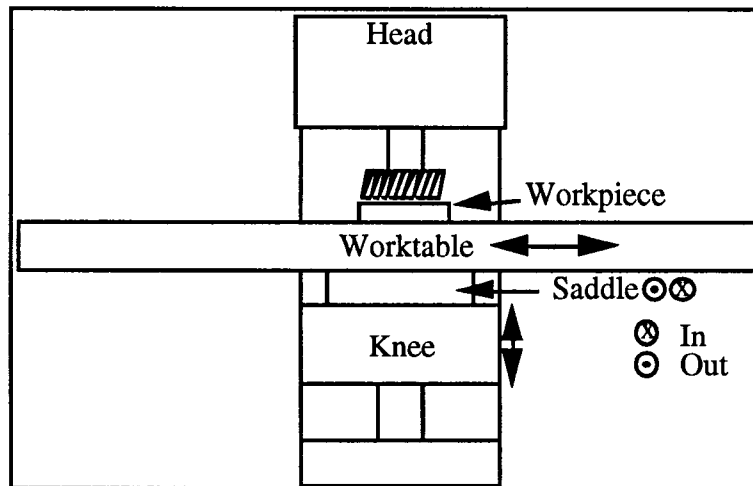


Figure 2.4 Vertical-Spindle Column-and-Knee Type Machine

In recent years, the advancement of computer numerical control (CNC) grinding and milling machines have allowed further control over the machining of advanced ceramic materials. In the CNC machines, a computer program is used to control the machining process. Overall, the program is divided into blocks which

contain codes to produce the desired workpiece. Parameters such as spindle speed, tool selection, feed rate, depth of cut, and the like, are controlled by the program. Therefore, CNC machining offer unique opportunities of performing systematic investigations related to machining topics [8]. In this thesis work, all of the machining tests are performed on a Matsuura MC-570 Milling Machining Center.

2.2.2 Machinability of DICOR/MGC Ceramic Material

As previously discussed, the advantageous properties of advanced ceramics over conventional engineering materials also pose unique machining problems, especially related to material processing. Ceramics are inherently brittle in nature. Hence, conventional machining rates, if they are used to machine ceramic materials, may induce problems in terms of product reliability. In experiments performed at the College of Dentistry at Ohio State University, DICOR/MGC was selected. DICOR/MGC is a type of material that is specifically designed for dental CAD/CAM applications. In their work, the authors point out that material removal rate for this material is relatively close to that of human enamel. Overall, the experiments consisted of using diamond dental burs to remove material from samples. Next, the material removal rate of different glass-ceramics in the first ten initial passes were computed. The load and infeed were maintained at fixed values for the tests [4]. The initial material removal rates (measured during the first 10 passes) for the different materials are listed in Table 2.2 [4].

Table 2.2 Initial Material Removal Rates for Different Glass-Ceramics

Material	Initial Material Removal Rate [cm ³ /min]
DICOR/MGC (Dark)	0.03

Enamel	0.05
Hydroxy Apatite	0.07
Macor	0.12

2.3 Designing with Advanced Ceramics

Issues which need to be addressed when designing a product include: thermal shock, chemical environment, stress distribution, cost, reliability, toxicity, temperature, load, safety requirements, surface finish, friction, and pollution [1]. However, beside these design considerations additional topics need to be addressed when designing with advanced ceramic materials. Although noted for their high strength, modern ceramics are relatively brittle, hence, making them susceptible to surface and subsurface cracks during processing. In turn, additional problems associated with component reliability and life cycle requirements need to be addressed. In addition, poor surface finish can significantly degrade the overall strength of the machined ceramic components. The overall quality of the surface finish may pose problems associated with quality control and reliability.

The selected material -- DICOR/MGC -- is primarily used in dental restorations. This particular application poses an additional design concern -- wear of human enamel against the ceramic restoration. Ideally, both the human enamel and ceramic restoration should wear at the same rate. However, since most ceramic materials are stronger than human enamel, the human enamel wears at a faster rate than the artificial restoration. In a study performed at the University of Minnesota, the wear of different dental ceramics against human enamel was simulated in an oral

environment [4]. The designed apparatus used to perform the wear tests allowed controlled force movement cycles in a simulated "artificial mouth." As shown in Figure 2.5 [4], the volume loss of the selected test material (DICOR/MGC - Dark) was fairly close to that of human enamel when compared to the other materials. However, when comparing the differences in volume losses between the dark and light DICOR/MGC samples, it can be observed that the dark samples wear more quickly than human enamel -- a desirable trait.

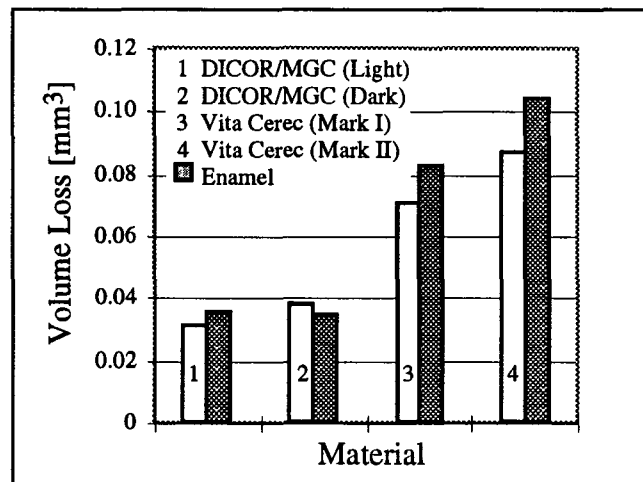


Figure 2.5 Wear Results for Different Dental Ceramics

Overall, current major impediments in the use of advanced ceramics include their high fabrication and machining costs. Due to a general lack of optimization data, machining costs associated with these materials are noticeably higher than costs associated with machining conventional engineering materials. There is an urgent need to determine the correct machining parameters to address economic and reliability concerns associated with the machining of advanced ceramics. In general, ceramics primarily fail from brittle fracture. Hence, any material, processing, or service defects which aid the propagation of crack formation may lead to catastrophic

failure of ceramic components. The detection and control of microcracks is a key issue of importance during the machining of ceramics [1,9].

2.4 Assessment of Surface Integrity

In many industrial applications, the characterization of surface topography cannot be underestimated. In particular, surface topography is critical in the study of lubrication, wear and tribology. In addition, surface roughness is an important performance index used to assess the quality of optical and semiconductor components. The processes used to manufacture ceramic parts -- cleaning, etching, coating, and heating -- can adversely effect the surface finish. Another important discipline which uses surface topography extensively is the medical industry. Specifically, changes in tomography of vital organs, tissues, and the like, are of utmost importance in the diagnosis and onset of diseases [10]. As described in the previous section, the assessment of surface integrity of machined ceramic components is of utmost significance for quality assurance of reliable components.

To characterize surface integrity, numerous instruments have been invented. Overall, the methodology to quantify surface finish can be divided into contact techniques and non-contact techniques. As implied by the name, contact techniques involve direct contact of the specimen and transducer. On the other hand, in non-contact techniques, the sample and measurement instruments are not in direct contact. In this thesis, a non-contact technique is used to characterize surface integrity in machined ceramic samples. Overall, the technique is used to assess surface finish. In addition, the feasibility of using the system to assess subsurface damage is also examined in the thesis.

2.4.1 Contact Techniques

There are numerous methods which measure surface topography by direct mechanical contact of the workpiece. However, traditionally, stylus based instruments have been extensively used to determine surface roughness. In addition, some of the newer contact techniques include scanning tunneling microscopy and atomic force microscopy.

2.4.1.1 Stylus Based Instruments

The basic stylus instrument consists of the following five components: a transducer, a chart recorder, an amplifier, a traverse unit, and a meter system. In the transducer, a linear variable differential transformer (LVDT), changes in amplitude or height vary the mutual inductance. In turn, the change in mutual inductance alters the phase of a high frequency carrier signal. Then, the signal is amplified and demodulated to represent the surface topography. Next, the signal can be recorded on a chart recorder. As shown in Figure 2.6 [10], the traverse unit displaces the entire instrument in the horizontal or spatial direction. The skid is used as a reference datum according to which the surface topography is recorded [10]. Some commercial stylus based profilometers include the Perthometer S8P, the Surfcom 475/575-3D, and the Surfscan 3-D.

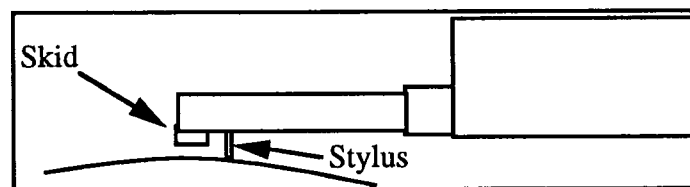


Figure 2.6 Stylus Instrument

The traditional stylus based profilometers are popular for several reasons. First, these instruments are not as expensive as other commercially available surface measurement systems. Second, workpieces do not need to be prepared before examination. In terms of resources and time, this is a considerable advantage of these measurement instruments. Third, the test procedure involved in obtaining a surface profile is relatively simple.

However, the disadvantages associated with stylus type profilometers considerably outweigh the aforementioned advantages. First, these profilometers have been traditionally used to produce two dimensional (2-D) representations of the surface texture. The primary problem with individual profiles obtained at different locations of the workpiece is their lack of a comprehensive representation of the machined surface. Recently, stylus type measurement instruments have been modified to create three dimensional (3-D) surface plots. However, the time required to record a 3-D surface plot using this measurement instrument is considerably longer than when using other measurement instruments. Hence, stylus based instruments cannot be adequately used in on-line monitoring systems.

Second, the effect of waviness in the overall roughness measurement is another issue of concern. Waviness, an index of the overall flatness of the surface, will be significant if a workpiece has topographical features with a wavelength larger than the skid length. Third, isolated extremities in the surface finish may yield erroneous measurement data. Fourth, the contact between the stylus and the specimen may result in permanent damage to both the stylus and the specimen, i.e., the measurement process may introduce damage.

Finally, wear of the stylus is also a significant disadvantage of stylus type instruments. With increasing wear, the geometry of the tip of the stylus changes, i.e., the radius of the stylus tip changes. In turn, as shown in Figure 2.7, the topography will not be accurately traced. Any irregularities smaller than the nose radius of the stylus will not be detected. Consequently, the resolution specified by the manufacturer will no longer be attained [11].

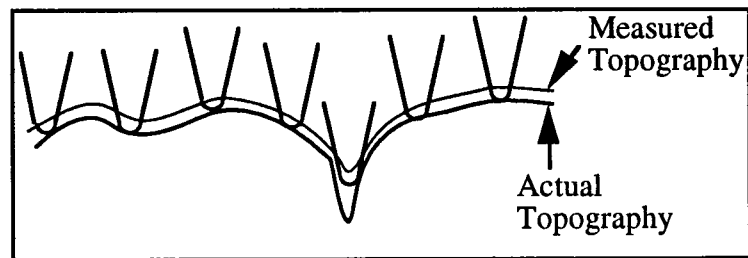


Figure 2.7 Effect of Worn Stylus Tip

2.4.1.2 Tactile Tests

In these tests, specimens are examined by running a finger over the surface. Next, the "feel" of the surface finish is compared to that of a calibrated standard. By matching the surface texture with a known standard, an estimate of the surface roughness is obtained. A particularly attractive advantage of this technique is its simplicity. The sample does not need be prepared before examination. Furthermore, in terms of time and resources, the test procedure is noticeably simpler and cheaper than the other measurement techniques.

However, a considerable number of disadvantages are also associated with this comparative measurement technique. First, tactile tests involve subjective decisions regarding the surface finish. Hence, the finish can only be qualitatively described. Second, the measurement technique is not fast enough to be implemented in an on-line

monitoring process. Third, relative differences in smooth surfaces may not be readily discernible if tactile tests are used [10].

2.4.1.3 Scanning Tunneling Microscopy

In this surface measurement technique, a sharp metal tip of microscale size is mounted on micro-driving unit capable of moving in 3 dimensions (x, y, z). A tunneling current consisting of an electron cloud is introduced between the stylus tip and the surface. The height data are obtained by traversing the stylus over a constant current area or by varying the current between the tip and the surface. In the first mode, a constant distance between the tip and surface is maintained by varying the current via a feedback control system. Changes in the current are used to quantify the height data. In the second mode, the height of the stylus is fixed. However, the tunneling current varies according to changes in height of the surface.

The primary advantage of using scanning tunneling microscopy to characterize the surface finish is its high vertical sensitivity. A change in displacement of order of 0.1 nm results in corresponding order of magnitude change in the tunneling current. However, there are two considerable disadvantages of using this technique to evaluate surface topography. First, to ensure a continuous tunneling current, this technique can only be used to characterize the surfaces of electrically conductive samples. Second, another primary disadvantage of this technique is maintenance of a sharp stylus tip. Due to its atomic size, conventional methods such as grinding, electromechanical etching, and so on, cannot be used to sharpen the tip [10].

2.4.1.4 Atomic Force Microscopy

A close derivative of the scanning tunneling microscope is the atomic force microscope. As shown in Figure 2.8, a sharp atomically sized metal tip connected to a cantilever beam is traversed over the surface of the sample. However, in an atomic force microscope, a direct contact between the surface and tip is established. The surface roughness is obtained from the atomic force between the stylus tip and the surface. As before, different modes of operation exist. In the first mode, the sample is modulated with the natural frequency of the cantilever beam in the height direction. In turn, the cantilever is deflected because of the force between the two objects. A tunneling current, which controls the feedback mechanism, is modulated to maintain a constant force. In the second mode of operation, the cantilever beam is modulated at its natural frequency. In turn, both the phase and amplitude of the tunneling current are changed. The amplitude controls the feedback mechanisms. In the third mode, the phase, instead of the amplitude is used to control the feedback mechanism. Finally, in the fourth mode, a constant gap between the workpiece and the stylus tip is maintained by varying the force on the tip [10].

There are several advantages of using the atomic force microscope for surface measurements. First, as before, the attainable resolution in the spatial and amplitude directions is noticeably better than that of conventional stylus type profilometers. Second, if the atomic force microscope is used, workpieces do not have to be conductive. Third, the speed with which the surface topography can be reconstructed allows for possible the use of the technique in on-line monitoring processes. As in the case of the scanning tunneling microscope, the primary disadvantage of the atomic force microscope is wear of the stylus tip [10, 11].

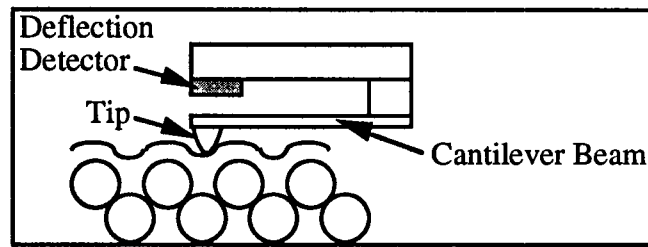


Figure 2.8 Atomic Force Microscopy

2.4.2 Non-contact Techniques

Several innovative non-contact methods of assessing surface roughness have been introduced. Due to the lack of mechanical contact between the measurement instruments and specimen, all these techniques are noted for inducing no damage in the workpiece during surface measurements. Furthermore, most of the non-contact surface measurement techniques can be readily adapted to provide a 3-D representation of the topography. Surface roughness parameters based on areas instead of profiles provide a significantly better indication of the surface finish. In addition, some of these techniques can be used in on-line monitoring processes. Overall, the non-contact techniques can be divided into optical methods and non-optical methods. In optical instruments, images are viewed using optical lenses. On the other hand, in non-optical instruments, images usually consist of computer images which are displayed on terminals.

2.4.2.1 Optical Methods

The optical based instruments can be classified into focus detection instruments and optical interferometric instruments. In both types of instruments, the detected amplitude is limited by the vertical resolution and vertical range of the

instrument while detection of the surface wavelength is restricted by the horizontal resolution and horizontal range.

Several focus detection instruments have been used to obtain 3-D measurements of surface topography. In these instruments, the topography is detected by scanning a focused beam of light over the surface. The surface height is correlated to the light intensity; which, in turn, is related to focus. Some of the techniques include the intensity detection method, confocal method, skew beam method, astigmatic method, and Foucault method. The primary advantage of focal detection techniques over conventional 3-D stylus profilometry is elimination of contact with the specimen. On the other hand, focus detection instruments are noted for the following disadvantages. First, these instruments are highly sensitive to surface inclination. Hence, care must be taken to ensure that specimen are correctly aligned. Second, this instruments can only be used if the machined samples reflect an adequate amount of light, i.e., poor reflectivity of the surface may result in poor data. Third, the interaction of light with impurities on the specimen's surface also complicates interpretation of the results [10].

Optical interferometric instruments, unlike focus detection instruments, operate on the principle of interference of two light incident beams. Specifically, if the crests of two incident beams coincide, the signal intensity is increased (constructive interference). However, if the crest and trough of two incident beams coincide, the overall signal intensity is reduced (destructive interference). In optical interferometric instruments, perfectly flat surfaces result in a series of equally spaced straight line dark and light fringes. Hence, deviations in the surface topography result in changes in the fringe patterns. The primary advantage of these devices is their enhanced vertical resolution over other optical detection based instruments. However,

the primary problem with optical interferometry lies in correlating the fringe patterns to surface topography. One additional problem includes the requirement of the sample to have a constant optical constant for the scanning area. Two of the interference based sensors are the phase shifting interferometric instrument and scanning differential interferometric instrument. Some commercially available optical interferometric instruments include the Micromap 512 optical profilometer and the Wyko RST microscope [10].

Furthermore, recent advancements have resulted in obtaining highly accurate profiles of smooth surfaces using laser profilometry. The basic procedure consists of focusing a laser beam normal to a given surface and then stepping the laser beam in the spatial direction. By integrating the changes in slopes detected by the reflected beam, the profile height at a given location is obtained. However, laser profilometry is noted for several disadvantages. First, the procedure is noticeably more time consuming than other measurement techniques. Care must be taken to ensure that the direction of the incident beam remains unchanged during the entire scanning process. Furthermore, drift of the laser must also be guarded against [10].

2.4.2.2 Non - optical Methods

Several non-optical methods have also been used to characterize surface topography. In fact, subsurface damage has also been assessed using some of these non-optical techniques.

One promising non-optical technique is ultrasonic detection of surface topography. A system has been devised to operate as a scatterometer (to obtain areal surface roughness data) and a profilometer (to obtain 2-D surface data). The basic

setup for such an instrument consists of a transducer connected to an oscilloscope, signal processor, and recording system. The transducer transmits as well as receives pulses for frequencies ranging from 1 to 30 MHz. By monitoring the pulses received by the transducer the surface roughness data is obtained. A distinct advantage is that the setup can operate in wet machining environments where sample is immersed. Furthermore, this technique shows promise as an on-line measurement system [12].

Another non-optical technique to characterize surface damage in machined workpieces uses acoustic emission data. Acoustic emission (AE) is caused by elastic waves, which are generated when a solid material undergoes stress changes. Standard parameters used to quantify the "acoustic emission" signature include the root mean square (RMS) value of the signal, amplitude distribution, kurtosis, and count rate. This technique has been used to monitor crack initiation and propagation in material testing. Recently, acoustic emission has been used monitor surface finish and subsurface damage in ground ceramic workpieces. The basic procedure consists of grinding the workpieces, immersing the samples in a hot water bath, and then recording the acoustic emission intensity. The grinding severity, surface and subsurface damage, is correlated to the acoustic emission count rate [13].

2.4.2.2 Image Processing Methods

Recently, image processing methods and electron microscopy have also been used to evaluate surface finish. In these methods, a picture of the machined surface is obtained first. Next, image processing techniques are used to digitize the picture and to characterize the surface integrity. Overall, electron microscopy can be divided into different categories depending on the type of microscope used. First, in transmission electron microscopy (TEM), an image is obtained by magnifying and focusing

scattered electrons. Resolutions of up to 0.3 nm have been achieved. However, the primary drawback of TEM is the requirement that the sample used be relatively thin (less than 1 μ m thick). Hence, a replicate of the surface needs to be made to ensure that the correct thickness is used. Second, scanning electron microscopy (SEM) has also been used to characterize surface topography. In SEM, a raster image is obtained after a focused beam of electrons scans the surface of the specimen. The primary advantage of scanning electron microscopy over transmission electron microscopy is the non-requirement of thin samples. However, workpieces to be examined need to be conductive. Overall, microdensitometric methods are used to convert electron micrographs into grayscale image data. Based on the grayscale level intensity (0 to 255 levels), height data are obtained. Next, after obtaining spatial and amplitude data, fractals, along with other image enhancement techniques, have been used to reconstruct surface topography [10].

Chapter 3

Surface Integrity Assessment System

3.1 Feasibility Analysis

Due to the brittle behavior of advanced ceramics, careful assessment of surface integrity (damage and finish) of components manufactured from these materials is of utmost importance. To evaluate surface integrity of machined workpieces, a nondestructive surface damage assessment system was designed in this thesis work. In this system, a microdensitometric technique is used to reconstruct surface topography from shadowed scanning electron micrographs. There are several advantages associated with this non-destructive surface integrity assessment system. First, since there is no mechanical contact between the workpiece and the detector, no additional damage is introduced during the assessment process. Second, issues of importance in stylus based profilometry such as the wear of the stylus tip and change in geometry of the detector are not encountered in this system. Third, the assessment of surface integrity can be obtained more rapidly using this system when compared to the traditional assessment techniques discussed in the previous chapter. Overall, the designed surface integrity assessment system is composed of three modules: data acquisition module, data processing module, and machining performance assessment module.

3.2 Data Acquisition Module

In the first module of the system, the machined workpiece is placed in the chamber of an environmental scanning electron microscope to obtain a image of the

surface magnified 500 times (500x). As indicated by Figure 3.1, both x-rays and electrons are emitted within the microscope. However, the image is primarily obtained from the generated electrons. An environmental scanning electron microscope (ESEM) is used instead of a transmission electron microscope (TEM) or conventional scanning electron microscope (SEM) for the following reasons. First, as mentioned in the previous chapter, in order to use a TEM, machined samples have to be relatively thin (less than $1\mu\text{m}$ thick). In practice, replicates of surface have to be made to achieve the required thickness. However, no such height thickness requirement problems are using an ESEM. Second, nonconducting samples do not need to be coated with a thin metallic layer when using the ESEM. In the conventional SEM, images can be obtained only from conducting samples. Hence, any nonconducting sample needs to be coated with a thin layer (5 to 10 nm) of metal. However, coating of the surface may result in additional problems such as thermal damage and surface etching. Thermal damage of the specimen results from the large temperature rise during deposition of the thin metal film. Furthermore, another potentially significant problem during deposition of the metal is surface etching. During the deposition process, metal particles could strike the sample with sufficient force to damage or etch the surface [14].

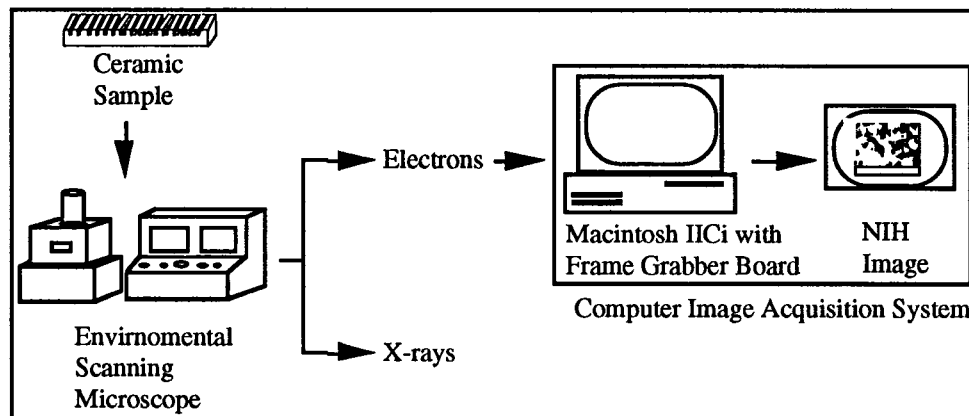


Figure 3.1 Data Acquisition Module

As indicated in Figure 3.2 [14], the typical scanning electron microscope is comprised of several components. First, an electron gun supplies a beam of electron ranging in energy from 1 to 40 keV. Next, the electron beam passes through a vacuum electron column which contains the electron lens. In turn, these lens reduce the diameter of the primary electron beam to create a focused high intensity electron beam. With the aid of scanning coils the focused electron beam is scanned across the surface of the specimen to obtain a raster image. The scanning coils deflect the beam to first strike the surface in a straight line. Then, a scanning circuit steps the beam incrementally until a rectangular raster image of the surface is obtained. Several phenomena such as absorption, radiation, and transmission are observed after the primary incident beam strikes the specimen. However, backscattered electrons and secondary electrons are primarily used to obtain an image of the surface (see Figure 3.8, Section 3.5). Back scattered electrons are electrons from the impinging electron beam which scatter laterally after striking the surface. Since they are deflected with only a slight loss of energy, these electrons can escape be detected if near the surface. However, some of the high energy electrons from the impinging beam penetrate the surface of sample. Once inside the sample, the high energy electrons excite electrons within the sample. The excited electrons which have sufficient energy to escape the surface are known as secondary electrons. The signals from both of these electrons is detected and intensified. Next, the created image is displayed on a cathode ray tube [14].

In terms of operation, the environmental scanning microscope (ESEM) is fairly similar to the conventional scanning electron microscope (SEM). As before, the incident electron beam travels through a vacuum column. However, in the ESEM, the environmental chamber is maintained at pressures ranging from 0.1 to 1 kPa.

Furthermore, depending on the type of material to be examined, different gases are introduced into the environmental chamber. The interactions between the gas molecules and the incident electron beam neutralize surface charging on samples which act as insulators and amplify the output signal. Overall, the different mode of operation of the ESEM provides enhanced depth perception over conventional SEMs. The electron cascade, created from the interaction between secondary electrons and the conducting gas, controls the depth information. In general, the deepest depth information obtainable is proportional to the depth of beam penetration [14].

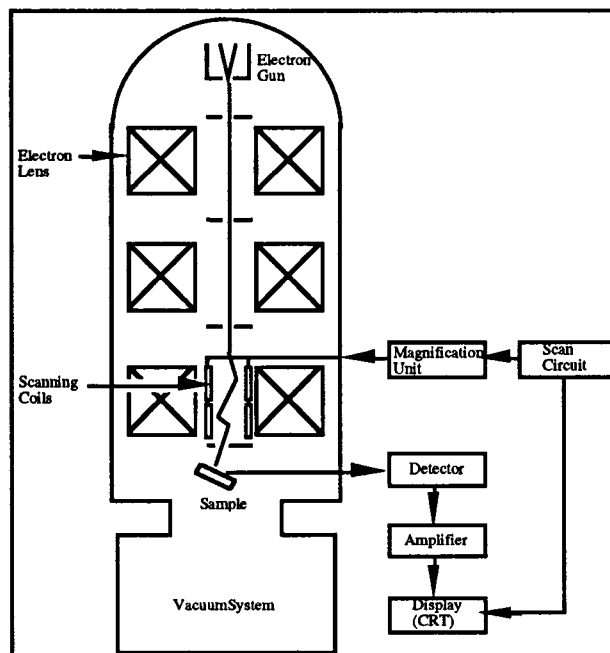


Figure 3.2 Scanning Electron Microscope

The image displayed on cathode ray tube of the ESEM is also directly observed on a computer equipped with video capturing capabilities. As shown in previously in Figure 3.1, a Macintosh IIfx model computer equipped with a QuickCapture frame grabber hardware board and NIH Image software is used to capture the image in real time. This computer system, which allows direct acquisition

of the image, ensures that additional errors obtained during scanning of electron micrographs are minimized. Furthermore, once captured, the image can be converted into numeric format data (gray levels ranging from 0 to 255) to allow further manipulation.

3.3 Data Processing Module

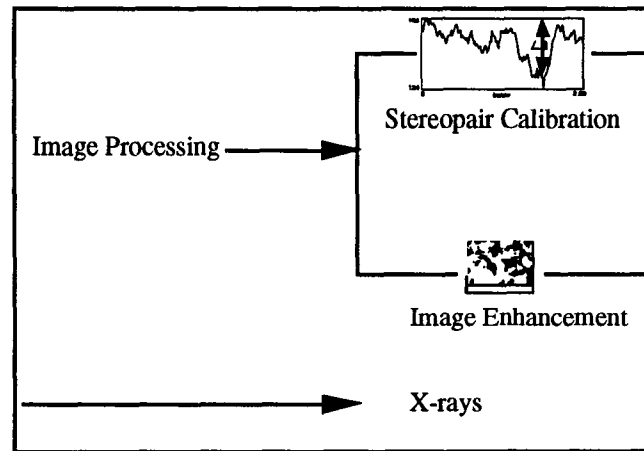


Figure 3.3 Data Processing Module

Next, as shown in Figure 3.3, after acquisition of the image data, image processing techniques are used to enhance the image and calibrate the height (amplitude) data. First, the image is enhanced ("smoothed") to remove noise from the signal. Second, stereopair calibration is used to quantify the height data. Traditionally, this technique consists of adding parallax to the two dimensional (2-D) image to obtain a three dimensional (3-D) image. As illustrated in Figure 3.4 [10], parallax is the difference between two points from different tilt angles. In the figure, the parallax is calculated from the projections of a tilted object. First, if the object is tilted by $\alpha/2$ degrees, then the projection Δx^+ is obtained. However, if the object is tilted by $-\alpha/2$ degrees, then the projection Δx^- is obtained. The parallax is the difference between the two projections (see Equation 3.1). Next, after the parallax

has been calculated the object's height is determined using Equation 3.2 [10]. In this thesis, a modified version of this technique is used to determine the height data. This model is described in detail in Chapter 5.

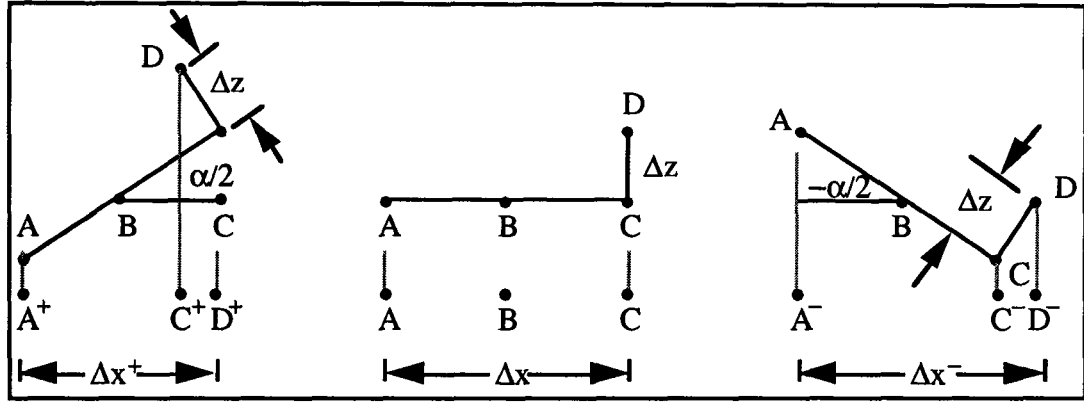


Figure 3.4 Stereopair Calibration

$$\begin{aligned}\Delta p &= \Delta x^- - \Delta x^+ \\ &= \left(\Delta x \cos\left(\frac{\alpha}{2}\right) + \Delta z \sin\left(\frac{\alpha}{2}\right) \right) - \left(\Delta x \cos\left(\frac{\alpha}{2}\right) - \Delta z \sin\left(\frac{\alpha}{2}\right) \right)\end{aligned}\quad (3.1)$$

where Δp represents the parallax between two projections

Δx represents the undistorted distance between two points

Δz represents the undistorted height between two points

α represents the tilt angle

$$\Delta z = \frac{\Delta p}{2M \sin \frac{\alpha}{2}} \quad (3.2)$$

where Δz represents the undistorted height between two points

Δp represents the parallax between two projections

M represents the magnification factor

α represents the tilt angle

3.4 Machining Performance Assessment Module

After the image has been enhanced and calibrated in the image processing module, several parameters are used to characterize the machining performance (see Figure 3.5). The numeric data file representing the 256 graylevels is manipulated using mathematical software (MATLAB version 4.2a). With the aid of MATLAB, the surface is reconstructed. The orientation this 3-D image may be easily manipulated to observe the surface topography from different perspectives. Furthermore, area based surface integrity parameters such as roughness average and root mean square roughness are obtained. These parameters provide a more realistic assessment of surface integrity their conventional 2-D counterparts. Furthermore, contour plots at different elevations are created to observe directionality as a function of height.

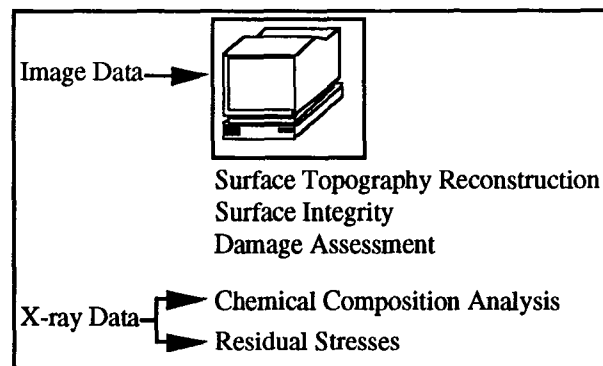


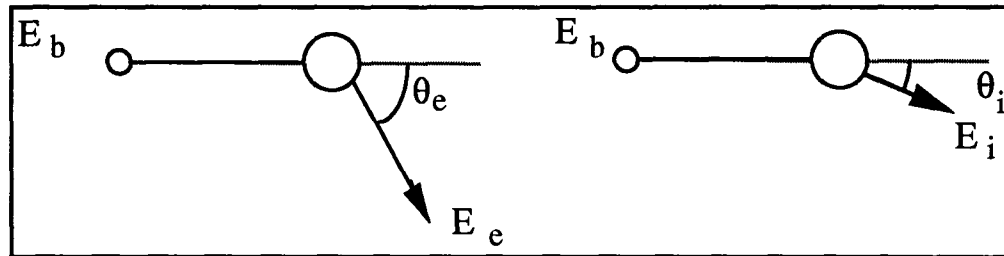
Figure 3.5 Damage Assessment Module

Based on the image file, non destructive evaluation of surface integrity is

obtained. The surface finish and surface damage can be quantified with the aid of the aforementioned area based surface integrity parameters. Furthermore, x-rays generated from the electron - workpiece interactions are used to evaluate residual stresses. Furthermore, chemical composition analysis is also performed.

3.5 Concerns of ESEM

Several phenomena result after the incident electron beam strikes the surface of the specimen. When electrons from the incident beam strike the sample, both elastic and inelastic scattering occur. Scattering changes both the trajectory (or path) and energy content of the incident electron. As shown in Figure 3.6a [14], elastic scattering results in a significant change in trajectory with minimal change in energy. Most electrons which undergo elastic scattering deflect relatively close to the surface of the sample and are easily detected. These electrons, known as backscattered electrons (BSE), contribute to the overall obtained image.



(a) Elastic Scattering

(b) Inelastic Scattering

Figure 3.6 Scattering Events

Inelastic scattering, on the other hand, results in a significant reduction of energy in the incident electron with minimal change in the electron's trajectory (see Figure 3.6b [14]). However, unlike elastic scattering, inelastic scattering results in

numerous other phenomena. First, the high energy incident electron may transfer energy to electrons located within the material. These electrons within the material, known as secondary electrons, may acquire sufficient energy to escape the surface and hence be detected. Other byproducts of inelastic scattering are Auger electrons, x rays, lattice vibrations, electron oscillations, and electromagnetic radiation. These phenomena are of primary importance in analyses of the chemical composition of the specimen and of residual stresses. Overall, the these phenomena and areas of their application in signal formation and chemical analysis are summarized in Figure 3.7 [14].

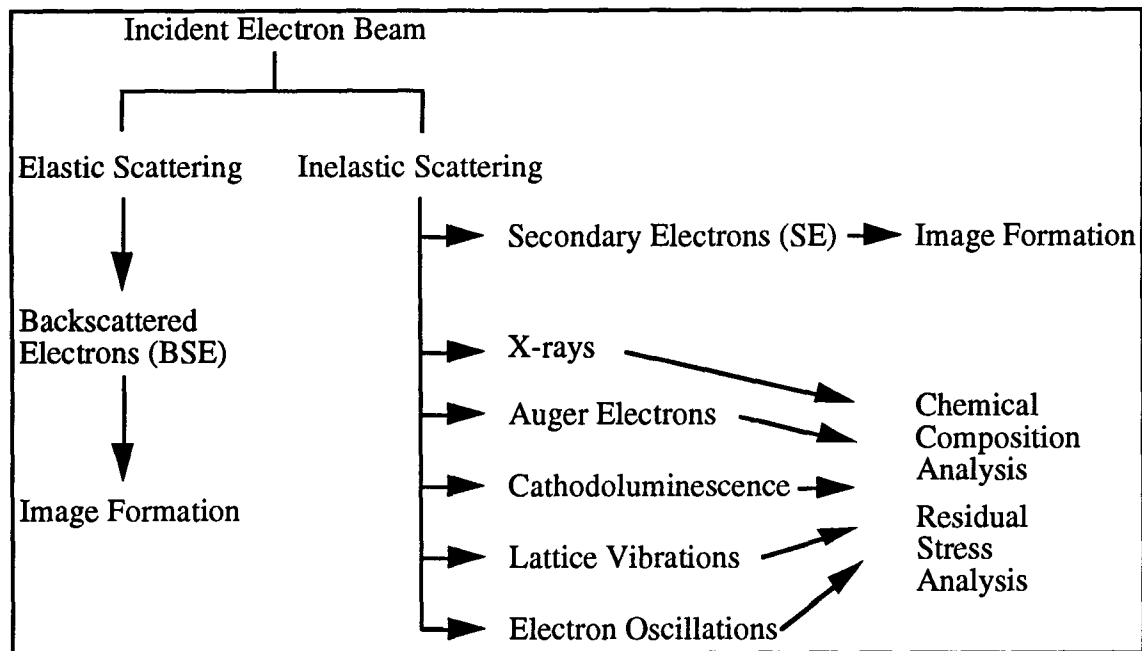


Figure 3.7 Electron Interactions

Backscattered electrons (BSE) and secondary electrons (SE), as shown in Figure 3.7, are used to form an image of the surface. Hence, the topographic contrast and brightness of the image is primarily dependent on these two types of electrons.

Issues such as the depth of electron penetration within a material and the escape depth of secondary electrons need to be addressed. First, the interaction volume, volume of material below the surface within which electron interactions occur, is a parameter used to characterize the range of electron penetration. The overall shape of certain low atomic number elements has been experimentally verified. For example, by observing polymethylmethacrylate (PMMA) under different etching rates, the shape of the interaction volume was observed to be that of a pear . Furthermore, the interaction volume, shown in Figure 3.8, is usually characterized by the depth of penetration of the incident electron beam. Furthermore, the escape depth is noted as the depth below which electrons do not have sufficient energy to escape. The three parameters are significantly affected by other factors such as the atomic number, energy of incident electron beam, and surface inclination of the specimen [14].

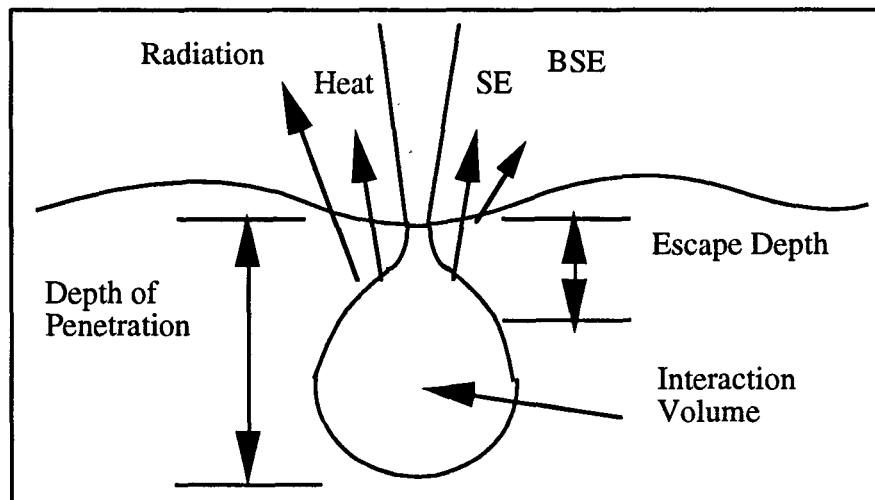


Figure 3.8 Interaction Volume

In materials with intermediate to high atomic numbers, direct observation of the interaction volume, depth of penetration and escape depth is not possible. Hence, simulation techniques have to be used to estimate the aforementioned parameters.

Monte Carlo simulation has been used to predict the penetration depth and escape depth of electrons in different materials. These simulation techniques calculate the trajectories of individual electrons using a random number generator and an energy loss model.

Two estimates of the depth of penetration are the Bethe range and the Kanaya-Okayama range. First, the Bethe range is based on the rate of energy loss as a function of distance traveled (dE/dS). As indicated in Equation 3.3 [14], the Bethe range is an estimate of the total distance an electron travels within the specimen until the electron has lost all its energy. Since the loss of energy due to elastic scattering is unaccounted for in this model, the range is usually overestimated. Overall, the inaccuracy of the estimate becomes more prominent as the atomic number increases.

$$R_B = \int_{E_0}^{E=0} \frac{1}{dE/dS} dE \quad (3.3)$$

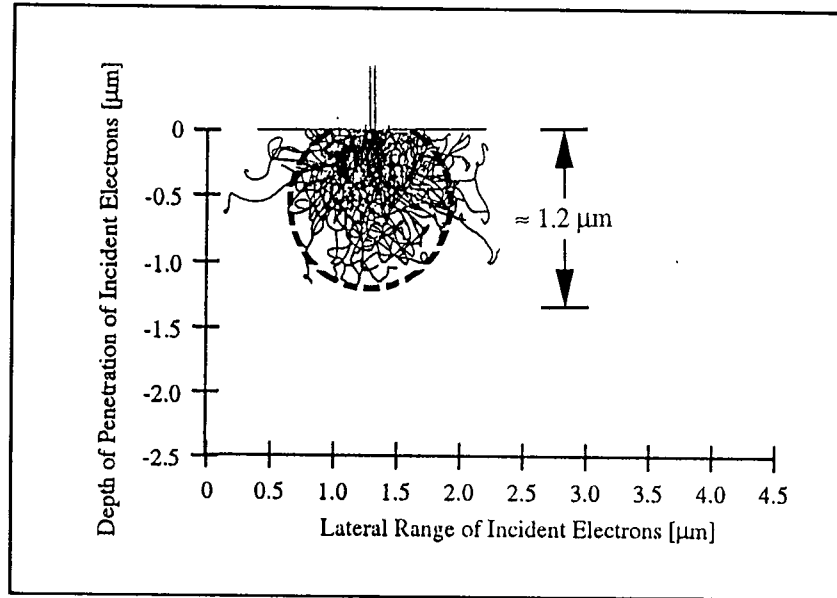
On the other hand, the Kanaya-Okayama model provides a more accurate estimate of the electron range since it accounts for energy losses due to both elastic and inelastic scattering. In this model, Equation 3.4 [14], parameter A represents the atomic weight in g/mole, parameter Z represents the atomic number, parameter E_0 represents the energy of the incident beam in keV, and parameter ρ represents the density in g/cm³.

$$R_{ko} = \frac{0.0276 A E_0^{1.67}}{Z^{0.89} \rho} \quad (3.4)$$

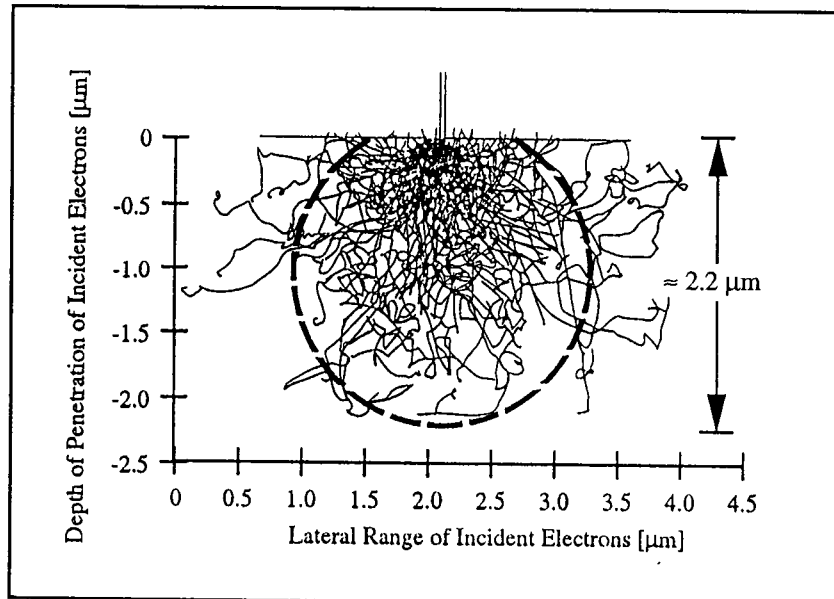
As previously mentioned, several variables influence the depth of penetration of an incident electron. First, the energy of the incident beam plays a significant role

in the penetration depth. Comparing Figures 3.9a and 3.9b [14], an increase in the energy of incident electrons results in a corresponding increase in the depth of penetration and lateral range of these electrons. In these figures, the dotted circle represents the maximum depth of penetration and lateral range of incident electrons. First, in Figure 3.9a, when the energy of incident electrons is $E_0 = 20$ keV, the maximum depth of penetration and the maximum lateral range of these electrons in iron is about $1.2\ \mu\text{m}$, respectively. On the other hand, as shown in Figure 3.9b, when the energy of incident electrons is increased to $E_0 = 30$ keV, the maximum depth of penetration and maximum lateral range are increased to about $2.2\ \mu\text{m}$, respectively. Overall, as the energy increases, the depth of penetration increases because fewer electrons are scattered elastically. This occurs because the trajectories of the incident beam near the surface become straighter as the energy increases.

Second, as illustrated in Figure 3.10 [14], the atomic number also plays a critical role in the interaction volume of incident electrons. Two parameters which are primarily used to characterize the interaction volume are the maximum depth of penetration and the lateral range of incident electrons. In general, an increase in atomic number results in a corresponding decrease in the depth of penetration and lateral range since incident electrons are elastically deflected more readily as the atomic number increases. In Figure 3.10a, the maximum depth of penetration of incident and lateral range electrons in an iron target (atomic number $Z = 26$) are about $1.2\ \mu\text{m}$, for an incident electron energy of $E_0 = 20$ keV. However the maximum depth of penetration of incident electrons and lateral range are reduced to about $0.65\ \mu\text{m}$ in a uranium target (atomic number $Z = 92$) for an incident electron energy of $E_0 = 20$ keV.

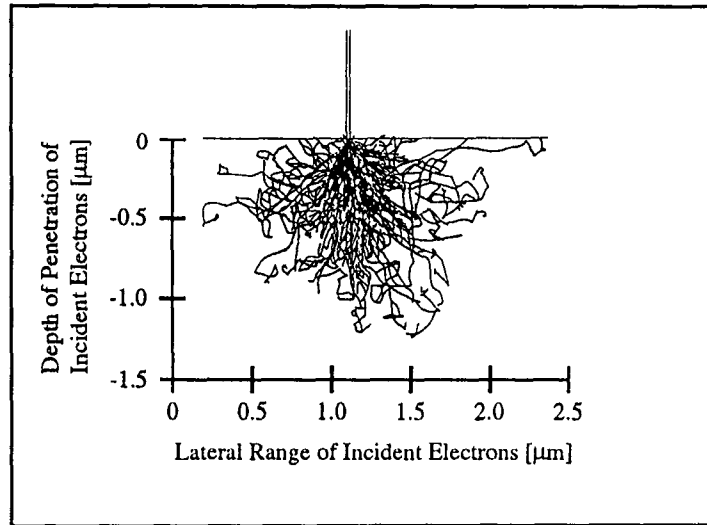


(a) Incident Energy $E_0 = 20$ keV

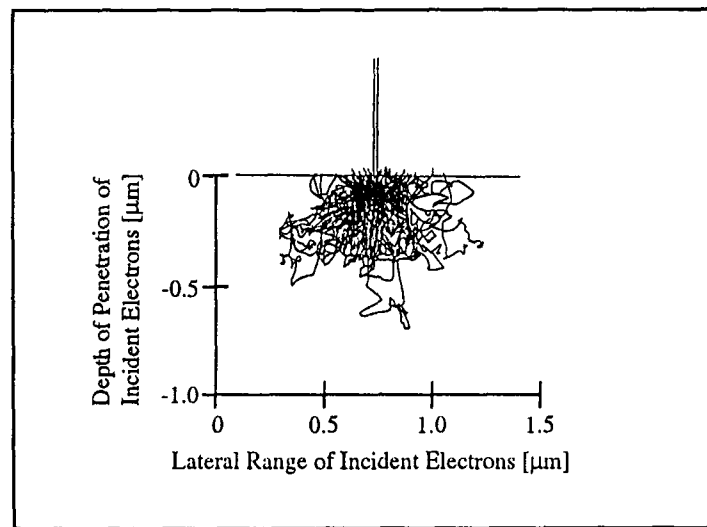


(b) Incident Energy $E_0 = 30$ keV

Figure 3.9 Interaction Volume vs Incident Energy in Iron, Tilt Angle = 0°



(a) Material = Iron, $Z = 26$

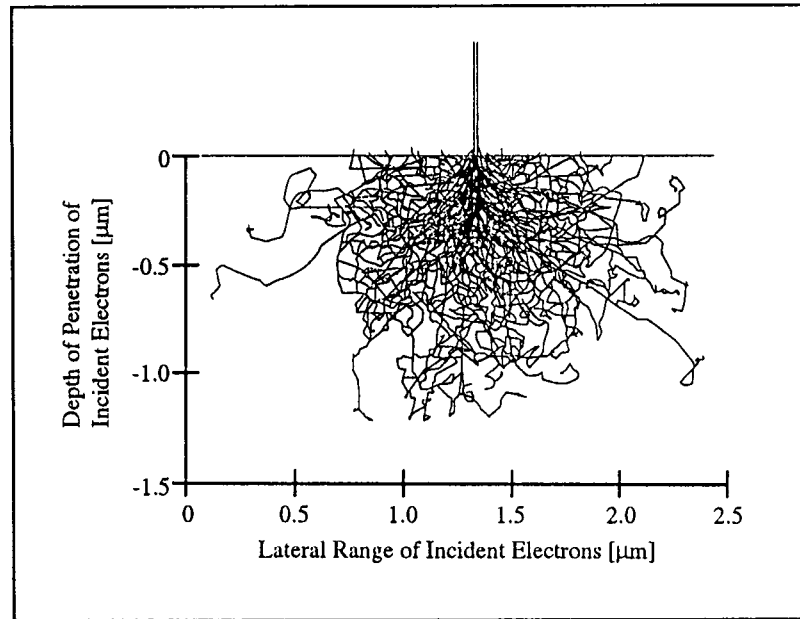


(b) Material = Uranium, $Z = 92$

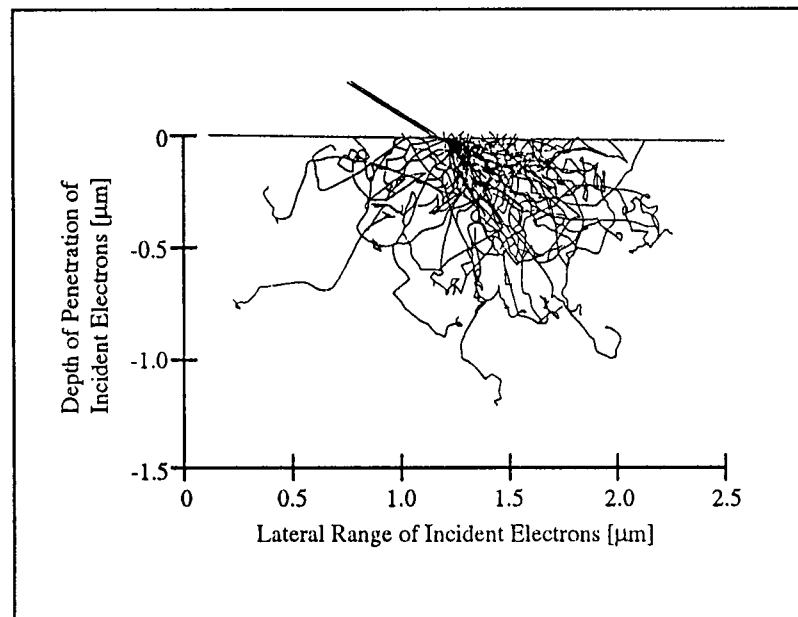
Figure 3.10 Interaction Volume vs. Material, $E_0 = 20$ keV, Tilt Angle = 0°

Third, as illustrated in Figure 3.11 [14], the depth of penetration decreases as the angle of tilt between the incident beam and surface increases. In Figure 3.11a, electron trajectories within the interaction volume of an iron target are plotted for a tilt angle of 0° and an incident beam energy of $E_0 = 20$ keV. If the extreme electron trajectories are ignored, the maximum depth of penetration is about $1.0\text{ }\mu\text{m}$. On the other hand, electron trajectories within the same iron target are plotted for a tilt angle of 60° and an incident beam energy of $E_0 = 20$ keV. For this tilt angle, the maximum depth of penetration is about $0.5\text{ }\mu\text{m}$ if extreme electron trajectories are ignored. Overall, the shape of the interaction volume becomes more asymmetric as the angle of tilt is increased. In turn, the depth of penetration within the target is reduced.

In this thesis, an analytical investigation of the interaction volume, depth of penetration and escape depth is provided in Chapter 4. In Chapter 4, the role of material dependent properties such as atomic mass and density, and physical parameters such as energy of incident electron beam, beam diameter, and tilt angle, and thickness of the sample on depth of penetration will be examined.



(a) Tilt Angle = 0°



(b) Tilt Angle = 60°

Figure 3.11 Interaction Volume vs Tilt Angle in Iron, $E_0 = 20$ keV

Chapter 4

Depth Sensitivity Analysis

4.1 Introduction

In Chapter 3, the significance of the two types of electrons critical in the image formation process was described. First, backscattered electrons, which undergo elastic scattering, retain most of their incident energy and are readily detected in the environmental scanning electron microscope (ESEM). On the other hand, secondary electrons result from inelastic scattering events, in which incident electrons retain their trajectories but lose most of their energy. In addition, secondary electrons are derived from within the material. These electrons have acquired a certain amount of energy from some of the incident electrons which have penetrated into the material. In turn, those electrons which have an adequate amount of energy escape from the surface. Since the proposed surface integrity assessment system relies heavily on the ESEM for surface topography reconstruction, the depth sensitivity of electrons involved in the image formation process is of crucial importance.

Three parameters which have been traditionally used to characterize the range of electrons within a bulk material are the depth of penetration, interaction volume, and escape depth. As shown in Figure 4.1 (and Figure 3.8), the three parameters provide estimates of the horizontal and vertical range of electrons within the target material. First, the depth of penetration provides an estimate of the total vertical range of incident electrons. On the other hand, the interaction volume provides an estimate of both the vertical and horizontal ranges of incident electrons. Finally, the last, but not the least significant parameter is the escape depth of incident electrons. The

escape depth is defined as the vertical range within which incident electrons have enough energy to escape. In general, the escape depth is only a fraction of the depth of penetration.

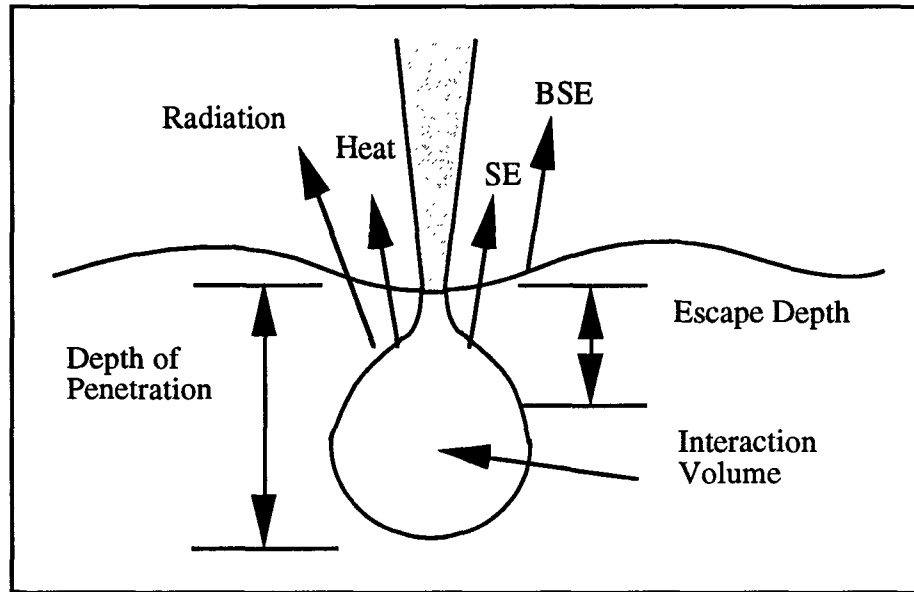


Figure 4.1 Depth Estimation Parameters

4.2 Monte Carlo Model

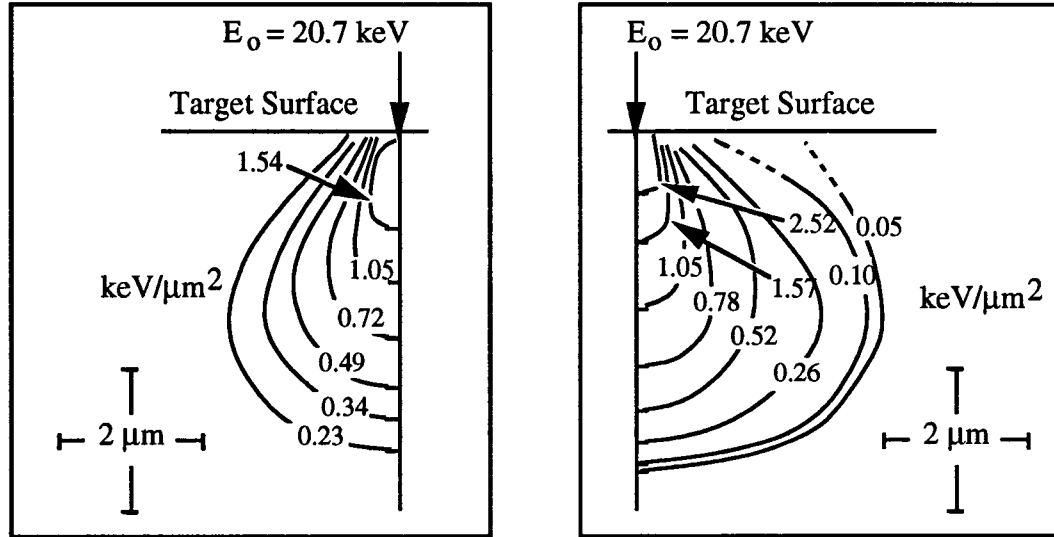
In certain materials, the interaction volume, depth of penetration, and escape depth of incident electrons within a target have been experimentally verified. One example of such a material in which the shape of the interaction volume has been directly observed is polymethylmethacrylate (PMMA). This particular material deforms when it is chemically etched after the material has undergone electron irradiation. Since the etching rate is dependent on the electron dose (e^- / cm^3), the shape of the interaction volume can be experimentally observed in a progressive manner by increasing the length of etching periods. Overall, as shown in Figure 4.2a, the interaction volume is estimated from the energy deposition contours, which are, in turn, controlled by the etching rate. Furthermore, the units of the contours are

keV/ μm^2 because the figure represents a two dimensional projection of the interaction volume. However, the interaction volume and depth of penetration cannot be directly observed in many conventional engineering materials since these materials do not indicate an observable deformation after electron irradiation. Hence, Monte Carlo simulation models are use to obtain theoretical estimates of the interaction volume, depth of penetration, and escape depth of incident electrons. Furthermore, these models are also used to track the trajectories of incoming electrons which strike the surface of the target. Overall, the term "Monte Carlo" is used to describe the use of random numbers to select individual parameters in the model. In particular, random number generators are extensively used to determine if an electron is backscatters or results in the generation of secondary electrons.

As shown in Figures 4.2a and 4.2b [15], experimental and theoretical data for experiments performed by Everhart in 1972 indicate good correlation for the PMMA material. First, in Figure 4.2a, experimental data for incident electrons with an initial energy of $E_0 = 20$ keV are plotted to visually observe the interaction volume. Specifically, the damage induced by etching for different time periods allowed direct visualization of the interaction volume. Overall, as observed in Figure 4.2a, the energy disposition level decreases from 1.54 keV/ μm^2 at a depth of about 1.33 μm to 0.23 keV/ μm^2 at a depth of 4.41 μm .

Next, to determine the accuracy of Monte Carlo simulation programs in determining the interaction volume, depth of penetration, and escape depth of incident electrons, Everhart compared simulation data, shown in Figure 4.2b, with the collected experimental data, shown in Figure 4.2a. In general, the simulation data obtained from a Monte Carlo simulation program are fairly representative of the experimental data [14]. For example, for the simulation data, the energy deposition level decrease

from $1.57 \text{ keV} / \mu\text{m}^2$ at a depth of about $1.47 \mu\text{m}$ to $0.05 \text{ keV} / \mu\text{m}^2$ at a depth of about $4.72 \mu\text{m}$. Overall, the accuracy of the model is reduced as the distance beneath the surface increases, i.e., at large distances from the free surface, the interaction volume and other parameters may not be extremely accurate.



(a) Experimental Data

(b) Theoretical Monte Carlo Data

Figure 4.2 Experimental Verification of Monte Carlo Simulation

In this thesis, a Monte Carlo Program developed by Newbury and Myklebust at the National Institute of Standards and Technology (NIST) is used to theoretically determine the role of certain parameters on the escape depth of backscattered electrons in a flat bulk surface. In the NIST Microanalysis Monte Carlo Electron Trajectory Simulation Program (NIST Micro MC) program [15, 16], inelastic and elastic scattering events are treated separately.

4.2.1 Inelastic Scattering

As previously discussed, an incident electron can scatter elastically (large change in trajectory with a small loss of energy) or inelastically (small change in trajectory with a large loss of energy). For inelastic scattering, the total depth of penetration of electrons within the target is given by the Bethe range R which is calculated using Equation 4.1 [16]. This range estimates the total distance traveled by an electron until complete loss of energy is achieved. The quantity dE/ds appearing in the Bethe range equation or the rate of energy loss per distance traveled is given by Equation 4.2 [16]. Overall, the energy loss model represented in the Bethe range is also extensively used to describe the energy loss for elastic scattering events.

$$R = \int_{1.03J}^{E_0} \left(-\frac{1}{dE/ds} \right) dE \quad (4.1)$$

where R represents the Bethe

E_0 represents the energy of the incident electron beam (keV)

dE/ds represents the energy loss per distance traveled ((keV· cm²) / g)

J represents the mean ionization energy (keV)

$$\frac{dE}{ds} = \frac{-78500 Z}{EA} \ln \left(\frac{1.66 E}{J} \right) \quad (4.2)$$

where dE/ds represents the energy loss per distance traveled ((keV· cm²) / g)

E represents the instantaneous energy (keV)

A represents the atomic weight (g/mol)

Z represents the atomic number

J represents the mean ionization energy (keV)

The mean ionization energy appearing in Equations 4.1 and 4.2, respectively, can be calculated using the Berger-Seltzer equation (Equation 4.3 [16]) or the Duncumb equation (Equation 4.4 [16]).

$$J = 9.76Z + 58.5Z^{-0.19} \quad (4.3)$$

where J represents the mean ionization energy (keV)

Z represents the atomic number

$$J = Z \left[14(1 - e^{-0.1Z}) + \frac{75.5}{Z^{(Z/7.5)}} - \frac{Z}{100 + Z} \right] \quad (4.4)$$

where J represents the mean ionization energy (keV)

Z represents the atomic number

4.2.2 Elastic Scattering

Elastic scattering events, unlike inelastic scattering events, involve more parameters since both energy loss and changes in trajectory need to be accounted for. As previously stated, in general, the Bethe range is still used to estimate the energy loss per distance traveled. In addition, for elastic scattering, the screened Rutherford cross section, given by Equation 4.5 [15] is used to describe the scattering events within the target. This equation is defined as the probability of a scattering event occurring with a scattering angle greater than given angle.

$$Q(> \phi_o) = 1.62(10^{-20}) \frac{Z^2}{E^2} \cot^2 \frac{\phi_o}{2} \quad (4.5)$$

where $Q(>\phi_o)$ represents the probability of a scattering event with a scattering angle greater than ϕ_o

Z represents the atomic number

E represents the instantaneous energy (keV)

ϕ_0 represents a given angle

Furthermore, since the Bethe range only accounts for inelastic scattering events, the maximum depth of penetration dimension of the interaction volume will be overestimated with this range. To obtain a realistic estimate of the depth of penetration which accounts for both the effects of both elastic and inelastic scattering events, the Kanaya - Okayama range R_{KO} (Equation 4.6 [15]) is used.

$$R_{KO} = \frac{0.0276AE_0^{1.67}}{Z^{0.89}\rho} \quad (4.6)$$

where R_{KO} represents the Kanaya - Okayama Range

A represents the atomic weight (g/mole)

E_0 represents the initial energy of incident electrons

Z represents the atomic number

ρ represents the density (g/cm³)

The energy loss between scattering events is assumed to be continuous and is given by the Bethe range (Equation 4.1). However, the energy at the end of a scattering event depends on the energy at the beginning of the scattering event and the mean free path. In turn, the mean free path, defined as average distance traveled between scattering events, is given by Equation 4.7 [15]. Overall, the total mean free path for all events is calculated using Equation 4.8 [15].

$$\lambda = \frac{A}{N_o\rho Q} \quad (4.7)$$

where λ represents the mean free path for a given scattering event

A represents the atomic weight (g/mole)

E_0 represents the energy of the incident electron beam (keV)

Z represents the atomic number

ρ represents the density (g/cm^3)

$$\frac{1}{\lambda_{total}} = \frac{1}{\lambda_a} + \frac{1}{\lambda_b} + \frac{1}{\lambda_c} + \dots \quad (4.8)$$

where λ_{total} represents the mean free path for all the scattering events

λ_i represents the mean free path for scattering event i ($i = a, b, c, \dots$)

4.2.3 Program Flowchart

Figure 4.3 is a flowchart of the program code describing the input parameters, the energy loss model, and the generated output table. Overall, the NIST Monte Carlo program can be divided into three subprograms. The first subprogram deals with the input information provided by the user. In this section, information about the material, beam properties, and geometry of the give structure are input. After specifying the desired unit of distance (Angstroms, micrometers, centimeters, or inches), the appropriate geometry is selected. Two possible configurations exist: embedded structure in film or film on substrate. In this following sections, the electron penetration within a flat bulk surface was examined by selecting the film on substrate option and setting the film thickness to an arbitrarily large value ($5000 \mu\text{m}$). Next, information about the material properties are input. The number of elements in the compound of interest , the atomic numbers of these elements, and the weight fractions of the elements are entered. The same steps are repeated for the substrate material. Next, input parameters corresponding to the beam properties are entered. The initial energy of beam is input along with direction cosines among the beam, specimen and detector. In addition, the beam diameter and the intensity distribution (homogeneous

or Gaussian) are input. The final inputs are an arbitrary odd positive integer between 0 and 32767 which serves as the seed for the random number generator and the total number of electron trajectories to simulate.

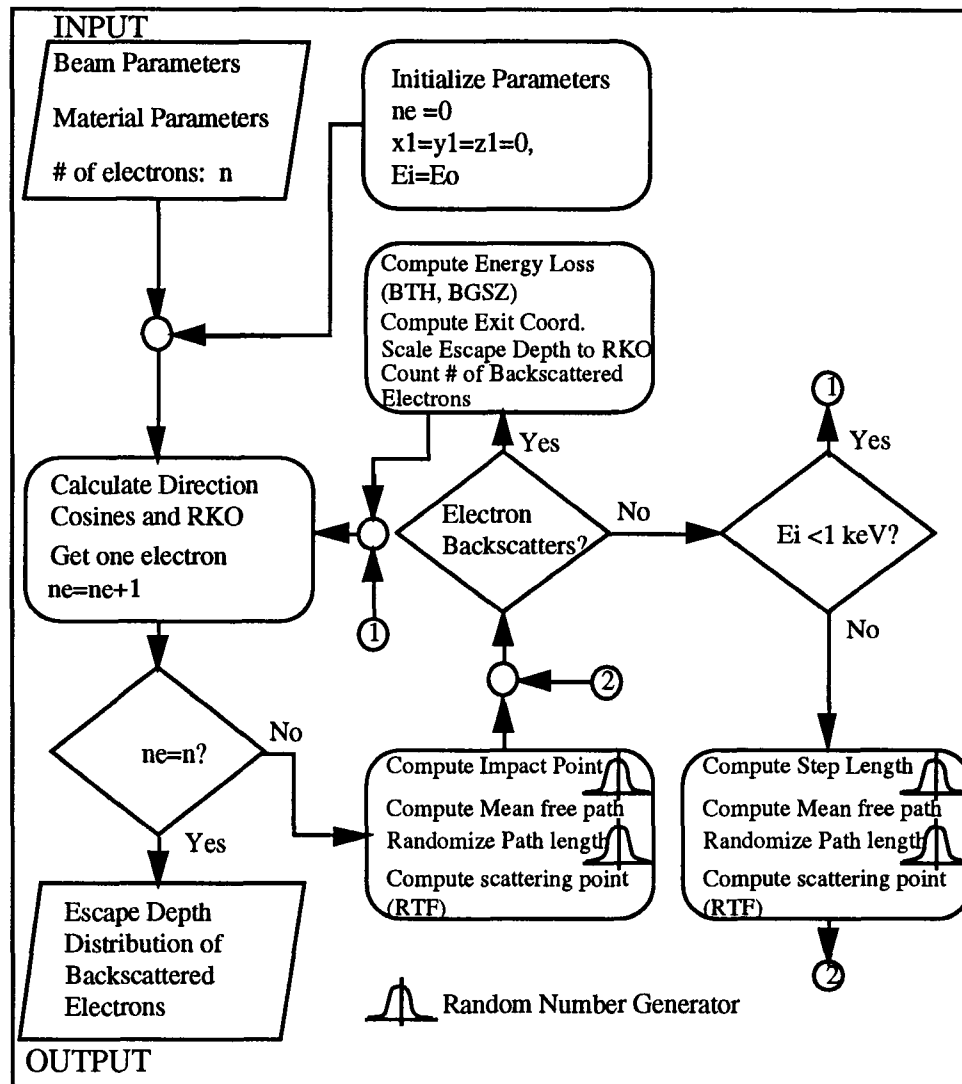


Figure 4.3 Flowchart of Monte Carlo Program

The second subprogram computes the trajectories for all of the input electrons. First, the pertinent coordinate points are initialized and the Kanaya-Okayama penetration depth for backscattered electrons is calculated. This range is scaled into

100 histogram bins of equal thickness. Next, the first electron is selected. A counter is used to ascertain if the total number of input parameters have been simulated. If the total number of electrons have not been simulated, the impact point is calculated using a random number generator. Next, after the mean free path is calculated and randomized, the Rutherford cross section is used to compute the coordinates of the scattered point are determined. Then, a test for backscattered electrons is performed. If the electron backscatters, the depth penetration of the electron is scaled according to the calculated Kanaya-Okayama Range and recorded. Furthermore, the energy loss, based on the Berger - Seltzer equation, and exit coordinates of the electron are also recorded. The trajectory for a new electron is then selected. On the other hand, if the electron does not backscatter, a test is performed to examine if the electron retains enough energy to undergo another scattering event. If the electron does not retain an adequate, a new electron trajectory is simulated. However, if the electron does retain enough energy, the step length is computed. Next, the mean free path is calculated and randomized. The coordinates of scattered point are then computed. The test for backscattered electrons is performed again. The aforementioned steps are performed until all of the electron trajectories have been simulated.

The third subprogram -- output file -- is executed after all the electron trajectories have been simulated. In this thesis, the output of interest is the distribution of backscattered electrons as a function of escape depth. As previously mentioned, the escape depth is scaled according to the Kanaya - Okayama Range, since the parameter accounts for both elastic and inelastic scattering. the direction cosines have been calculated, a test for backscattering is performed. If an electron backscatters, the energy loss is computed using the Bethe and Berger-Seltzer equations. In addition, a counter determines the total number of backscattered electrons and the number of electrons from the predetermined histogram bin. Next, a new electron is selected and

the same procedural steps are followed. If, on the other hand, the electron does not backscatter, the energy of the electron is determined. The total mean free path is calculated and randomized. Next, the coordinates of the next scattering point are determined. Based on these coordinates, a test for backscattering is again performed. The aforementioned steps are performed until the interactions of all of the electrons have been individually examined. Finally, a table of the escape depth of backscattered electrons is obtained from the output file.

4.3 Analytical Electron Microscopy

Several factors; such as beam energy, beam diameter, distribution of electrons within the incident beam, atomic number, and specimen angle of tilt, effect the depth of penetration and escape depth of electrons within the target material. A two level three factor (2^3) factorial design experiment is performed to quantify the pertinent main effects and interaction among three selected variables [5]. The three factors of interest denoted as factors A, B, and C, respectively, are material, energy of incident electron beam, and the specimen angle of tilt. The specimen geometry considered is a flat bulk surface, i.e., a closed flat surface as shown in Figure 4.4.

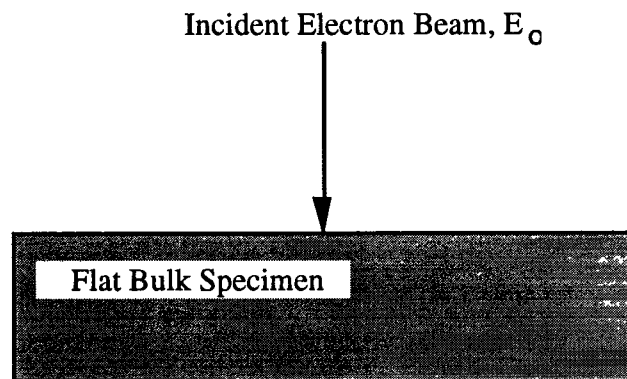


Figure 4.4 Test Surface

The selected materials for the low and high levels of the experiment are DICOR/MGC glass ceramic and alumina. Currently, DICOR/MGC glass ceramics are used in dental applications. Specifically, the feasibility of using this ceramic as dental restorative material is being examined since the physical and mechanical properties including density, hardness, thermal conductivity, translucency, and refractive index, of this ceramic closely resemble the properties of human enamel. The density of DICOR/MGC glass ceramic material is $\rho = 2.8 \text{ g/cm}^3$. Overall, DICOR/MGC glass ceramic is a tetrasilicic glass ceramic whose chemical formula is given by $\text{K}_2\text{O-MgF}_2\text{-MgO-SiO}_2$ [4]. The other material selected for analysis, alumina (Al_2O_3), is primarily used in high temperature applications. For example, crucible components to contain molten metal are constructed from alumina. Furthermore, some of the other applications of alumina include its use in spark plugs, orthopedic implants, and bone filler. The density of alumina is $\rho = 3.98 \text{ g/cm}^3$ [2]. The pertinent weight fractions of the elements in these two compounds is listed in Table 4.1.

Table 4.1 Weight Fractions of Dicor and Alumina

Dicor			Alumina		
Element	Atomic Number	Weight Fraction	Element	Atomic Number	Weight Fraction
F	9	0.143	Al	13	0.520
K	19	0.302	O	8	0.480
Mg	12	0.190			
O	8	0.254			
Si	14	0.111			

The other two factors in the experiment are energy of the incident electron beam in keV (factor B) and specimen angle of tilt (factor c). The selected low and high levels for factor B are 10 keV and 30 keV, respectively. Likewise, the selected low and high levels of factor C are 0° (normal incidence between beam and target) and 45° (45° between incident electron beam and target). Overall the high and low levels for the three variables are summarized in Table 4.2. In the table, the high level of a given factor is denoted by the lower case letter corresponding to that factor, i.e., the high level of factor A is denoted by a, and so forth. The low level of a factor is denoted as 1. The output parameter used to generate the pertinent model is the maximum escape depth of backscattered electrons, i.e., the deepest penetration an electron can achieve in a closed flat bulk surface and still acquire enough energy to escape.

Table 4.2 Levels for 2³ Factorial Experiment

Factor	Low Level	High Level
A (g/cm ³)	2.8 (1)	3.98 (a)
B (keV)	10 (1)	30 (b)
C (Degrees)	0° (1)	45° (c)

The parameters which remained unchanged during the trials are listed in Table 4.3. In addition, a design matrix containing the settings for each of the eight unique tests along with obtained results is provided in Table 4.4. Note that two replicates were obtained for each unique test for a total sixteen tests. As previously mentioned, the output parameter of the experiment is the maximum escape depth of backscattered electrons. Distributions of the escape depth of backscattered electrons corresponding to the unique tests are also discussed.

Table 4.3 Constant Test Parameters

Geometry	Film on substrate
Thickness of Film	5000 μm
Angle between Impinging Electron Beam and Detector	20 °
Azimuthal Angle between Detector and x-axis	0
Beam Cross Section	Gaussian
Beam Diameter	0.01 μm
Number of Electron Trajectories	10,000
Minimum Energy for Secondary Electron Generation	2 keV

As previously mentioned in this experiment, a total of sixteen tests were performed. Overall, there are three variables of interest ($k = 3$) and two replicates for each unique test combination ($n=2$).

Table 4.4 Design Matrix for 2^3 Factorial Experiment

Treatment	Effects								Maximum Escape Depth [μm]		
Combinations	I	A	B	AB	C	AC	BC	ABC	Trial 1	Trial 2	Sum
(1)	+1	-1	-1	+1	-1	+1	+1	-1	0.463	0.488	0.950
a	+1	+1	-1	-1	-1	-1	+1	+1	0.311	0.319	0.630
b	+1	-1	+1	-1	-1	+1	-1	+1	3.211	3.211	6.422
ab	+1	+1	+1	+1	-1	-1	-1	-1	2.109	2.217	4.326
c	+1	-1	-1	+1	+1	-1	-1	+1	0.450	0.438	0.888
ac	+1	+1	-1	-1	+1	+1	-1	-1	0.311	0.294	0.604
bc	+1	-1	+1	-1	+1	-1	+1	-1	2.820	2.898	5.718
abc	+1	+1	+1	+1	+1	+1	+1	+1	1.947	1.948	3.895

The orthogonal products for each of the effects are defined as the summation of the terms obtained by the product of signs +1 and -1 and the sum of the two trials. For example, the orthogonal product for the main effect of factor A (material) is calculated as shown in Equation 4.7 [5]. Next, the effects and the sum of squares for the effects are determined from the products as shown in Equations 4.8 and 4.9 [5], respectively.

$$Product_A = - (1) + a - b + ab - c + ac - bc + abc = -4.523 \quad (4.7)$$

$$Effect = \frac{Product}{n2^{k-1}} \quad (4.8)$$

where n represents the number of replicated runs

k represents the number of variables

$$SS = \frac{(Product)^2}{n2^k} \quad (4.9)$$

where n represents the number of replicated runs

k represents the number of variables

Based on the equations above, the appropriate products, effects and sum of squares are summarized in Table 4.5. Next, an analysis of variance (ANOVA) summary table, Table 4.6, is created to determine the statistical significance of the main and interaction effects. The significance level used in the hypothesis test is $\alpha = 0.10$. In Table 4.6, any effect with a probability lower than the significance level is deemed significant.

Table 4.5 Products, Effects and Sum of Squares

	Product	Effect	Sum of Squares
A	-4.523	-0.565	1.279
B	17.288	2.161	18.679
C	-1.224	-0.153	0.094
AB	-3.316	-0.414	0.687
AC	0.311	0.039	0.006
BC	-1.047	-0.131	0.069
ABC	0.237	0.030	0.004

Table 4.6 ANOVA Summary Table

Variation	Sum of	Degrees of	Mean	Observed	
Source	Squares	Freedom	Square	f value	P-Value
A	1.279	1	1.279	7.919	0.023
B	18.679	1	18.679	115.695	0.000
C	0.094	1	0.094	0.580	0.468
AB	0.687	1	0.687	4.256	0.073
AC	0.006	1	0.006	0.037	0.852
BC	0.069	1	0.069	0.425	0.533
ABC	0.004	1	0.004	0.022	0.886
Error	1.292	8	0.161		
Total	20.826	15			

As shown in Table 4.6, the only statistically significant effects for a significance level of $\alpha = 0.10$, are the main effect of factor 1 (material), the main effect of factor 2 (energy of incident electron beam), and the two factor interaction of

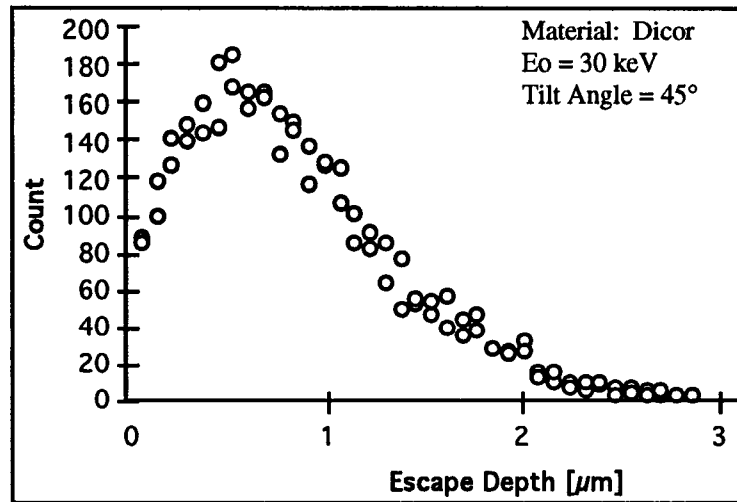
variables 1 and 2 (material - energy interaction). Overall, a model incorporating these effects is given by Equation 4.10.

$$Escape\ Depth\ [\mu m] = 1.465 - \frac{0.565}{2}A + \frac{2.161}{2}B - \frac{0.414}{2}AB \quad (4.10)$$

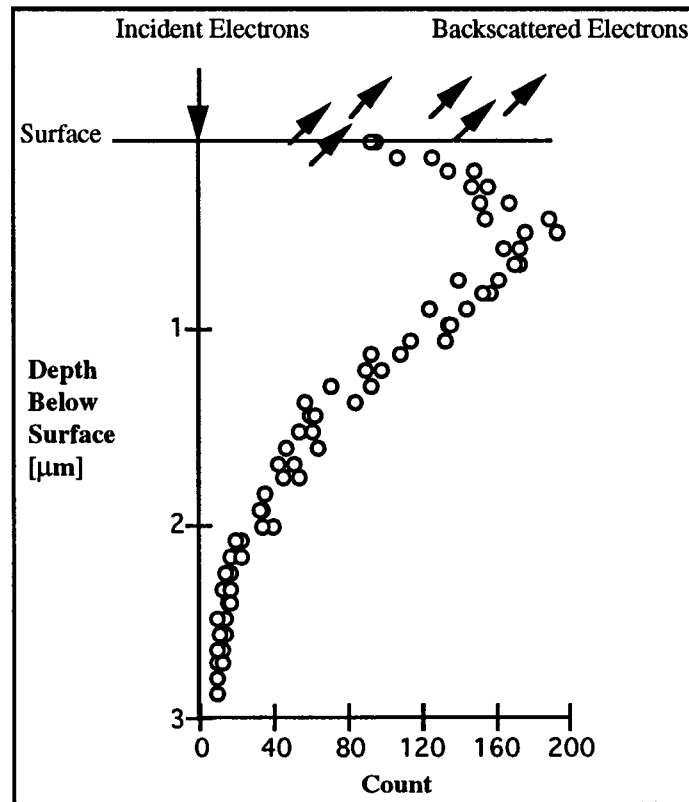
This model indicates the response (maximum escape depth) is effected by factors A and B as follows. First, an increase in material density results in less penetration of incident electron beams. Second, increasing the voltage of incident electron beams results in greater penetration of incident electrons. Finally, increasing both the material density and beam voltage simultaneously tends to reduce the penetration of electron beams. However, increasing one of the factors while decreasing the other factor increases the depth of penetration. Overall, the optimal escape depth is obtained by setting factor A (material density) at its low level while setting factor B (beam voltage) at its high level.

4.4 Observations from Analytical Electron Microscopy

Several additional trends are apparent when observing the distribution of backscattered electrons. In these distributions, escape depth is plotted as a function of number (count) of backscattered electrons detected (Figure 4.5a). The distribution shown in Figure 4.5a can be interpreted as the number of backscattered electrons which escape from a given depth (Figure 4.5b). Furthermore, in the subsequent plots, data from the two replicated runs are plotted simultaneously -- the test order is denoted in the legend. Also, in the plots, triangular markers denote alumina while circular markers denote Dicor.



(a) Distribution of Backscattered Electrons

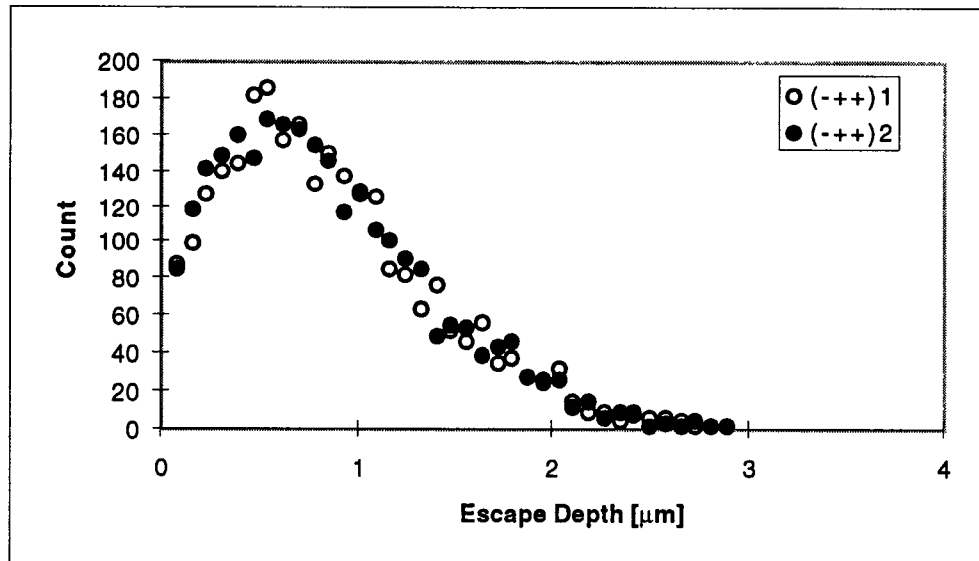


(b) Escape Depth of Backscattered Electrons

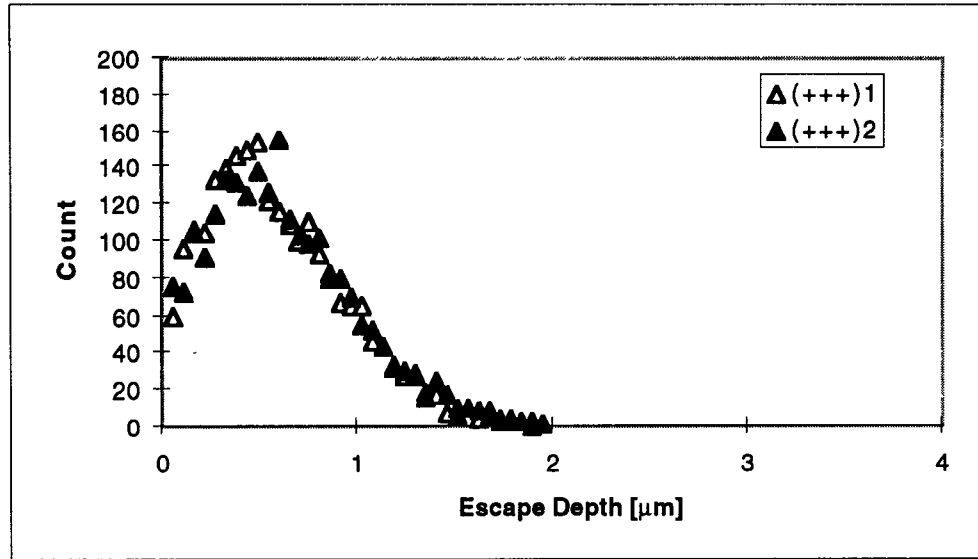
Figure 4.5 Frequency Distributions of Backscattered Electrons

4.4.1 Test condition 1bc (-++) versus abc (+++)

In the first set of experiments, the energy of the incident electron beam (30 keV) and tilt angle (45°) are set at high levels. Then, a comparison is made between the distributions obtained for alumina (low level) and Dicor (high level). Figures 4.6 indicate the distributions of backscattered electrons are positively skewed (shifted to the right). Furthermore, the overall mean response of the distribution of alumina ($0.853 \mu\text{m}$) is less than that of Dicor ($0.603 \mu\text{m}$). Overall, the replicated runs result in relatively same distributions in terms of shape.



(a) Material: Dicor

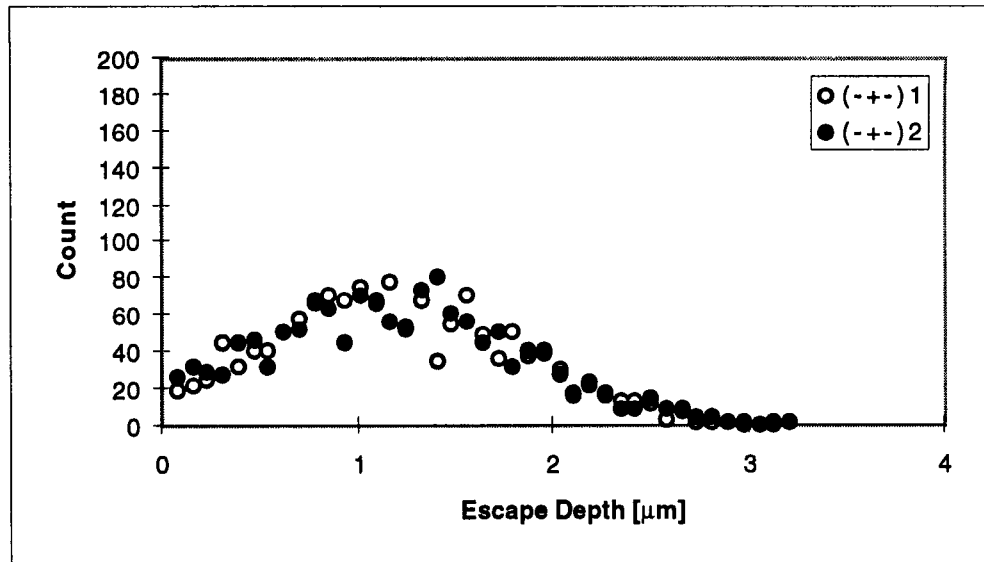


(b) Material: Alumina

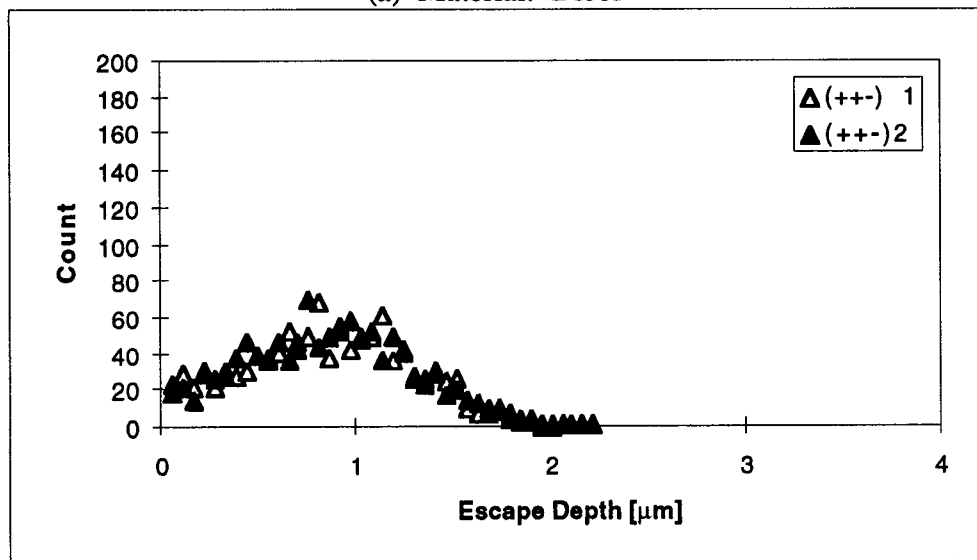
Figure 4.6 Beam Voltage = 30 keV, Tilt angle = 45°

4.4.2 Test condition 1b1 (-+-) versus ab1 (++-)

In the next set of experiments, the energy of the incident electron beam (30 keV) is set at the high level while tilt angle (0°) is set at the high level. As before, a comparison is made between the distributions obtained for alumina (low level) and Dicor (high level). In these experiments, in which the tilt angle is zero (normal incidence), the distributions of backscattered electrons for both materials is fairly symmetric. In addition, the overall mean response (mean escape depth) of the distributions for both alumina (0.858 μm) and Dicor (1.202 μm) is larger the corresponding values for the first set of experiments. As before, the mean response for Dicor is larger than that of alumina.



(a) Material: Dicor

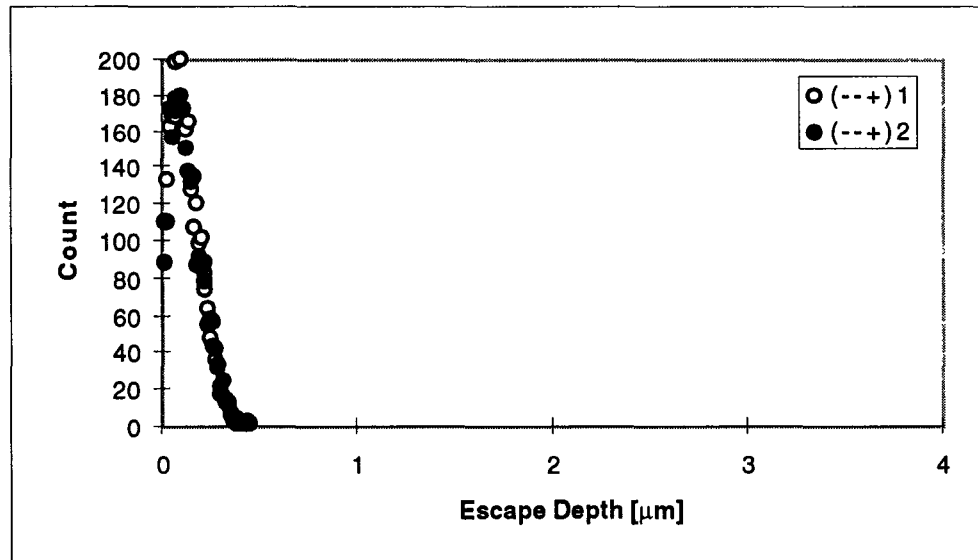


(b) Material: Alumina

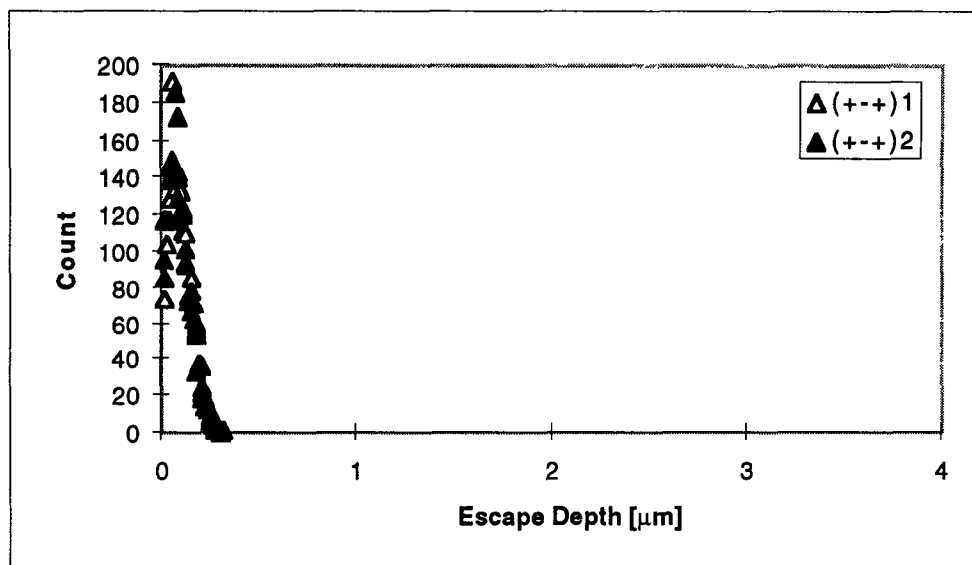
Figure 4.7 Beam Voltage = 30 keV, Tilt angle = 0°

4.4.3 Test condition 11c (--+) versus a1c (+++)

In the third set of experiments, the energy of the incident electron beam (10 keV) is set at the low level while tilt angle (45°) is set at the high level. A comparison is made between the distributions obtained for alumina (low level) and Dicor (high level). As indicated in Figures 4.8, the overall mean escape depth for both Dicor ($0.129\ \mu\text{m}$) and alumina ($0.093\ \mu\text{m}$) is notably lower than for the response for the other test conditions. In particular, the energy of the incident electron beam appears to be directly correlated to the mean escape depth, i.e., increasing the voltage increases the escape depth and vice versa. This trend is also observed by the large positive coefficient corresponding to factor B (incident beam voltage) in the model developed in section 4.3.



(a) Material: Dicor



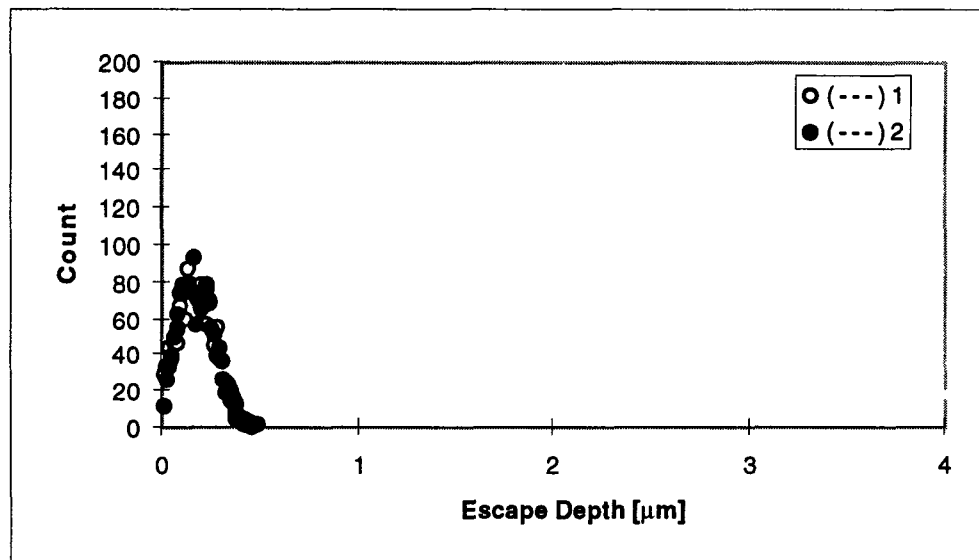
(b) Alumina

Figure 4.8 Beam Voltage = 10 keV, Tilt angle = 45°

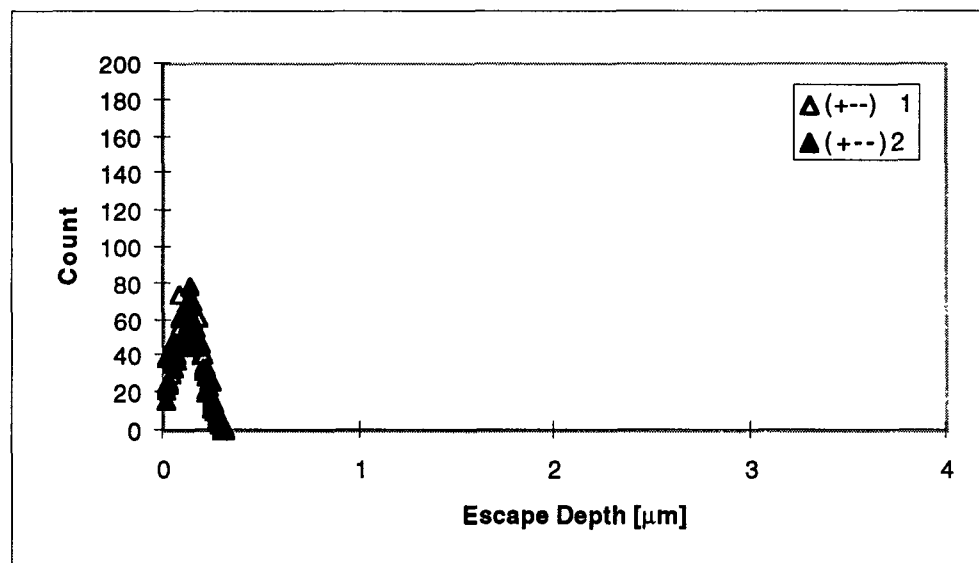
4.4.4 Test condition 111 (---) versus a1c (+--)

In the final set of experiments, the energy of the incident electron beam (10 keV) tilt angle (0°) are both set at low levels. Again, a comparison is made between the distributions obtained for alumina (low level) and Dicor (high level). As shown in Figures 4.9, the distribution of backscattered electrons for both alumina and Dicor are relatively symmetric when compared to the third set of experiments. Overall, the distributions for this set of experiments parallel the results obtained for the following test conditions: incident beam energy = 30 keV, and tilt angle = 0°. However, since the beam voltage is set at the low level, the overall mean response for this set of experiments is noticeably lower than the results for the second set of experiments.

Overall, the mean escape depth for of Dicor ($0.182\ \mu\text{m}$) is larger than that of alumina ($0.129\ \mu\text{m}$).



(a) Material: Dicor



(b) Material: Alumina

Figure 4.9 Beam Voltage = 10 keV, Tilt angle = 0°

4.4.5 Quantitative Investigation of Distribution of Backscattered Electrons

A statistical parameter which can be used to further investigate the shape characteristics of the distribution of backscattered electrons is skewness, Equation 4.11 [17]. This parameter provides an estimate of the symmetry of distributions. A perfectly symmetric distribution will have a skewness of $\beta = 0$. Furthermore, the skewness value estimates the degree of non-symmetry and the direction of the non-symmetry. For example, a skewness value of -3 indicates a distribution skewed to the left by three standard deviations [17].

$$\beta = \frac{1}{s^3} \sum_{i=1}^n (x_i - \bar{x})^3 f(x_i) \quad (4.11)$$

where s represents the standard deviation

x_i represents the i th depth thickness level

$\bar{x}$ represents the mean escape depth

$f(x_i)$ represents the probability density function corresponding to x_i

Overall, several observations can be made from the skewness values corresponding to the different distributions observed from the experiments (see Table 4.7). First, for the given test conditions, all the distributions are positively skewed indicating that which are closer to the surface escape more readily than those electrons which have penetrated further into the material. However, the degree of skewness significantly varies depending of the test conditions.

For example, the average skewness values for experiments involving Dicor are larger than the corresponding values for alumina. Second, the most significant changes in skewness values are observed when the tilt angle is changed. For all of the

experiments in which the first two factors (material and energy of incident electron) are fixed, increasing the tilt angle significantly increases the degree of positive skewness. In turn, the mean and maximum escape depths are noticeably reduced. Finally, varying the energy of the incident electron beam while holding the other two factors (material and tilt angle) constant does not significantly effect the skewness values. In other words, the shape of the distribution of the escape depth of backscattered electrons is not significantly effected by the energy of incident electron beams.

Table 4.7 Skewness Values

Skewness			
Test	Trial 1	Trial 2	Average
abc (+++)	0.751	0.660	0.706
ab1 (++-)	0.103	0.222	0.163
a1c (+-+)	0.629	0.727	0.678
a11 (+--)	0.238	0.140	0.189
1bc (-++)	0.802	0.811	0.807
1b1 (-+-)	0.275	0.273	0.274
11c (--+)	0.704	0.749	0.726
111 (---)	0.248	0.323	0.285

Chapter 5

Experimental Investigation

5.1 Significance of Selected Material

As described in Chapter 2, the material selected for the milling tests in this thesis study is DICOR/MGC, a machinable glass ceramic material. For dental restorations, a particularly important consideration is the closeness of physical properties of the restorative material to that of human enamel. Overall, in terms of translucency, thermal conductive, density, and hardness, DICOR/MGC closely resembles the physical properties of human enamel. A comparison between some of the important properties of human enamel and DICOR/MGC are presented in Table 5.1 [18, 19]. Other physical properties of DICOR/MGC ceramic were presented in Table 2.1 [4]. Furthermore, in addition to having a material removal rate close to that of human enamel when compared to other advanced ceramic materials (as indicated in Table 2.2), experimental studies indicated that DICOR/MGC wore at higher rate than human enamel (see Figure 2.5).

5.2 Milling Experiments

For the experimental investigations, a series of milling tests are performed using the Matsuura MC-570 Machining Center -- a state-of-the-art computer numerical control (CNC) machine -- in the Advanced Design and Manufacturing Laboratory. Specifically, a 2 level - 3 factor factorial design experiment is performed to assess the machinability of DICOR/MGC dental ceramic material. As illustrated in Figure 5.1, the three factors of interest are feed (factor F), spindle speed (factor S), and depth of cut (factor D). Note that a full axial depth of cut is taken for all experiments, i.e., the radial depth of cut

is entirely governed by the diameter size of the end mill. The high and low levels for each of the variables are listed in Table 5.2.

Table 5.1 Properties of Human Enamel and DICOR/MGC Material

Property	Enamel	DICOR/MGC
Modulus of Elasticity [MPa]	4.6 (10^4)	6.8 (10^4)
Density [g/cm^3]	2.9	2.8
Index of Refraction	1.65	1.52
Thermal Conductivity [$\text{W}/(\text{m}\cdot\text{K})$]	0.93 [20]	1.6
Thermal Diffusivity [mm^2/s]	0.469 [20]	0.79
Microhardness, [KHN]	343 [19]	330

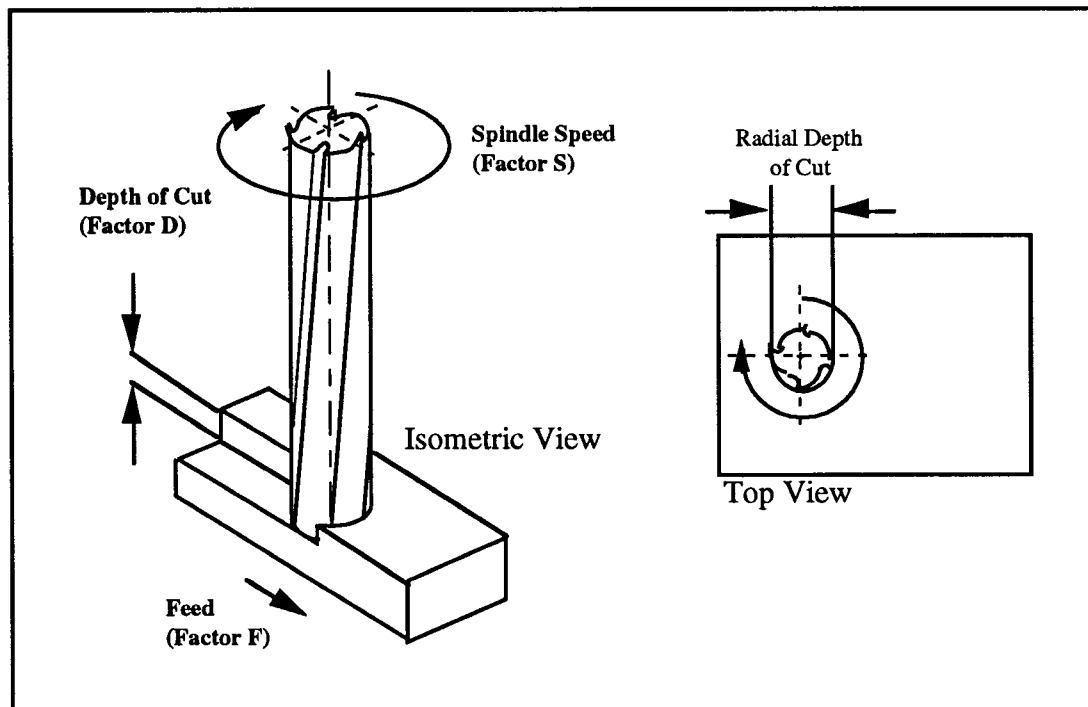


Figure 5.1 Machining Parameters: Feed, Depth of Cut, Spindle Speed

Table 5.2 Settings for Factors

Factor	Unit	Low Level (-1)	High Level (+1)
Feed	[in/min]	0.2	0.4
Spindle Speed	[rpm]	600	900
Depth of Cut	[in]	0.01	0.02

Overall, as shown in Figure 5.2, all of the milling tests are performed in a submerged machining environment to ensure that the surface is immersed in an adequate volume of coolant. A commercial multi-purpose water soluble cutting fluid (Rust Lick WS 5050) is used to provide lubrication and assist in the material removal process. A 0.125 (1/8) inch diameter high speed steel (HSS) end mill with two flutes (cutting edges) is used to mill slots in a ceramic sample. As indicated in Figure 5.3, the slots are first cut on the left edge of the sample and then cut further to the right. A total of two ceramic samples are used to accommodate the eight unique cutting combinations. Note that order of the milling tests has been randomized as indicated in Figure 5.3. The output parameters used to evaluate machining performance in terms of surface integrity.

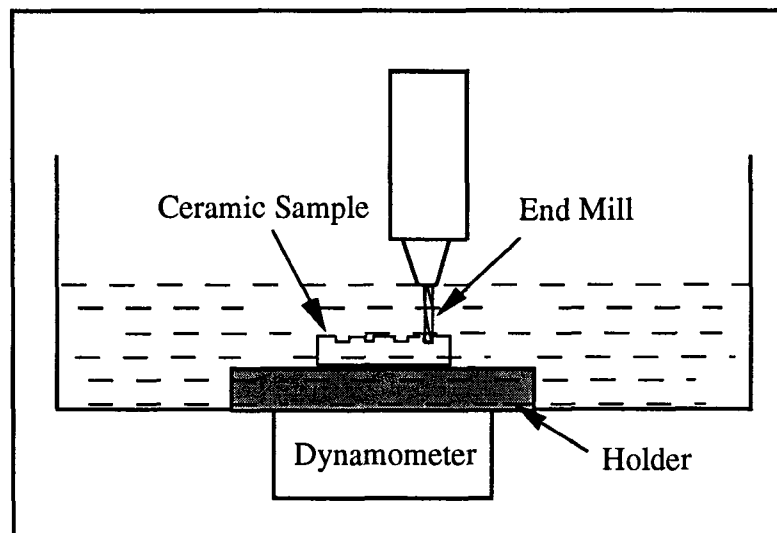


Figure 5.2 Submerged Machining Environment

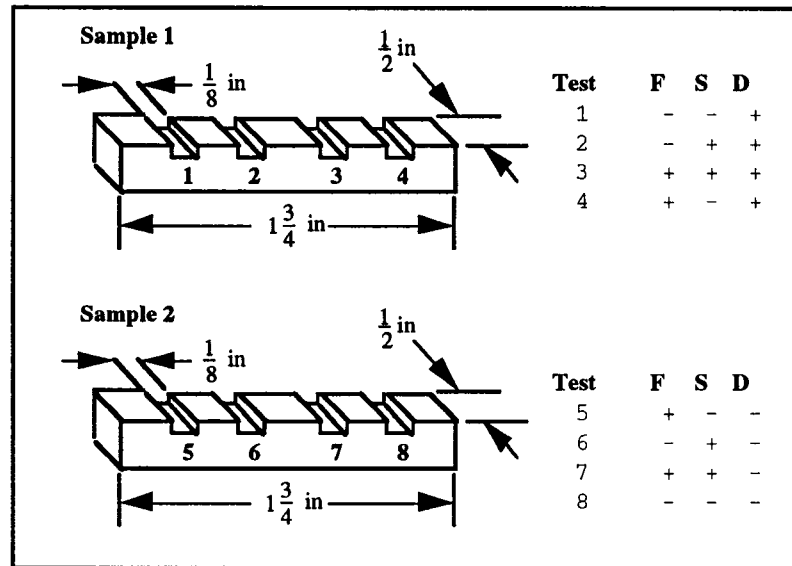


Figure 5.3 Order of Milling Tests

5.3 Cutting Forces

A dynamometer was used during the machining tests. Figure 5.4 illustrates that three cutting force components -- tangential, normal, and transverse components -- were monitored during machining. The transducer used three full Wheatstone Bridge circuits with strain gages as the sensing elements. A commercial data acquisition software package (LabView) and a commercial spreadsheet software package (Excel) were used to record the signals and perform data analysis.

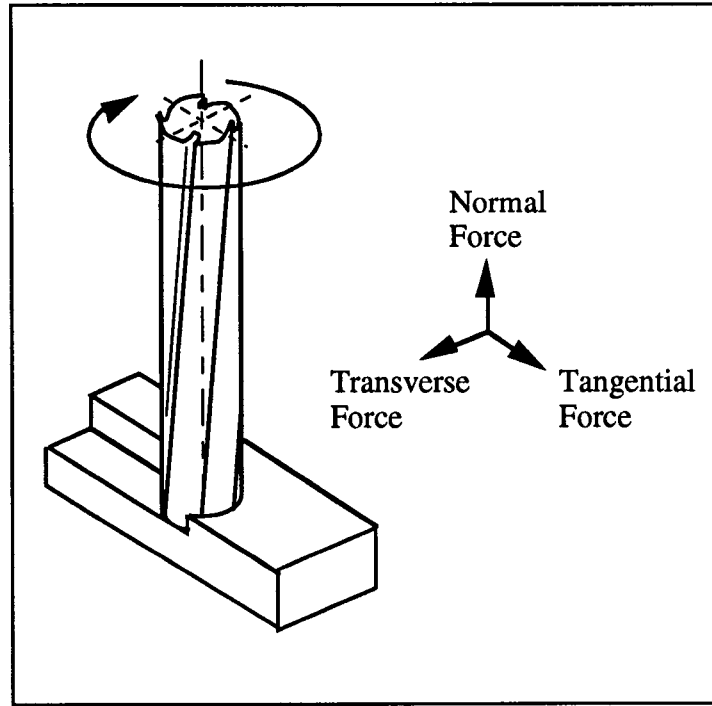


Figure 5.4 Cutting Forces

A typical plot of the three force signals is given in Figure 5.5. These signals represent the three cutting force components measured during the machining of slot 1. By examining these signals, the mean forces represent the average (or static) forces and the observed deviation about the mean forces represent the dynamic components of the three forces. Overall, a summary of the static (average) and dynamic (standard deviation about the average) force components for the normal, tangential, and transverse force components for the eight tests is provided in Table 5.3.

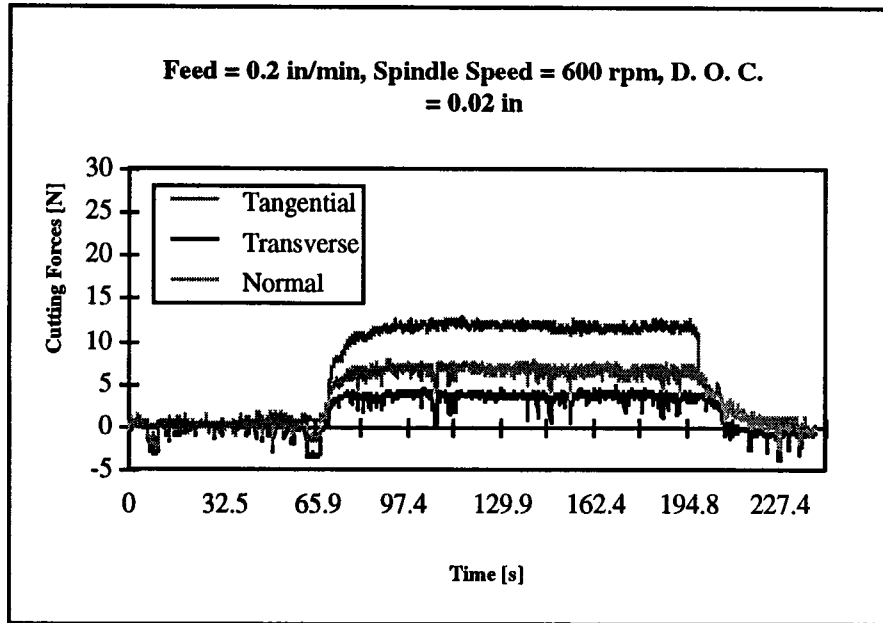


Figure 5.5 Cutting Force Signals

Table 5.3 Cutting Forces

Condition				Tangential Force		Transverse Force		Normal Force	
Slot	F	S	D	Avg	Std	Avg	Std	Avg	Std
1	-	-	+	11.49	1.26	3.64	0.61	6.66	0.74
2	-	+	+	16.84	1.36	1.98	1.23	4.94	0.84
3	+	+	+	23.49	0.88	5.43	1.04	3.89	0.56
4	+	-	+	24.29	0.53	9.22	0.88	1.85	0.61
5	+	-	-	9.91	1.21	2.34	0.62	3.51	0.70
6	-	+	-	8.77	0.97	1.83	0.61	2.89	2.03
7	+	+	-	12.65	0.75	2.44	0.74	2.99	1.15
8	-	-	-	9.19	0.54	7.82	0.67	1.47	1.48

Avg = Mean Force

Std = Standard Deviation about Mean Force

5.4 Image Processing to Evaluate Surface Integrity

As described in Chapter 3, a surface integrity assessment system based on environmental scanning microscopy (ESEM) and image processing is used to characterize the surface finish of the milled samples (see Figure 5.6). A Macintosh IIfx desktop computer equipped with a QuickCapture frame grabber hardware board and a public domain image processing software allows images to be captured in real time. After an ESEM micrograph of the surface topography is captured, the picture file can be saved in different file formats.

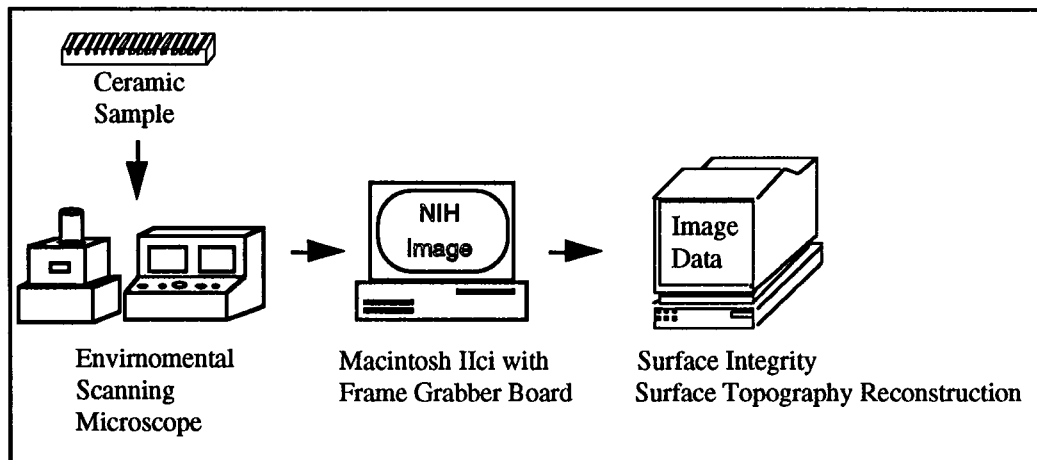


Figure 5.6 Surface Integrity Assessment System

In the two subsequent sections image processing is used for two purposes. First, the role of the milling parameters on surface texture is examined using two statistical parameters -- roughness average R_a and cavity density CD. Second, the role of the machining parameters on edges of the slots is examined with the aid of the developed image processing system.

5.4.1 Formation of Surface Texture

To obtain height data from the ESEM micrographs, a stereopair calibration similar to that described in Chapter 3 is performed. In the calibration operation, two pictures (micrographs) of the same location on the machined surface are required. First, a micrograph of the untilted surface is obtained (tilt angle $\alpha = 0^\circ$). Next, another micrograph of the same location is obtained when the specimen is tilted by 5° ($\alpha = 5^\circ$). The two images are acquired and digitized using a public domain image processing software -- NIH Image Software (Version 1.58b33) [21]. After digitization, the image data are represented by numerical values, i.e., pixels within the picture are assigned numerical values ranging from 0 to 255 depending on their contrast (level 255 corresponds to the darkest contrast value). Next, as shown in Figure 5.7, two points with different contrast levels are selected for the image obtained from the untilted sample. The coordinates (x_1 and x_2) and the gray levels (Level 1 and Level 2) of the points are recorded. The difference in the coordinates (D), which is calculated in pixels, is converted to length data (Δd) using the scale parameter shown in the micrograph. Next, the same points are located in the ESEM micrograph for the tilted surface. Again, the difference in the coordinates (D_t) are recorded and converted into length data (Δd_o). The variation in height (Δz) is calculated using Equation 5.1. Finally, as indicated by Equation 5.2, a height to gray level ratio (Δz / difference in gray levels) using the difference in gray levels obtained from the first ESEM micrograph and the calculated height value. The ratio is important for reconstruction of the surface topography.

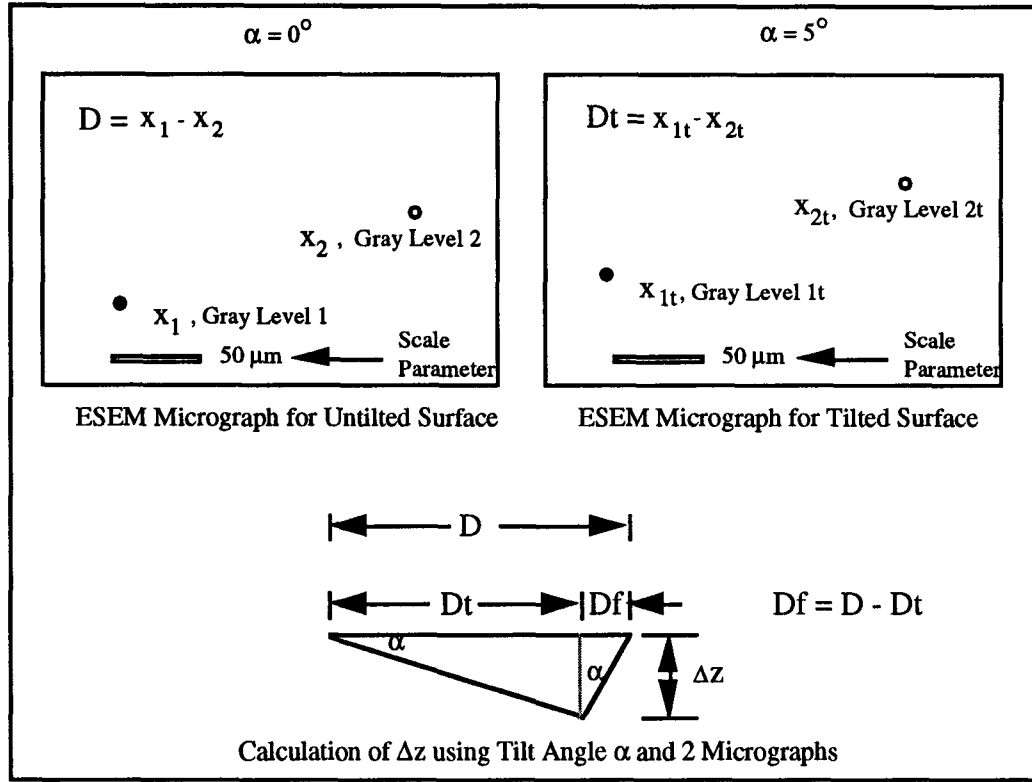


Figure 5.7 Stereopair Calibration

$$\Delta z = \frac{|\Delta d - \Delta d_o|}{\tan \alpha} \quad (5.1)$$

where Δz represents the undistorted height between two points

Δd represents the distance between points a and b for the untilted surface,
characterized as label D in Figure 5.7

Δd_o represents the distance between points a and b for the tilted surface,
characterized as label Dt in Figure 5.7

α represents the tilt angle

$$Ratio = \frac{\Delta z}{|GR1 - GR2|} \quad (5.2)$$

where Δz represents the undistorted height between two points

GR1 represents the gray level intensity of point a for the untilted surface

GR2 represents the gray level intensity of point b for the tilted surface

After calibration, the gray levels are scaled according to the height calibration constant. Finally, the center plane CP for the data is obtained by averaging the height data, as indicated in Equation 5.3. This plane is referenced as the origin for subsequent calculations, or the center plane. The height data are rescaled as shown in Equation 5.4.

$$CP = \frac{1}{PQ} \sum_{i=1}^P \sum_{j=1}^Q z(x_i, y_j) \quad (5.3)$$

where CP represents the center plane

P represents the total number of rows in the data file

Q represents the total number of columns in the data file

$z(x_i, y_j)$ represents the elevation at the point (x_i, y_j)

$$zs(x_i, y_j) = z(x_i, y_j) - CP \quad (5.4)$$

where $zs(x_i, y_j)$ represents the rescaled data point

$z(x_i, y_j)$ represents the elevation at the point (x_i, y_j)

CP represents the center plane

5.4.1.1 Estimation of Surface Integrity Parameters

Two area statistical parameters are used to assess surface integrity -- roughness average and cavity density. The roughness average R_a , computed by Equation 5.5 [22],

has been the traditionally used parameter to assess surface finish. In this thesis, an area estimate of the roughness average is obtained using scanning electron microscopy and image processing to provide an estimate of the surface finish. The proposed technique is better suited to characterizing fracture surfaces than traditional stylus profilometers since the technique does not encounter resolution problems caused by a finite stylus tip.

$$R_a = \frac{1}{PQ} \sum_{i=1}^P \sum_{j=1}^Q |zs(x_i, y_j)| \quad (5.5)$$

where R_a represents the area roughness average

P represents the total number of rows in the data file

Q represents the total number of columns in the data file

$zs(x_i, y_j)$ represents the rescaled data point

The second statistical area parameter used to characterize surface intensity is cavity density CD, defined in Equation 5.6. This parameter, which is defined in Equation 5.6, is the percentage of area below the selected height level to the total sampling area. This new parameter can be used to characterize the surface texture formed during machining. The parameter quantifies the area occupied by valleys or cavities at a given elevation on the surface texture.

$$CD = \frac{Area_1}{Area_T} (100) \quad (5.6)$$

where CD represents the cavity density at a selected height location

$Area_1$ represents the area of below the selected height location

$Area_T$ represents the total sampling area

5.4.1.2 Examination of ESEM Micrographs

As previously discussed, a total of eight slots are milled using different machining parameters. The stereopair calibration technique allows reconstruction of surface topography from ESEM micrographs using a numeric computation and visualization software -- MATLAB version 4.2a [23]. A representative numeric data file of an ESEM micrograph is given by Figure 5.8. Altogether, the total scan area consists of 640 points by 480 points (640 pixels by 480 pixels). Since all the images are obtained at a magnification factor of 250, the total scan area represents a physical area of $0.48 \times 0.36 \text{ mm}^2$, i.e., the scale parameter is $0.75 \text{ }\mu\text{m/pixel}$. Performing a statistical analysis samples are taken. Each of them has 360 pixels by 360 pixels or $0.27 \times 0.27 \text{ mm}^2$. As shown in Figure 5.8, the selected sampling area is further divided into 4 smaller equal sampling areas (180 pixels by 180 pixels). Each of these areas, denoted as upper left (UL), upper right (UR), lower left (LL), and lower right (LR), represents a total sampling area of $0.135 \times 0.135 \text{ mm}^2$. Next, to characterize surface integrity, the two statistical parameters -- roughness average R_a and cavity density CD -- are calculated based on the reconstructed topographies of the four sampling areas. The pertinent MATLAB script files are included in Appendix A. Furthermore, ESEM micrographs of locations of the initial tool engagement and tool exits provide further insight about damage associated with edge effects.

In the following sections, surface integrity is examined as a function of the different cutting parameters. The following notation is used to differentiate the different cutting parameters. First, the low level of each of the machining parameters -- feed (F), spindle speed (S), and depth of cut (D) -- is denoted as 1. On the other hand, the high level for each parameter is denoted by the lowercase letter corresponding to the parameter, i.e., the high level for the factor feed is denoted by the symbol f, and so

forth. The pertinent numerical values for the high and low levels for each factor are given in Table 5.3. A visual comparison of the role of depth of cut on the formation of surface texture is provided. Next, based on the provided image, statistical surface integrity parameters (roughness average R_a and cavity density (CD) are computed. Then, these parameters are used as output parameters to characterize the main, two-factor effects, and three-factor interactions of the three machining parameters.

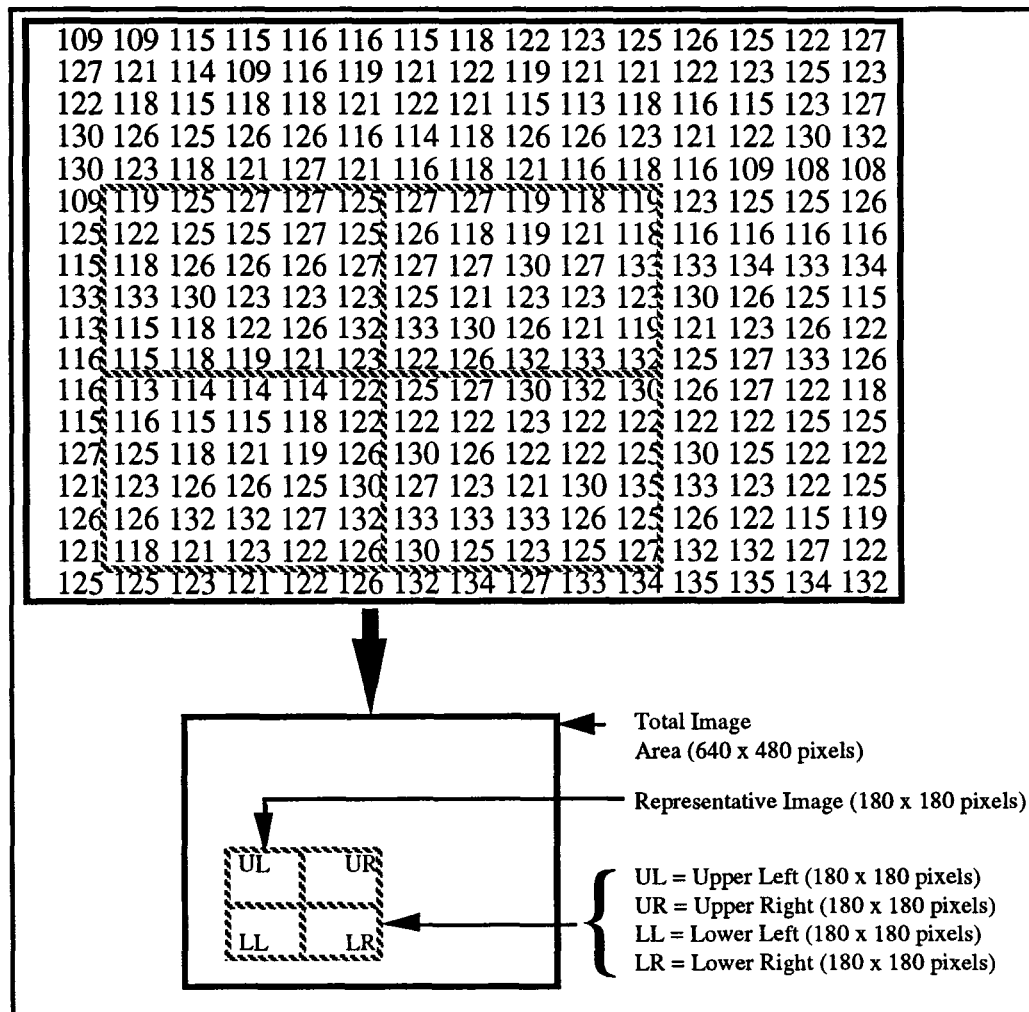


Figure 5.8 Selection of Sampling Areas

5.4.1.2.1 Milling test (111) versus (11d)

For the milling test corresponding to condition 111 (slot 8), the following cutting parameters are used: feed = 5.08 mm / min (0.2 in / min), spindle speed = 600 rpm, and depth of cut = 0.254 mm (0.01 in). An ESEM micrograph of the machined surface is shown in Figure 5.9. The area shown in the ESEM micrograph is divided into four equal sampling areas denoted as previously indicated. The two parameters, roughness average R_a and cavity density CD, are calculated for each of the sampling areas. The cavity density is evaluated at a depth of 1 μm below the center plane. Next, using MATLAB software, the surface topography corresponding to the upper left (UL) sampling area is reconstructed (see Figure 5.10). The calculated mean values for roughness average and cavity density are 0.21 μm and 0.21%, respectively. The associated standard deviation among the four samples are 0.03 μm and 0.15 % for roughness average and cavity density, respectively.

Slot 1 is milled using test condition 11d: feed = 5.08 mm / min (0.2 in / min), spindle speed = 600 rpm, and depth of cut = 0.254 mm (0.01 in). The ESEM micrograph for the surface of this milled surface is provided in Figure 5.11. Furthermore, comparison of the reconstructed topographies between slot 1 (11d) and slot 8 (111) indicates a coarser finish for slot 1. For slot 1, the calculated parameters for roughness average and cavity density are $R_a = 0.94 \pm 0.03 \mu\text{m}$ and $CD = 23.23 \pm 1.47 \%$, respectively. The effect of increase in depth of cut is also evident in the calculated mean values of roughness average (0.21 vs. 0.94 μm) and cavity density (0.21% vs. 23.23 %). Finally, Figure 5.13 provides a comparison between the contour plots for the two machining conditions. Note that the plots are evaluated at an elevation of 1 μm below the center plane.

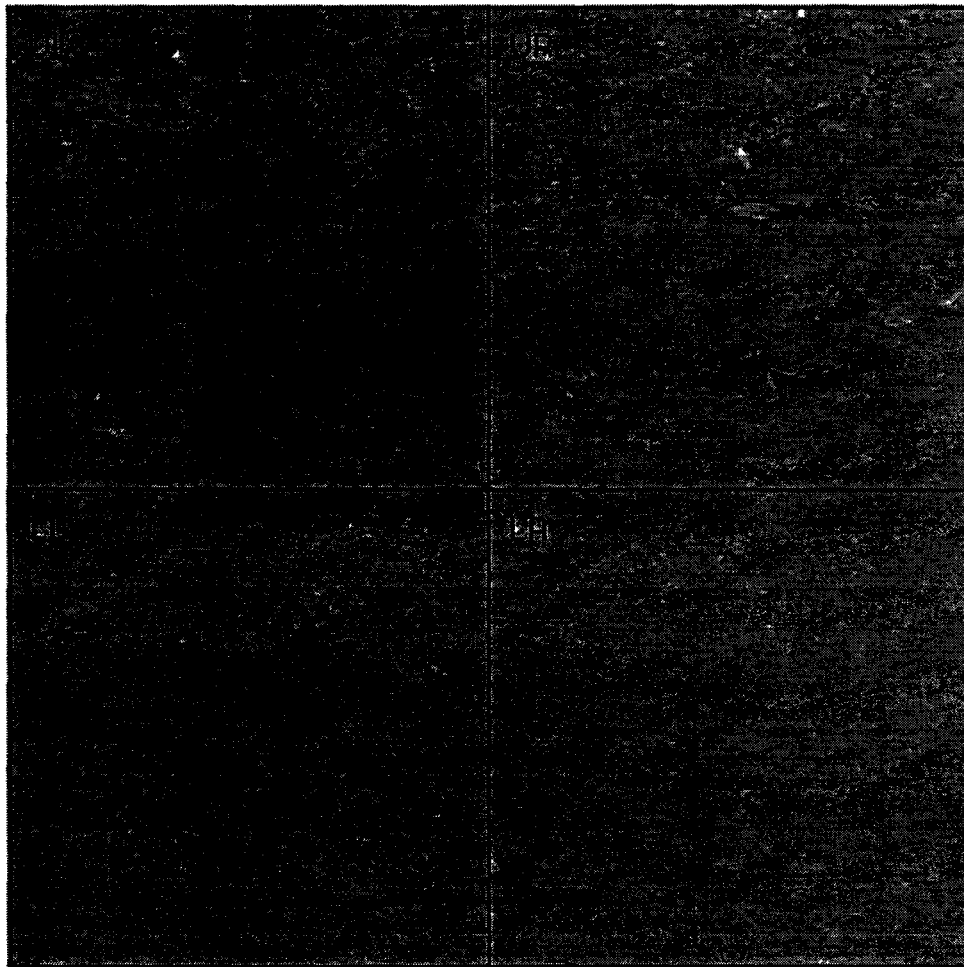


Figure 5.9 ESEM Micrograph for Cutting Condition 111

Slot 8, $R_a = 0.21 \mu\text{m}$

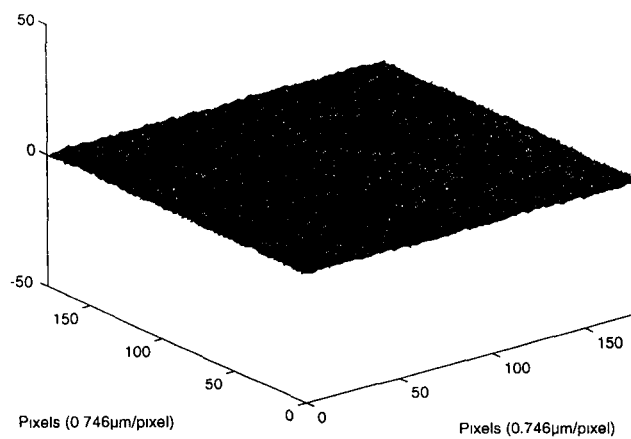


Figure 5.10 Reconstructed Surface Topography for Condition 111 (UL section)

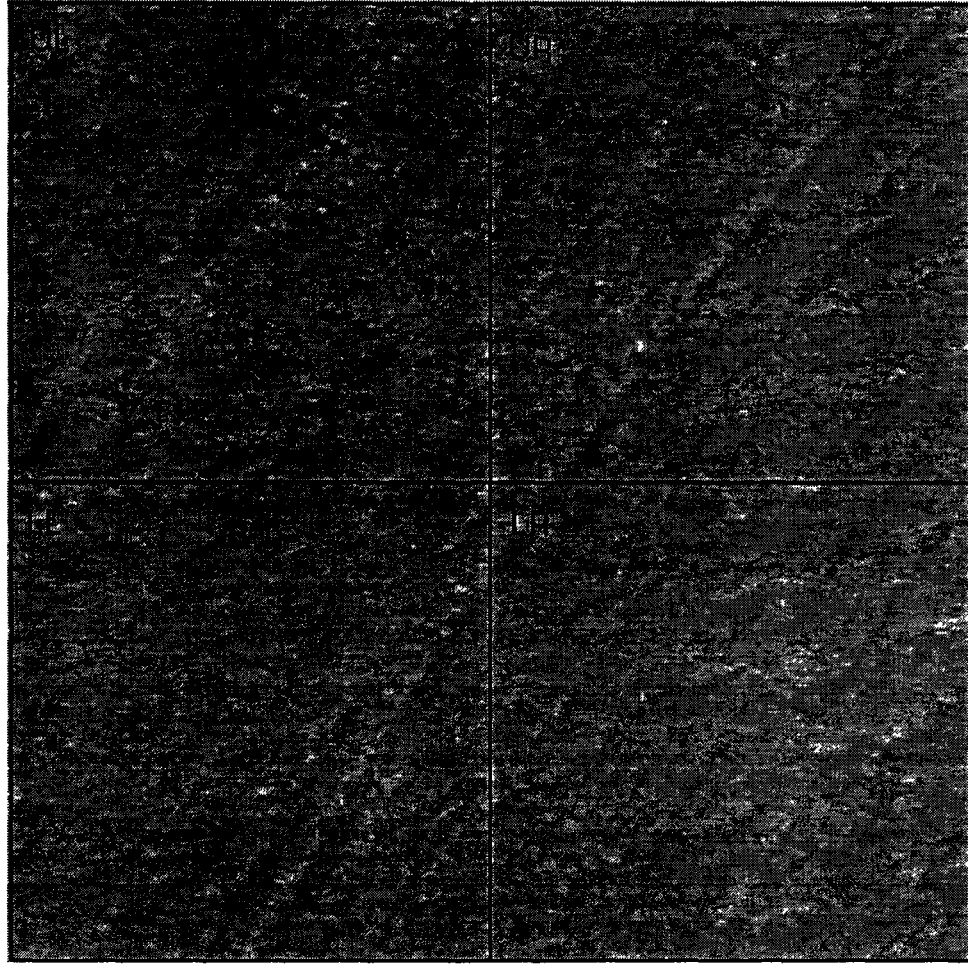


Figure 5.11 ESEM Micrograph for Cutting Condition 11d

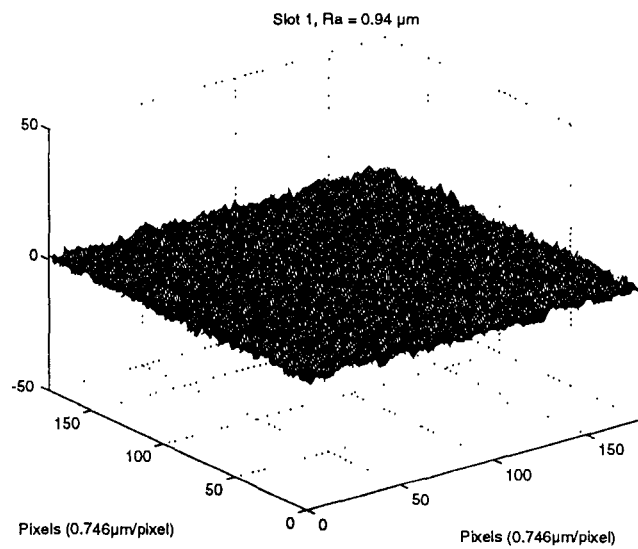
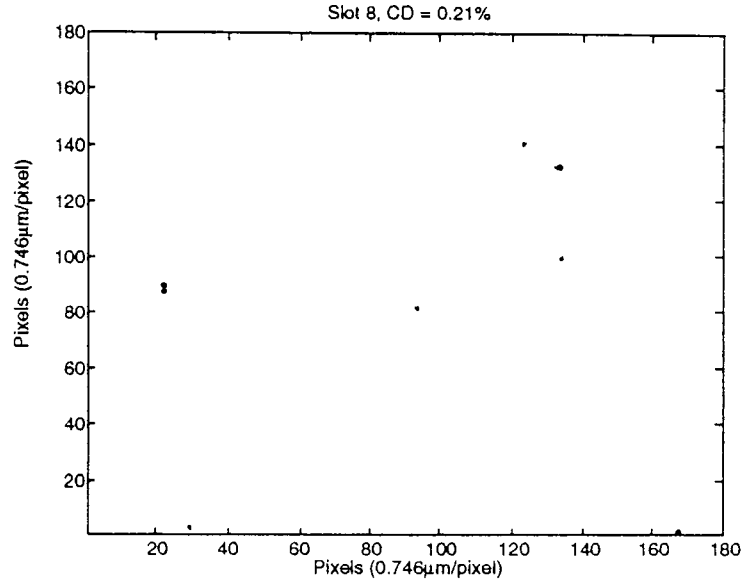
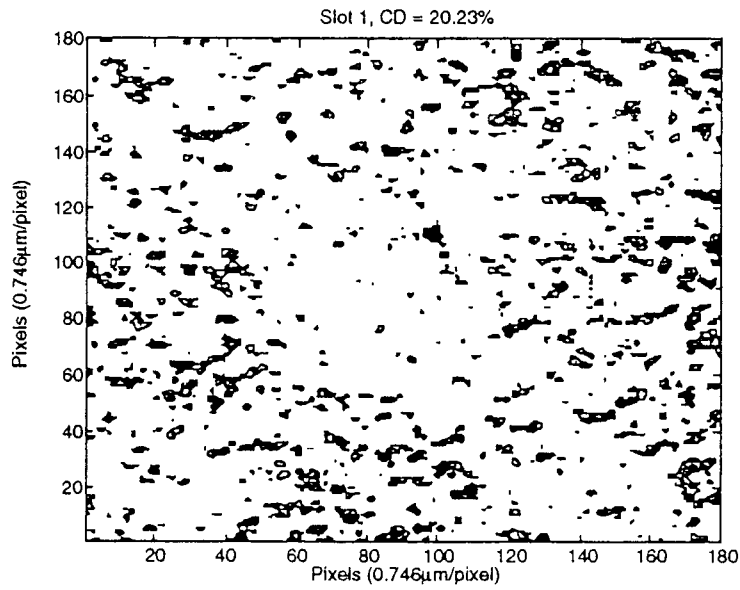


Figure 5.12 Reconstructed Surface Topography for Condition 11d (UL section)



(a) Condition 111 (Slot 8)



(b) Condition 11d (Slot 1)

Figure 5.13 Cavity Densities (111 vs 11d), Elevation = -1 μm

5.4.1.2.2 Milling test (f11) versus (f1d)

For milling test condition f11 (slot 5), the following cutting parameters are used: feed = 10.16 mm/min (0.4 in/min), spindle speed = 600 rpm, and depth of cut = 0.254 mm (0.01 in). The roughness average mean value and the cavity density value for this condition are $1.43 \pm 0.11 \mu\text{m}$ and $28.37 \pm 2.49\%$, respectively. These values are noticeably higher than those for cutting condition 111 or condition 11d. Hence, increasing the feed rate seems to adversely effect the surface quality. The dimensional surface topography for area UL is illustrated in Figure 5.15.

The milling test corresponding to condition f1d (slot 4) uses identical machining parameters as condition f11 except that the depth of cut is increased to 0.508 mm (0.02 in). The mean roughness average value ($1.73 \pm 0.15 \mu\text{m}$) and average cavity density ($32.59 \pm 4.32\%$) do not differ appreciably from that of cutting condition f11. Examining the two ESEM micrographs, Figures 5.14 and 5.16, reveals that the two surfaces are relatively similar in terms of topographical features. As before, surface topography of the upper left area of the ESEM micrograph is reconstructed (see Figure 5.17).

Contour plots evaluated at an elevation of $1 \mu\text{m}$ below the center plane corresponding to machining conditions f11 and f1d are provided in Figures 5.18 a and b, respectively. Examining the two figures, the difference in cavity densities for the two conditions is not statistically significant ($CD = 28.37 \pm 2.49\%$ for condition f11 vs. $CD = 32.59 \pm 4.32\%$ for condition f1d).

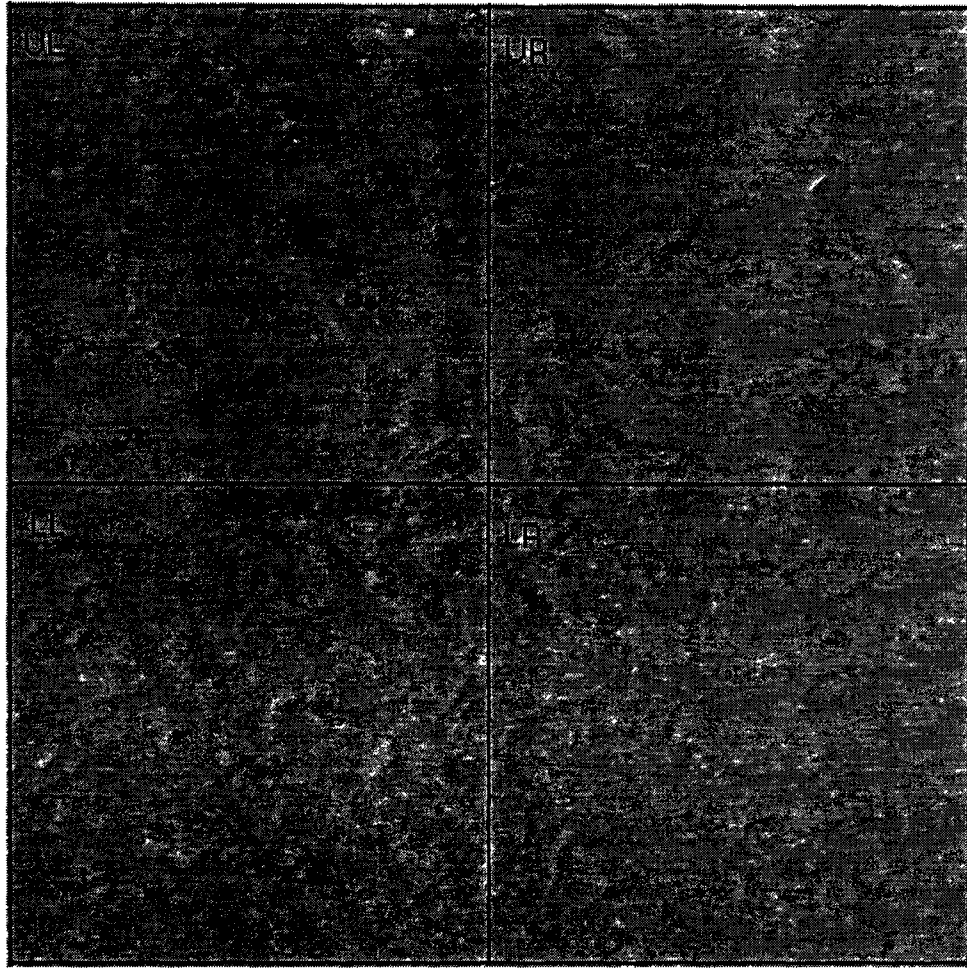


Figure 5.14 ESEM Micrograph for Cutting Condition f11

Slot 5, Ra = 1.43 μm

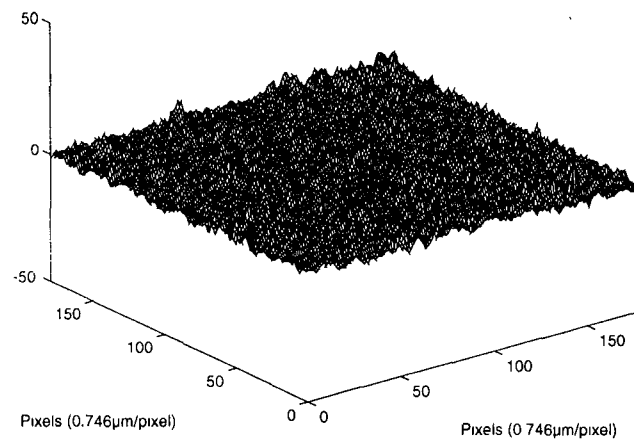


Figure 5.15 Reconstructed Surface Topography for Condition f11 (UL section)

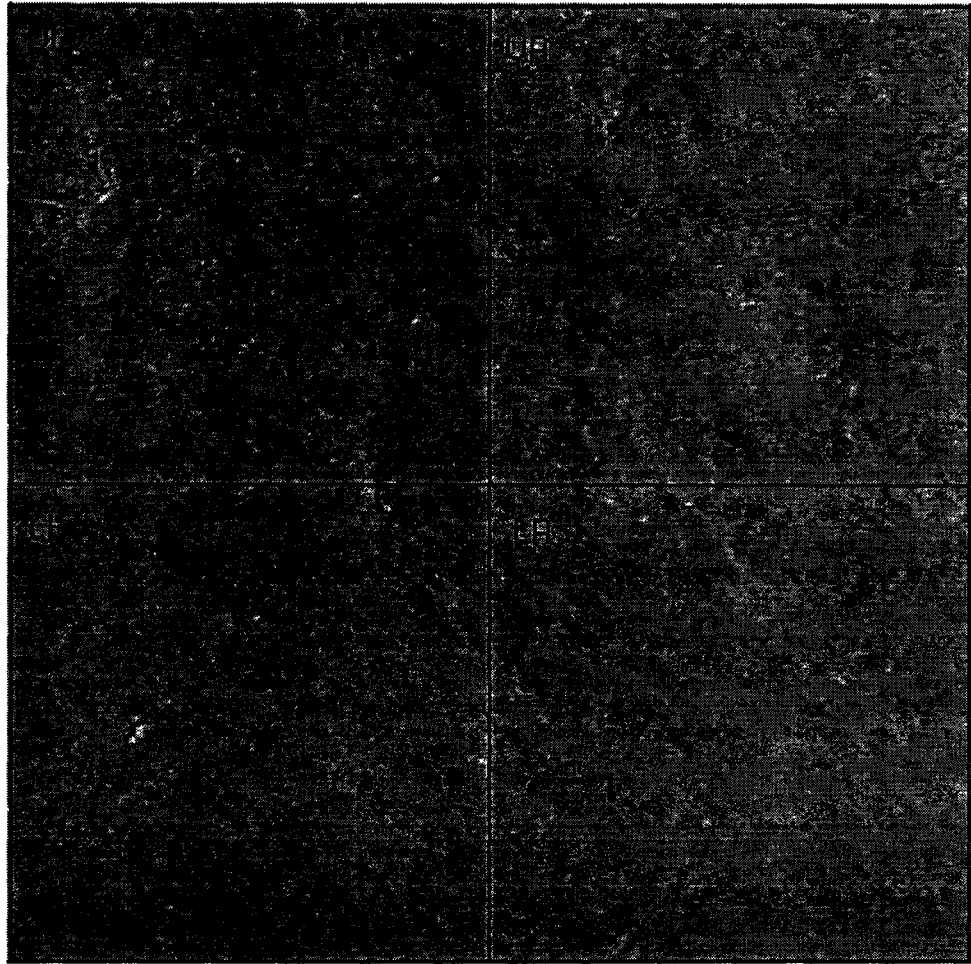


Figure 5.16 ESEM Micrograph for Cutting Condition f1d

Slot 4, $R_a = 1.73 \mu\text{m}$

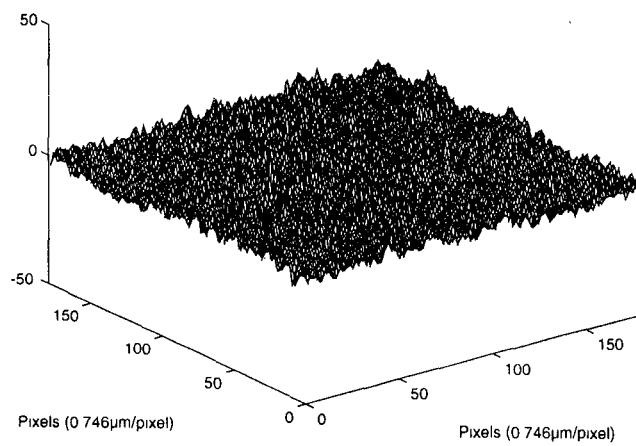
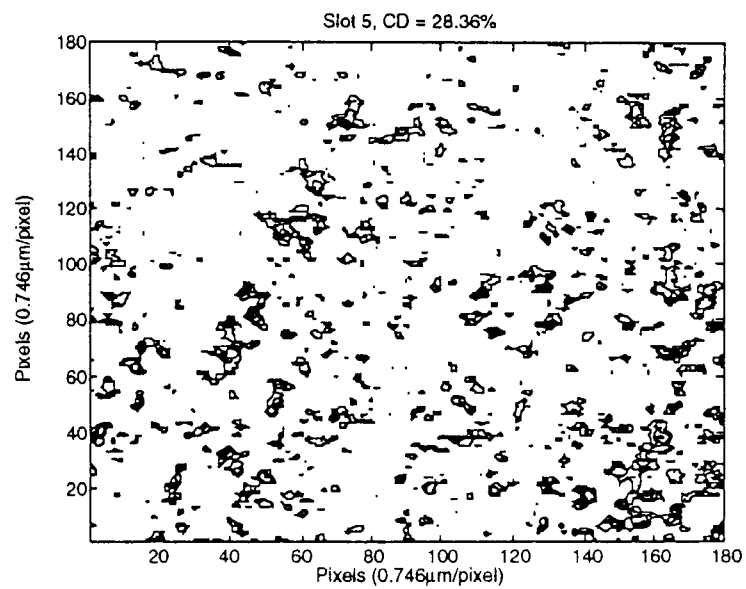
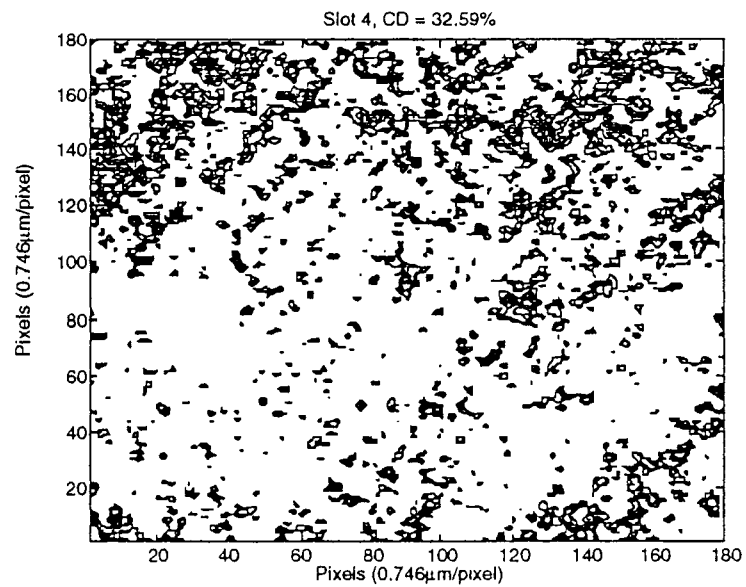


Figure 5.17 Reconstructed Surface Topography for Condition f1d (UL section)



(a) Condition f11 (Slot 5)



(b) Condition f1d (Slot 4)

Figure 5.18 Cavity Densities (f11 vs f1d), Elevation = $-1\ \mu\text{m}$

5.4.1.2.3 Milling test (1s1) versus (1sd)

The milling test corresponding to condition 1s1 is performed in slot 6. The following machining parameters are used: feed = 5.08 mm / min (0.2 in / min), spindle speed = 900 rpm, depth of cut = 0.254 mm (0.01 in). For condition 1s1 the mean values for roughness average and cavity density are $0.53 \pm 0.04 \mu\text{m}$ and $5.55 \pm 1.59 \%$. Comparison of the statistical parameters for this condition with that for slot 1 (111) provides an estimate of the role of spindle speed when a low depth of cut is used. Overall, a slight increase in roughness is observed from $0.21 \mu\text{m}$ to $0.53 \mu\text{m}$ as the spindle speed is increased. However, a more noticeable increase from 0.21% to 5.55% is observed in the cavity density as the spindle speed is increased. As before, the surface topography corresponding to area UL is reconstructed for illustrative purposes (see Figures 5.10 and 5.20).

To compare the effect of depth of cut, when the spindle speed is at the high level, cutting condition 1sd (slot 6) is compared to cutting condition 1s1. The only difference in machining parameters is the increase in depth of cut from 0.254 mm (0.01 in) condition 1s1 to 0.508 mm (0.02 in) condition 1sd. For condition 1sd, the calculated mean values for roughness average and cavity density are $0.80 \pm 0.03 \mu\text{m}$ and $15.74 \pm 0.72 \%$, respectively. Clearly, increasing the depth of cut degrades the surface integrity, and is likely to induce more machining damage for condition 1s1. A reconstructed surface topographic plot of area UL of Figure 5.21 is given in Figure 5.22. This trend is also observed in a comparison of contour plots between conditions 1s1 and 1sd indicated in Figure 5.23. As before, the contour plots are evaluated at an elevation of $1 \mu\text{m}$ below the center plane.

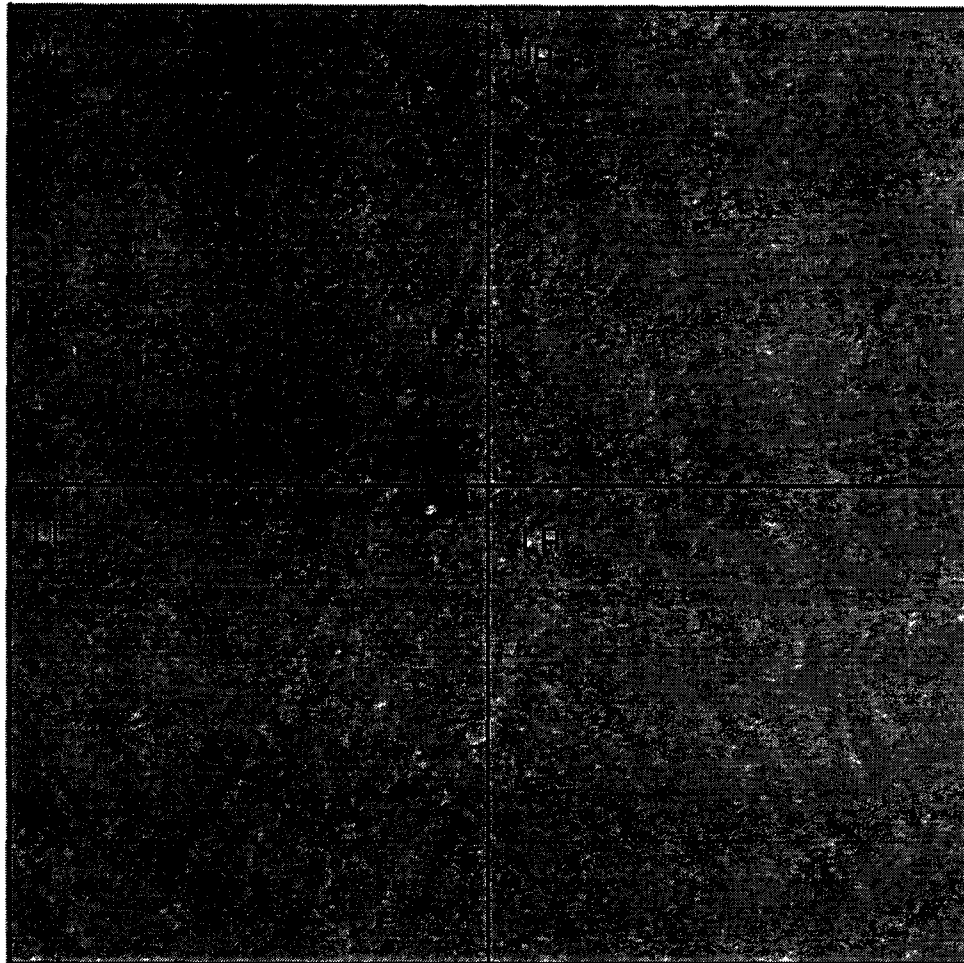


Figure 5.19 ESEM Micrograph for Cutting Condition 1s1

Slot 6, $R_a = 0.53 \mu\text{m}$

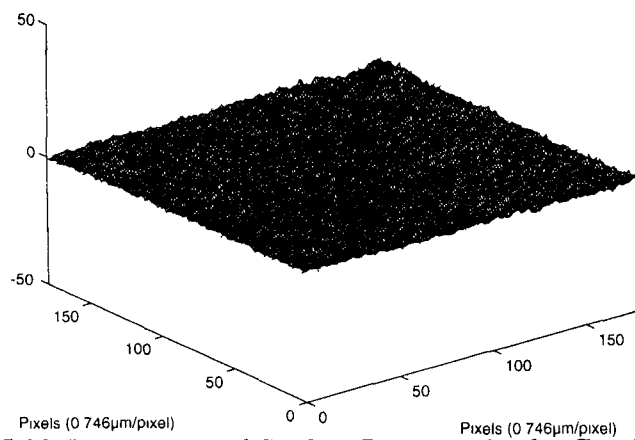


Figure 5.20 Reconstructed Surface Topography for Condition 1s1

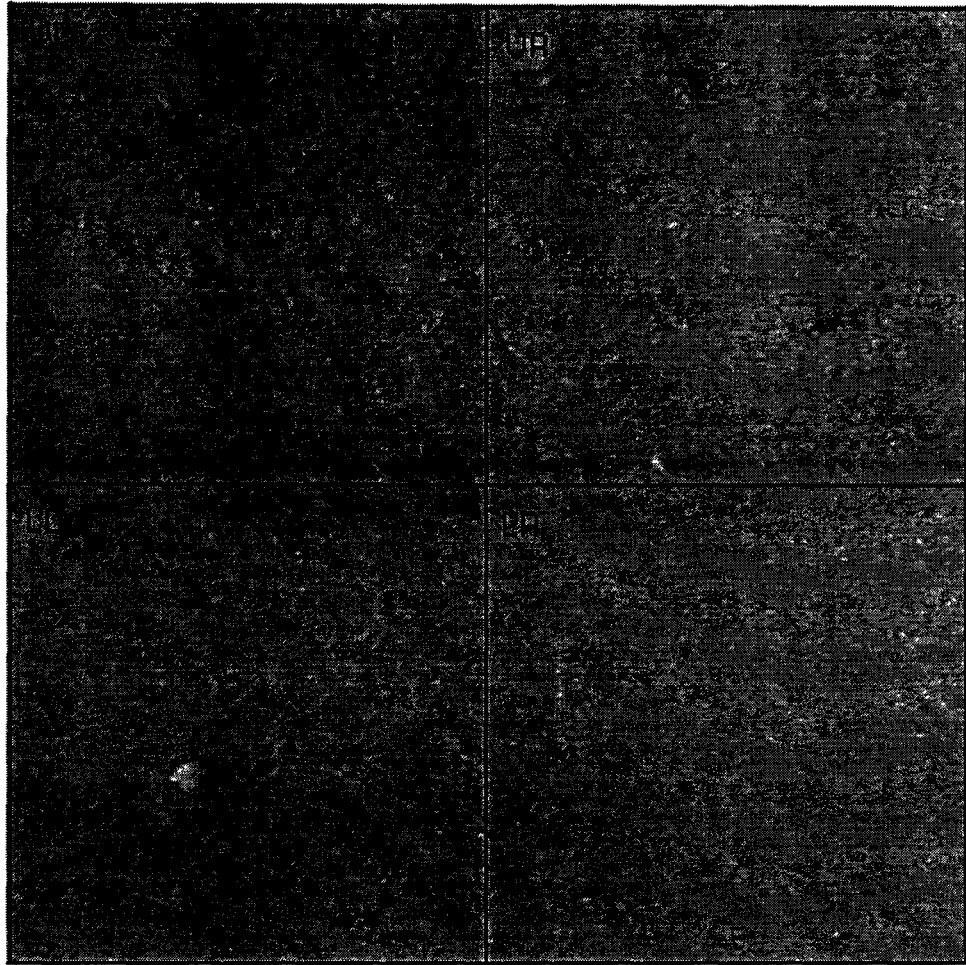


Figure 5.21 ESEM Micrograph for Cutting Condition 1sd

Slot 2, $R_a = 0.80 \mu\text{m}$

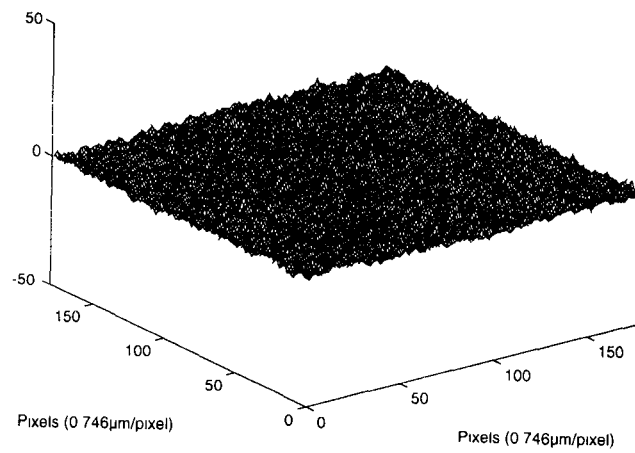
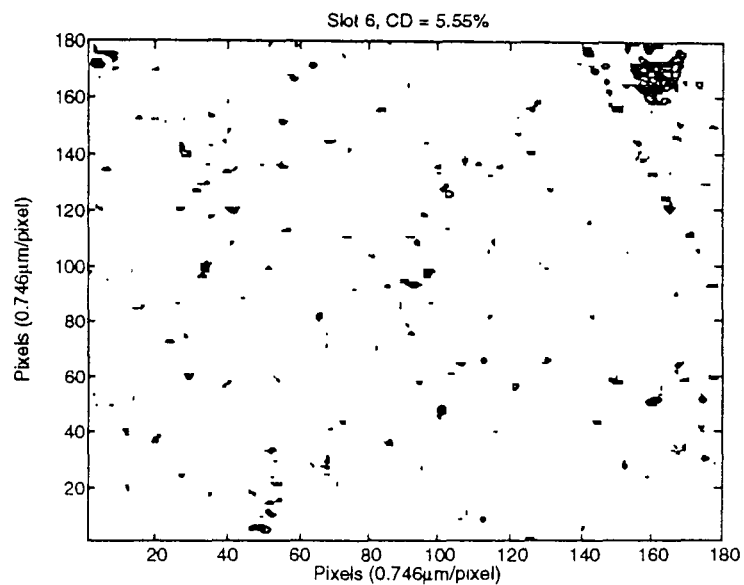
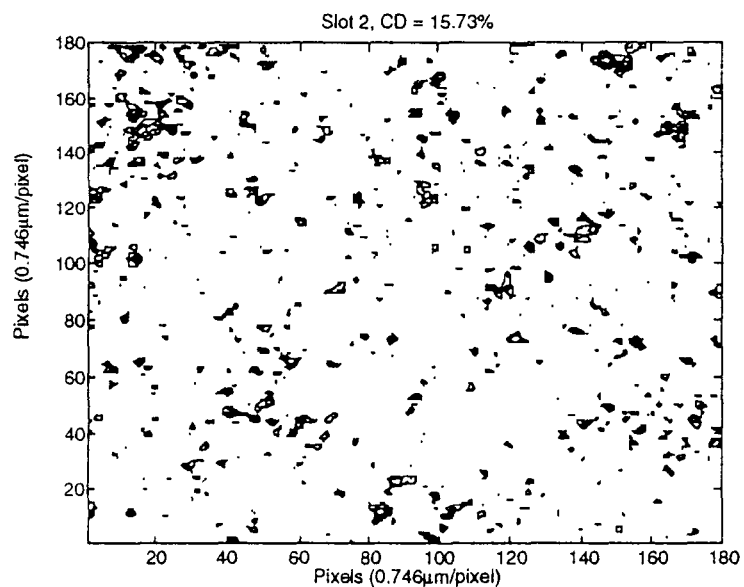


Figure 5.22 Reconstructed Surface Topography for Condition 1sd (UL section)



(a) Condition 1s1 (Slot 6)



(b) Condition 1sd (Slot 2)

Figure 5.23 Cavity Densities (1s1 vs 1sd), Elevation = -1 μ m

5.4.1.2.4 Milling test (fs1) versus (fsd)

The final series of milling experiments compare the effect of depth of cut when high levels of the other two machining parameters are used. First, for condition fs1 (slot 7), the following machining conditions are used: feed = 10.16 mm/min (0.4 in/min), spindle speed = 900 rpm, and depth of cut = 0.254 mm (0.01 in). An ESEM micrograph of the milled surface, see Figure 5.24, indicates a rougher surface than that for the mild milling condition 111. For condition fs1, the mean values of the roughness average and cavity density are $1.21 \pm 0.09 \mu\text{m}$ and $27.33 \pm 2.18 \%$. As before, area UL is reconstructed in Figure 5.25.

The final milling test to be examined is condition fsd. For this test, all machining parameters are set at their high levels: feed = 10.16 mm/min (0.4 in/min), spindle speed = 900 rpm, and depth of cut = 0.508 mm (0.02 in). As illustrated in Figure 5.26, extensive roughness and cavities can be observed in the machined surface. The machining grooves are clearly visible for this test condition. The severity of this machining parameter is also observed in the mean roughness average value ($41.59 \pm 1.63 \mu\text{m}$) and mean cavity density value ($39.93 \pm 0.66 \mu\text{m}$). A reconstructed surface plot corresponding to area UL is provided in Figure 5.27.

Finally, a comparison of contour plots evaluated at an elevation of $1 \mu\text{m}$ below the center plane provide a clear indication of the damage caused by the increase in depth of cut. As illustrated in Figure 5.28, increasing the depth of cut from 0.254 mm to 0.508 results in a corresponding increase in cavity density from 27.33% to 41.59%.

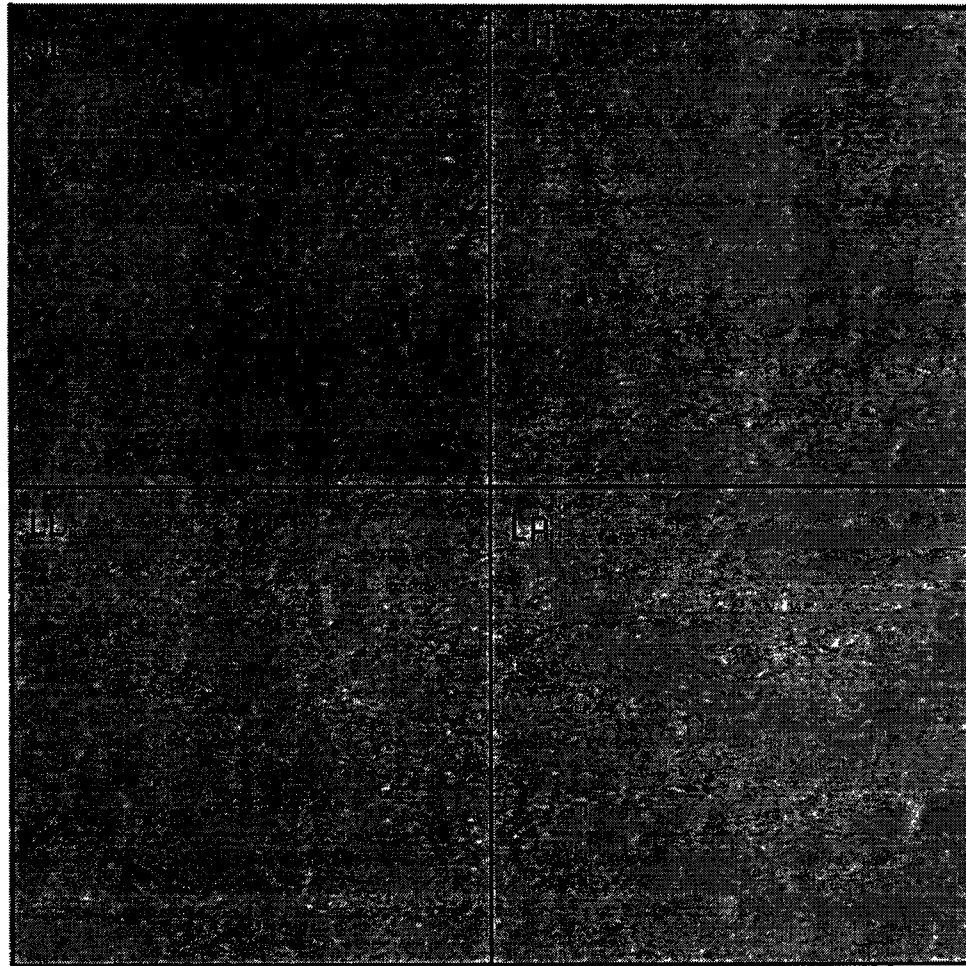


Figure 5.24 ESEM Micrograph for Cutting Condition fs1
Slot 7, Ra $\approx 1.21 \mu\text{m}$

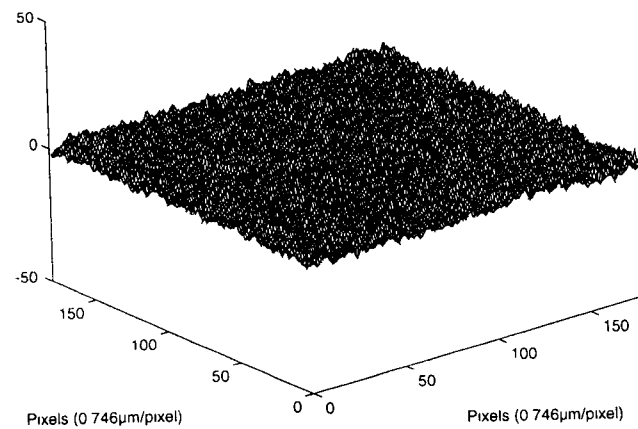


Figure 5.25 Reconstructed Surface Topography for Condition fs1 (UL section)

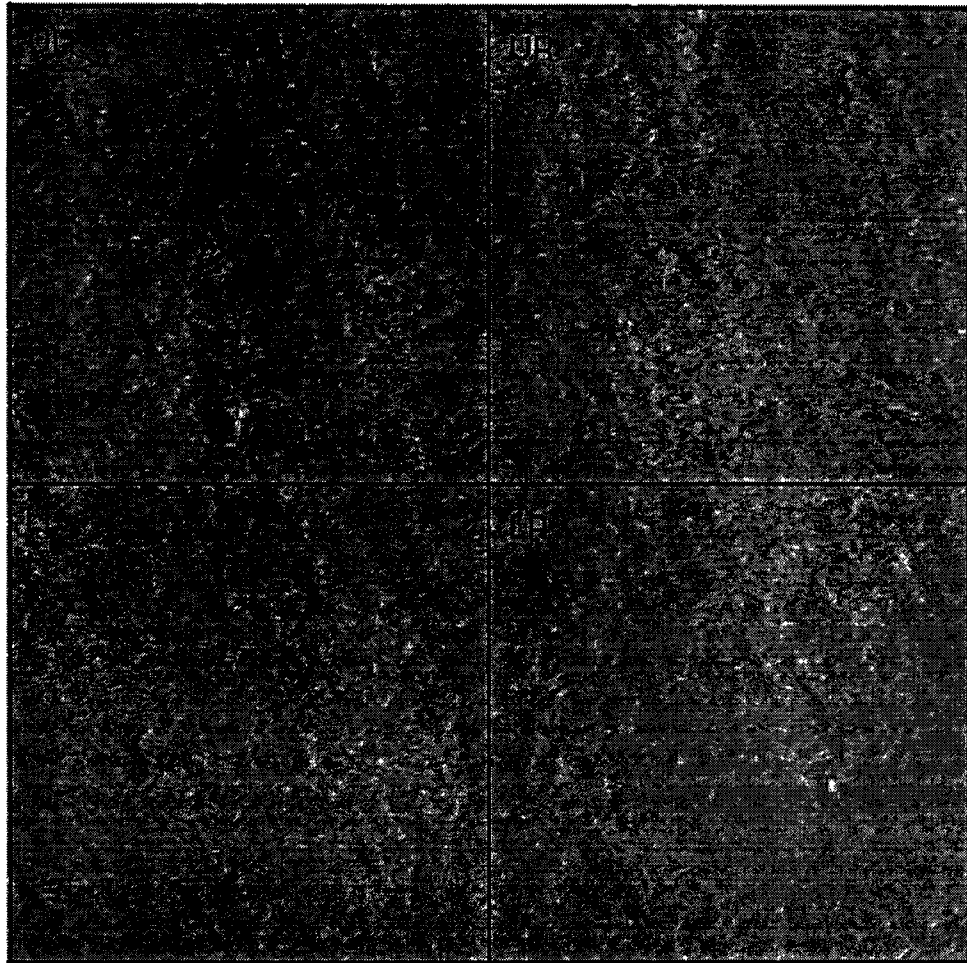


Figure 5.26 ESEM Micrograph for Cutting Condition fsd

Slot 3, $R_a = 2.85 \mu\text{m}$

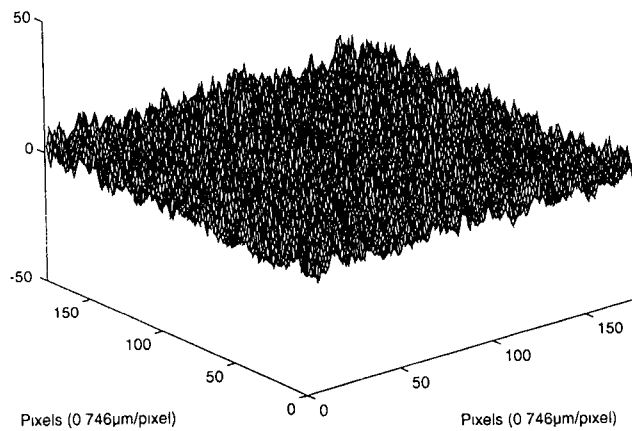
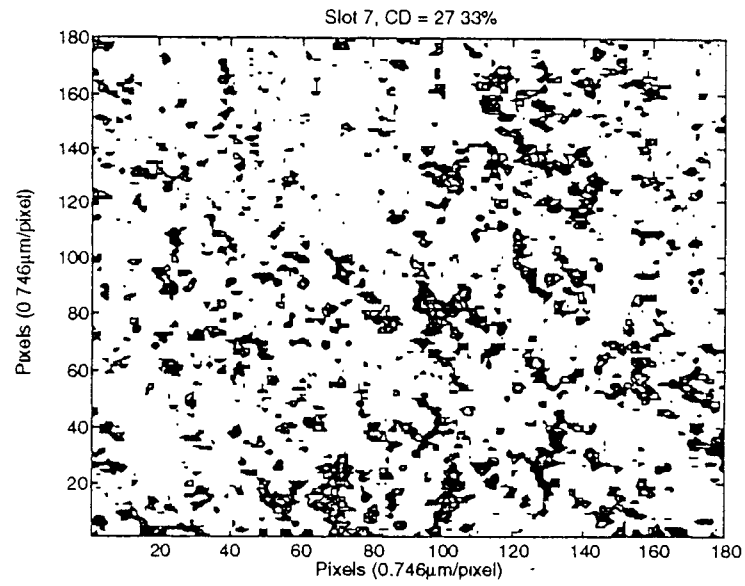
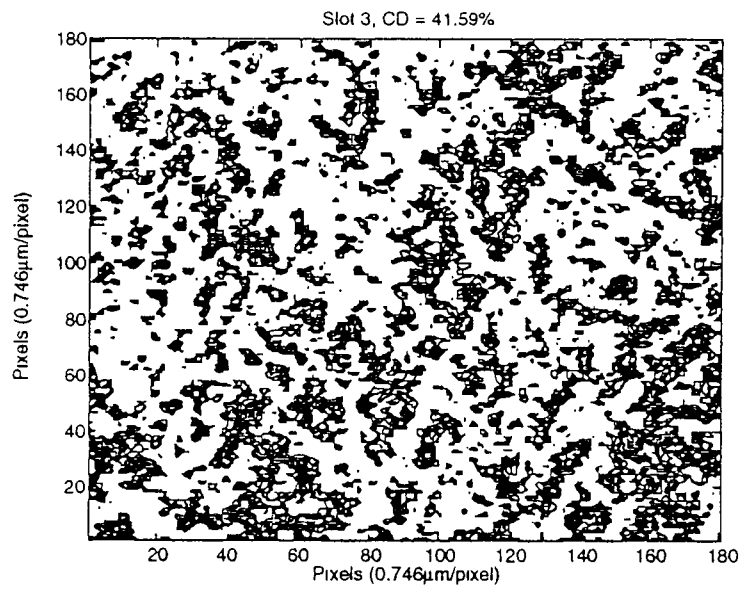


Figure 5.27 Reconstructed Surface Topography for Condition fsd (UL section)



(a) Condition fs1 (Slot 7)



(b) Condition fsd (Slot 3)

Figure 5.28 Cavity Densities (fs1 vs fsd), Elevation = -1 μ m

Overall, a summary table listing all the conditions and statistical parameters is given by Table 5.4.

Table 5.4 Summary of Milling Test Results

Condition	Slot	Parameter	UL	UR	LR	LL	Average	Std
111	8	Ra [μm]	0.24	0.23	0.20	0.18	0.21	0.03
		CD %	0.05	0.29	0.37	0.12	0.21	0.15
f11	5	Ra [μm]	1.32	1.35	1.53	1.53	1.43	0.11
		CD %	26.92	25.60	30.59	30.36	28.37	2.49
1s1	6	Ra [μm]	0.48	0.55	0.56	0.52	0.53	0.04
		CD %	4.09	7.56	6.07	4.48	5.55	1.59
fs1	7	Ra [μm]	1.10	1.27	1.29	1.17	1.21	0.09
		CD %	24.98	26.58	30.19	27.56	27.33	2.18
11d	1	Ra [μm]	0.97	0.91	0.96	0.93	0.94	0.03
		CD %	21.39	18.17	21.17	20.19	20.23	1.47
f1d	4	Ra [μm]	1.88	1.84	1.67	1.54	1.73	0.15
		CD %	38.29	37.84	36.84	36.82	37.45	0.73
1sd	2	Ra [μm]	0.78	0.82	0.82	0.77	0.80	0.03
		CD %	16.40	16.30	15.31	14.94	15.74	0.72
fsd	3	Ra [μm]	2.82	3.02	2.86	2.70	2.85	0.13
		CD %	40.28	43.86	41.68	40.55	41.59	1.63

5.4.1.3 Factorial Design Experiment using Roughness Average as the Output Variable

To further examine the main and interaction effects of the three machining parameters -- feed, spindle speed, and depth of cut -- a 2^3 factorial design experiment with $n = 4$ replicates is performed. For the experiment, the first output parameter used to quantify the effects is roughness average as defined by Equation 5.5. Using the same method described in section 4.3, the products, effects, and sum of squares are calculated and tabulated in Table 5.5. The critical level for statistical significance is selected to be $\alpha = 0.10$. An analysis of variance summary (ANOVA) table (Table 5.6) is used to determine the statistically significant main and interaction effects. When

roughness average is used as the output parameter, only three effects are significant -- the main effect of feed rate, the main effect of depth of cut, and the three factor interaction of feed rate, spindle speed, and depth of cut. Overall a model based on roughness average is given by Equation 5.7. The model indicates that the largest roughness average parameter ($R_a = 2.398 \mu\text{m}$) is observed if all three factors are set at their high levels. On the other hand, if all three factors are set at their low levels, the least amount of machining damage is introduced ($R_a = 0.026 \mu\text{m}$).

Table 5.5 Products, Effects and Sum of Squares (Output Parameter = R_a)

	Product	Effect	Sum of Squares
F	18.972	1.186	11.248
S	4.253	0.266	0.565
D	11.758	0.735	4.320
FS	2.875	0.180	0.258
FD	3.761	0.235	0.442
SD	3.571	0.223	0.398
FSD	7.215	0.451	1.627

Table 5.6 ANOVA Summary Table (Output Parameter = R_a)

Variation Source	Sum of Squares	Degrees of Freedom	Mean Square	Observed f value	P-Value	P-Value>0.10
F	11.248	1	11.248	20.651	0.000	No
S	0.565	1	0.565	1.038	0.318	Yes
D	4.320	1	4.320	7.932	0.010	No
FS	0.258	1	0.258	0.474	0.498	Yes
FD	0.442	1	0.442	0.812	0.377	Yes
SD	0.398	1	0.398	0.731	0.401	Yes
FSD	1.627	1	1.627	2.986	0.097	No
Error	13.072	24	0.545			
Total	19.056	31				

$$R_a [\mu\text{m}] = 1.212 + \frac{1.186}{2} F + \frac{0.735}{2} D + \frac{0.451}{2} FSD \quad (5.7)$$

5.4.1.4 Factorial Design Experiment using Cavity Density as the Output Variable

The second parameter used to quantify machining damage for the aforementioned 2^3 factorial design experiment is cavity density, which is calculated using Equation 5.6. As previously mentioned, this parameter provides an estimate of the required amount of finishing required to remove machine induced damage. In this thesis work, the amount of damage at a depth of $1\text{ }\mu\text{m}$ below the center plane is observed using contour plots. In these plots, enclosed regions represent cavities or damage caused by the milling process. Figure 5.13, for example, provides a comparison of the cavity density between experimental conditions 111 (slot 8) and 11d (slot 1). It can be clearly observed that more noticeable damage is caused by the increase in the depth of cut. Examination of Table 5.5 reveals that the cavity density for conditions 111 and 11d are 0.21% and 27.33% respectively. Each of the three remaining plots (Figures 5.18, 5.23, and 5.28) reveal the significance of depth of cut on machining damage. Each figure indicate reveals that increasing the depth of cut results in additional damage.

Further examination of the four plots indicates that increasing the feed rate also results in additional machining damage. Overall, the largest cavity density value ($CD = 41.59\%$) and roughness average value ($R_a = 2.85\text{ }\mu\text{m}$) are associated with most severe milling parameters (condition fsd). Similarly, the smallest cavity density value ($CD = 0.21\%$) and roughness average value $R_a = 0.21\text{ }\mu\text{m}$) are associated with the least severe milling parameters (condition 111). This indicates that the roughness average may be used to provide an estimate of machining damage.

Again, as before, the selected significance level for the statistical hypothesis is $\alpha = 0.10$. For this parameter, the products, effects and sum of squares is provided in Table 5.7. Furthermore, the ANOVA table for this parameter is provided in Table 5.8.

Table 5.7 Products, Effects and Sum of Squares (Output Parameter = CD)

	Product	Effect	Sum of Squares
F	352.611	22.038	3885.445
S	35.251	2.203	38.831
D	194.791	12.174	1185.731
FS	28.449	1.778	25.293
FD	-46.891	-2.931	68.710
SD	0.829	0.052	0.021
FSD	79.511	4.969	197.561

Table 5.8 ANOVA Summary Table (Output Parameter = CD)

Variation Source	Sum of Squares	Degrees of Freedom	Mean Square	Observed f value	P-Value	P-Value>0.10
F	3885.445	1	3885.445	22.226	0.000	No
S	38.831	1	38.831	0.222	0.642	Yes
D	1185.731	1	1185.731	6.783	0.016	No
FS	25.293	1	25.293	0.145	0.707	Yes
FD	68.710	1	68.710	0.393	0.537	Yes
SD	0.021	1	0.021	0.000	0.991	Yes
FSD	197.561	1	197.561	1.130	0.298	Yes
Error	4195.572	24	174.816			
Total	5514.159	31				

A model based on cavity density, given by Equation 5.8, indicates that only the main effects of feed and depth of cut are significant factor. In general, Equation 5.8 indicates that increasing both the feed rate and depth of cut from the low level to the high level results in the most severe damage. On the other hand, the model predicts that the least amount of damage is observed when the feed rate and depth of cut are maintained at their low levels.

$$CD[\%] = 21.45 + \frac{22.04}{2} F + \frac{12.17}{2} D \quad (5.8)$$

5.4.2 Assessment of Edge Effects

To examine machining damage, the edges of the slots are examined at locations where the cutting tool enters and exits the material. Machining damage can be particularly severe at these locations because of abrupt changes in the cutting action. For the eight unique milling experiments, a series of ESEM micrographs illustrates the machining damage at tool entry locations. As illustrated in Figure 5.29, the pictures indicate top views of the surface. Finally, an initial beam voltage of $E_0 = 30$ keV is used to obtain the images at a magnification factor of 250.

In Figure 5.30, a comparison in machining damage caused by changing the depth of cut from 0.01 in to 0.02 in is examined. First, for the least severe milling condition (111), chipping observed along the top edge of the surface at which the tool enters the workpiece is severe. However, if the depth of cut is increased to 0.02, as in condition 11d, less damage is evident on the top surface.

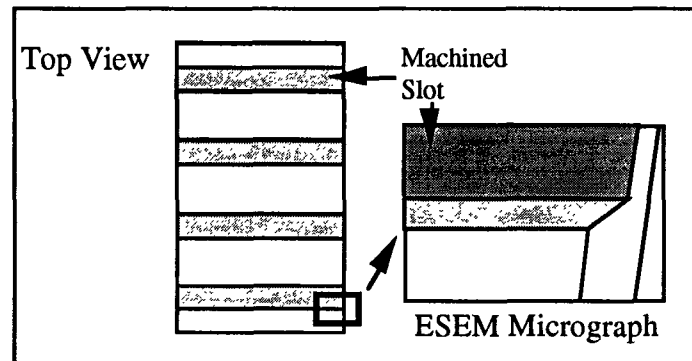


Figure 5.29 Magnification Area

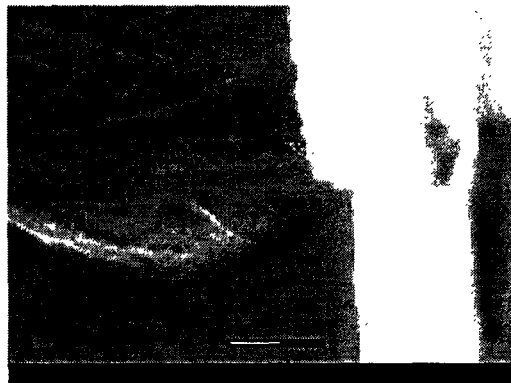


(a) Condition 111 (Slot 8)

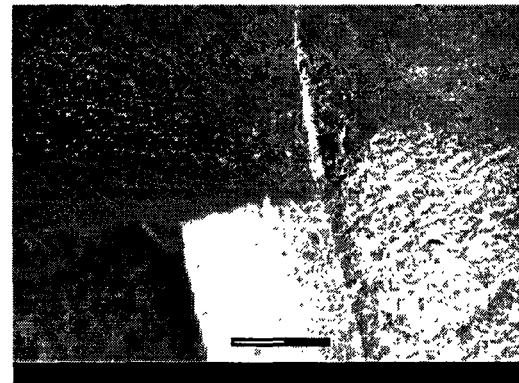


(b) Condition 11d (Slot 1)

Figure 5.30 Tool Entry Damage for Conditions 111 and 11d



(a) Condition 1s1 (Slot 6)



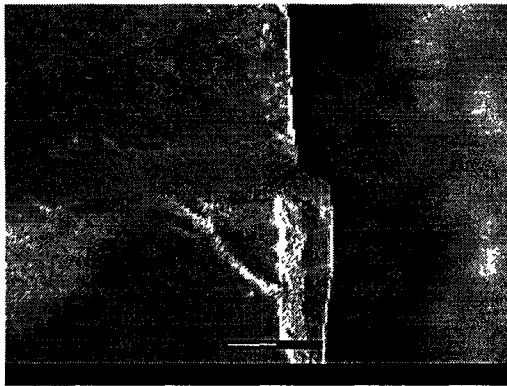
(b) Condition 1sd (Slot 2)

Figure 5.31 Tool Entry Damage for Conditions 1s1 and 1sd

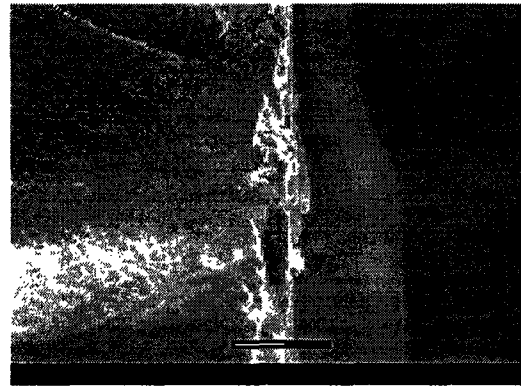
Figure 5.31 provides a comparison in machining damage caused by tool entry for conditions 1s1 and 1sd. As before, increasing the depth of cut results in less damage to the edge. This indicates that mechanism of material removal may differ for the two different conditions. When a low depth of cut is taken, more material is removed from chipping on the top surface. However, as the depth of cut is increased,

the damage on the top surface is not as significant as in the case when a low depth of cut is taken. Comparison of condition 1s1 and 111 indicates that increasing the spindle speed from 600 rpm to 900 rpm results in the generation circular chipping patterns on the top surface of the edge than when a low feed rate is used.

The final two sets of micrographs (Figures 5.32 and 5.33) correspond to the remaining set of experiments. As before, increasing the depth of cut results in improved finish along the top edge of the unmachined slot. However, comparison of condition fs1 and f11 indicates that circular chipping patterns are observed on the top surfaces for both slots. This indicates that the feed rate also plays a significant role in the material removal process. If the feed rate is set at a low level, increasing the spindle speed results in circular chipping patterns on the edge. However, if a high feed rate is used, circular patterns are discernible for regardless of the spindle speed. Overall, if a low depth of cut is used, a low spindle speed must be maintained to ensure that damage of edges caused by the tool entry motion is minimized.

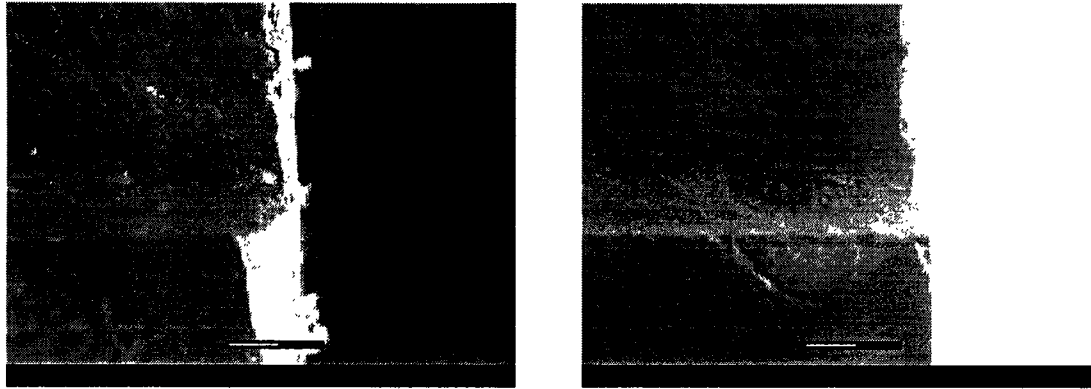


(a) Condition fs1 (Slot 7)



(b) Condition fsd (Slot 3)

Figure 5.32 Tool Entry Damage for Conditions fs1 and fsd



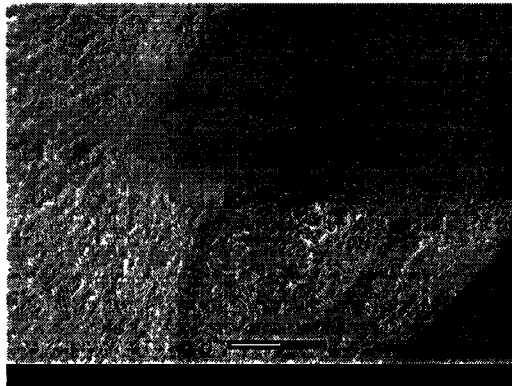
(a) Condition f11 (Slot 5)

(b) Condition f1d (Slot 4)

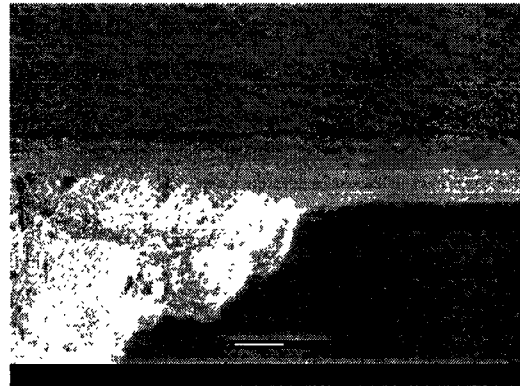
Figure 5.33 Tool Entry Damage for Conditions f11 and f1d

Overall, in ceramic components, care must be taken to ensure high quality of surface finish. If the surface integrity is poor, product reliability and maintainability become areas of high concern. In general, smooth surfaces are preferred over surfaces with irregularities. As discussed in this section, the tool entry motion can introduce significant damage along the surface edges. Furthermore, the exit motion of tool can cause even more severe machining damage along edges than the entry motion. This trend is observed for all the cutting conditions, regardless of the three machining parameters -- feed, spindle speed, and depth of cut. Figure 5.34 illustrates representative damage along the edges caused by the tool exit motion. As indicated in Figure 5.35, even the damage along the edges caused by the tool entry motion is minimized, the tool exit motion can result in severe fracture of the surface along the edges. In general, care must be taken to machine the tool exit area less aggressively than the tool entry area. Using the same machining conditions for the tool entry and exit motions can result crack initiation sites at the tool exit area. Furthermore, the edge effects at the tool exits may be alleviated if support is provided at the tool exit area. Providing additional material at the end of workpiece ensures that the workpiece will not be adversely effected when the tool exits the geometry. If left unchecked, the edges

serve as crack initiation sites which may cause fracture resulting in sudden catastrophic brittle failure of a machined component.

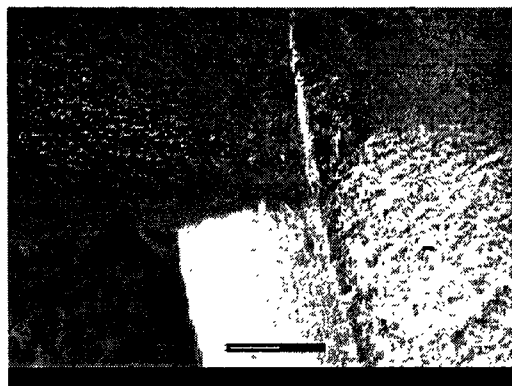


(a) Condition 1s1 (Slot 6)

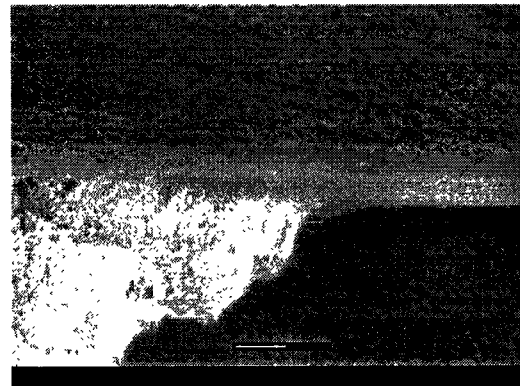


(b) Condition 1sd (Slot 2)

Figure 5.34 Tool Exit Damage for Conditions 1s1 and 1sd



(a) Tool Entry Area



(b) Tool Exit Area

Figure 5.35 Machining Damage Associated along Edges for Condition 1sd

Chapter 6

Analysis of Internal Stress Distributions

6.1 Introduction

In the previous chapter, the influence of three cutting parameters -- feed, spindle speed, and depth of cut -- on surface integrity was discussed. In this chapter, finite element analysis in two dimensional (2-D) space is performed to further examine the influence of these parameters on the internal stress distribution induced during machining. The finite element analysis (FEA) allows prediction of the stress intensity development during machining. As indicated in Figure 6.1, the proposed model consists of a rectangular workpiece with a step, representing the axial depth of cut. The feed direction is assumed to be from left to right.

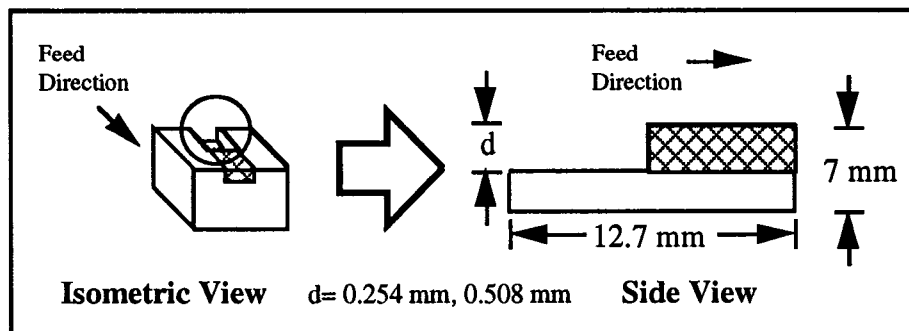


Figure 6.1 Model Geometry

Figure 6.2 provides a more detailed illustration of the geometry together with the loading conditions. A total of nine keypoints, numbered counterclockwise beginning in the lower left hand corner, are used to define the workpiece. The locations of the keypoints corresponding to a depth of cut of 0.254 mm (0.01 in) are

listed in Table 6.1. For a depth of cut of 0.508 mm (0.02 in), the vertical coordinate of keypoints 5 through 9 changes from $Y = 6.746$ mm to $Y = 6.492$ mm. The distance between keypoints 5 and 8, whose length is equal to the tool diameter, represents the tool location, i.e., the trailing edge of the cutting tool is located at keypoint 8 and the leading edge is located at keypoint 5. Overall, as illustrated in Figure 6.3, the meshed area around keypoints 4, 5, 6, 7, and 8 is more refined than in other regions since the largest stress gradients are expected to occur in the neighborhood of these points. Furthermore, the measured tangential (F_t) and normal forces (F_n) are applied as distributed loads which are indicated in the figure. The base of the workpiece is constrained from movement in the horizontal and vertical directions.

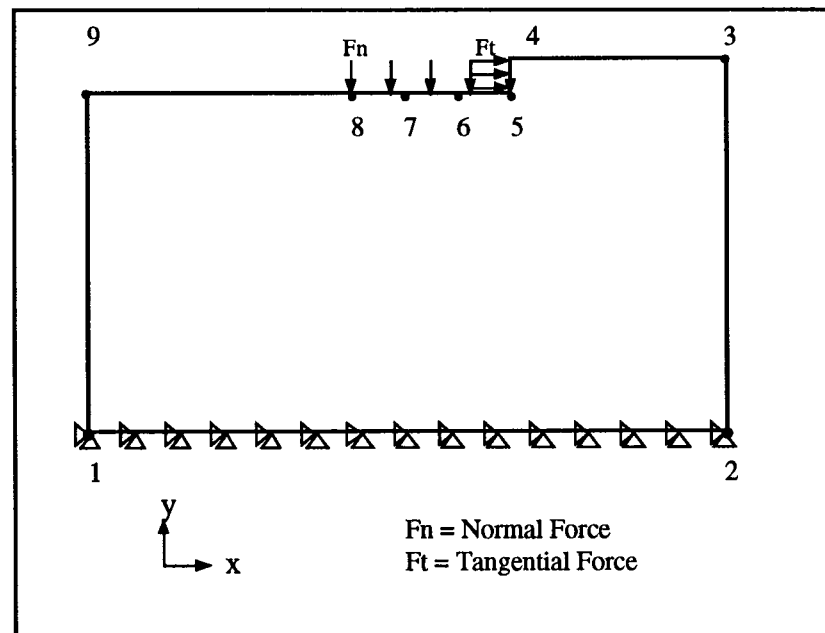


Figure 6.2 Loading Configuration

A commercial software package -- ANSYS [24] -- is used to perform the two dimensional finite element analysis (FEA). In two dimensional FEA, stress intensity is calculated using the constitutive elasticity equations of either plane stress or plane

strain. The selection of plane stress or plane strain equations is determined by the geometry of the test specimen and the loading conditions. Plane strain equations are used when modeling long members which are uniformly laterally loaded over their lengths. As indicated in Figure 6.4, each cross section of the member is assumed to undergo identical deformations. Overall, the underlying assumption indicates that strains associated with the normal direction are negligible ($\epsilon_z = \gamma_{xz} = \gamma_{yz} = 0$). On the other hand, Figure 6.5 illustrates loading conditions for which the use of plane stress equations is valid. Plane stress equations are applicable to thin members which are laterally loaded (along the edges) and are based on the following assumptions. First, since the member is thin, the normal force component is negligible ($F_z = 0$). Furthermore, the lateral forces are assumed to be functions of only the horizontal and vertical directions ($F_x = f(x,y)$ and $F_y = g(x,y)$). Based on these assumptions, the stresses associated with the normal direction are negligible ($\sigma_z = \tau_{xz} = \tau_{yz} = 0$) [25].

Table 6.1 Location of Nodes for Depth of Cut = 0.254 mm (0.01 in)

Node	X Coordinate [mm]	Y Coordinate [mm]
1	0.000	0.000
2	12.700	0.000
3	12.700	7.000
4	8.400	7.000
5	8.400	6.746
6	7.600	6.746
7	6.800	6.746
8	6.000	6.746
9	0.000	6.746

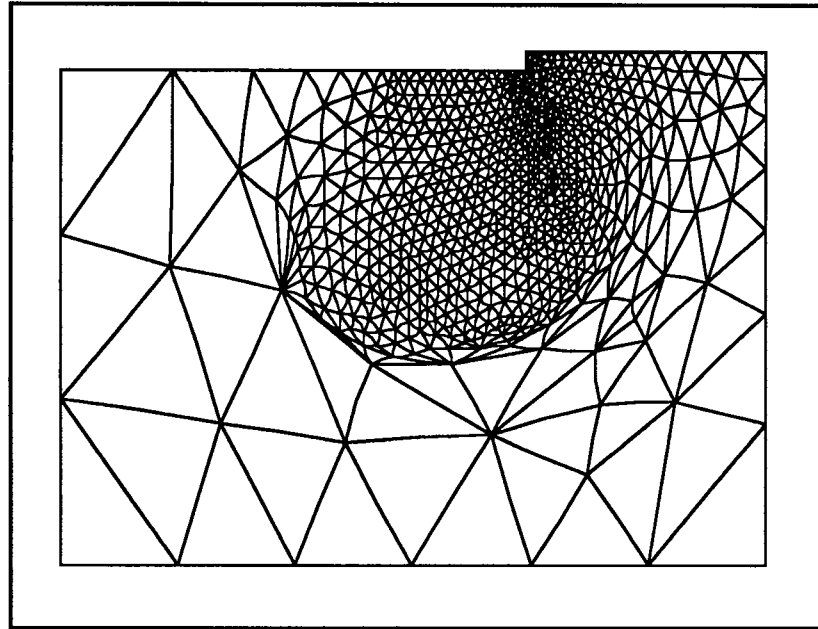


Figure 6.3 Meshed Representation of Model

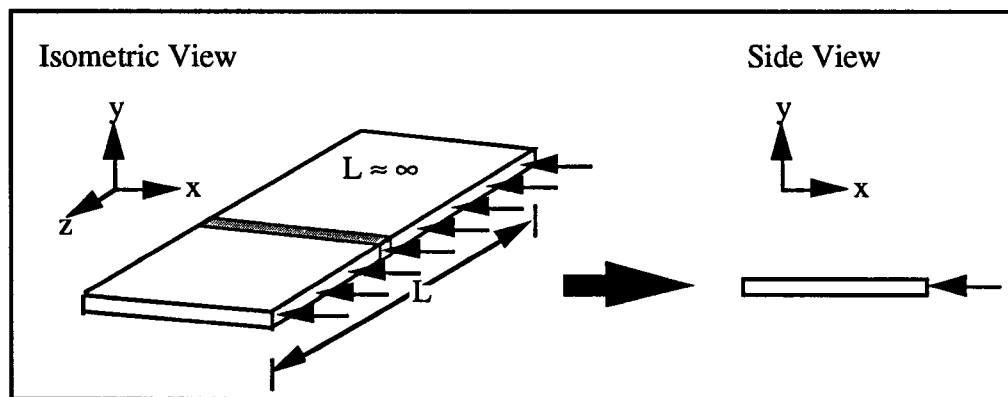


Figure 6.4 Plane Strain Loading Condition

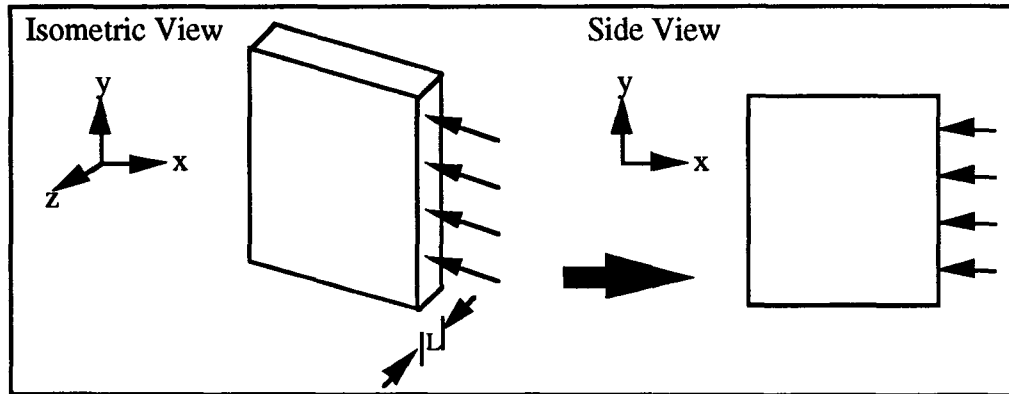


Figure 6.5 Plane Stress Loading Condition

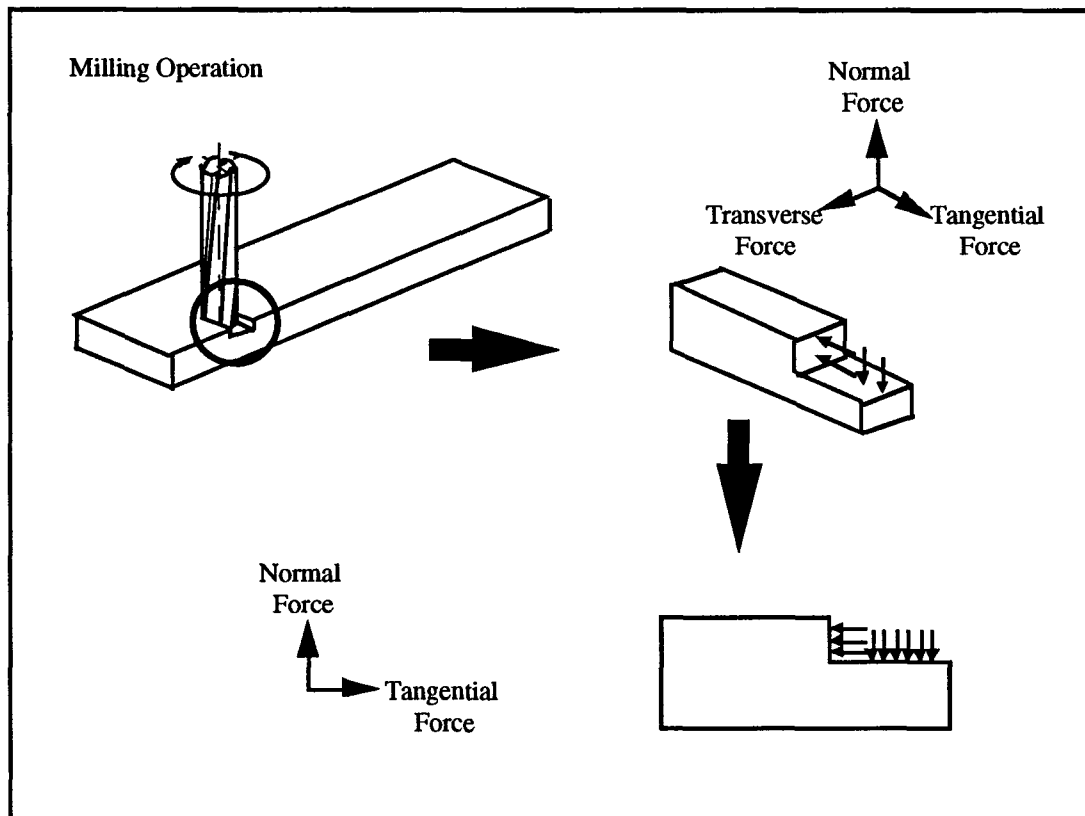


Figure 6.6 Selection of Plane Stress Equations

For the geometry indicated in Figures 6.1 and 6.6, the loading conditions indicate that a plane stress solution is appropriate since the section of a machined slot is relatively thin, i.e., the machined slot does not have an infinite length ($L \neq \infty$). Based on the plane stress assumptions, the out of plane force component is assumed to be negligible (i.e., $F_z = 0$ in Figure 6.5). Hence, as indicated in Figure 6.6, only the normal and axial (tangential) force components are used in the simulation. A comparison of the forces provided in Chapter 5 indicates that, in general, the tangential and normal force components have larger magnitudes than the transverse force component. Overall, triangular structural plane stress elements with six nodes (allowing two displacements at each node) are used to mesh the geometry (see Figure 6.7 [24]). Furthermore, out of plane loading conditions cannot be supported by the two dimensional structural plane stress elements.

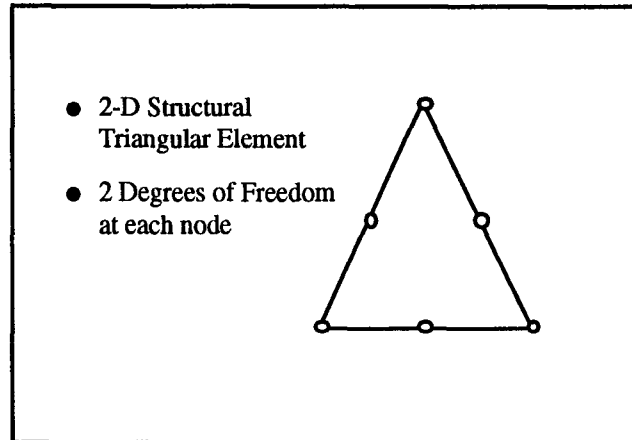


Figure 6.7 Triangular Elements Used in Finite Element Analysis

The pertinent material properties of DICOR/MGC material used in the FEA are provided in Table 6.2 [4]. Finally, numeric values for the normal (F_n) and tangential (F_t) applied force components are listed in Table 6.3. To provide more

accurate loading conditions the force component are converted to uniform pressure loads and applied as indicated in Figure 6.2. The use of point loads distributed across the surfaces results in stress concentrations at the points of application. The application of uniformly distributed pressure loads eliminates that the problem of stress concentrations. Overall, the normal force component is applied as a uniformly distributed vertical pressure load to the line segment between keypoints 5 and 8. On the other hand, the tangential force component is applied as a uniformly distributed horizontal pressure load to the line segment between keypoints 4 and 5.

Table 6.2 Material Properties

Property	Unit	Value
Modulus of Elasticity	Pa	$6.8(10)^{11}$
Poisson's Ratio	-	0.29
Density	kg/m ³	2800

Table 6.3 Loading Conditions

Feed [in/min]	Spindle Speed [rpm]	Depth of Cut [in]	Slot	Tangential Force [N]	Normal Force [N]
0.2	600	0.01	8	9.19	1.47
0.4	600	0.01	5	9.91	3.51
0.2	900	0.01	6	8.77	2.89
0.4	900	0.01	7	12.65	2.99
0.2	600	0.02	1	11.49	6.66
0.4	600	0.02	4	24.29	1.85

0.2	900	0.02	2	16.84	4.94
0.4	900	0.02	3	23.49	3.89

6.2 Design Criteria

As discussed in previous chapters, the reliability of ceramic components is of utmost importance. The presence of cracks and microcracks in machined parts can significantly degrade the strength and overall life of the designed component. In ceramics, the predominant mode of failure is fracture. For the propagation of cracks originating from surface flaws (Figure 6.8 [26]), the theoretical stress intensity factor K_I is given by Equation 6.1 [26]. The parameter K_I is a function of the system configuration dependent on the geometry and loading conditions. Conversely, the fracture stress σ_c required for catastrophic failure of the component is related to the fracture toughness K_{IC} , a material property. At the instant of fracture, the stress intensity factor K_I given in Equation 6.1 is equivalent to, or exceeds, the fracture toughness K_{IC} (i. e., at fracture, $K_I \geq K_{IC}$) [26]. Furthermore, in Equation 6.1, the stress intensity and crack length required for fracture are denoted as σ_c and a_c , respectively.

$$K_I = 1.12 \sigma \sqrt{\pi a} \quad (6.1)$$

where K_I represents the theoretical stress intensity factor

σ represents the stress intensity

a represents the crack length

For the theoretical analysis, during the machining process, the mode of material removal is assumed to consist of both plastic deformation and brittle fracture.

As illustrated in the Chapter 5, the presence of fracture surfaces and plow marks indicate that both of the material removal mechanisms are involved. The length of the cracks in the layer of material removed during the milling operation is calculated using Equation 6.1. In this equation, the stress intensity factor K_I is set to the material fracture toughness ($K_{IC} = 1.5 \text{ MPa}\cdot\text{m}^{1/2}$ [4]) indicating that brittle fracture has occurred.

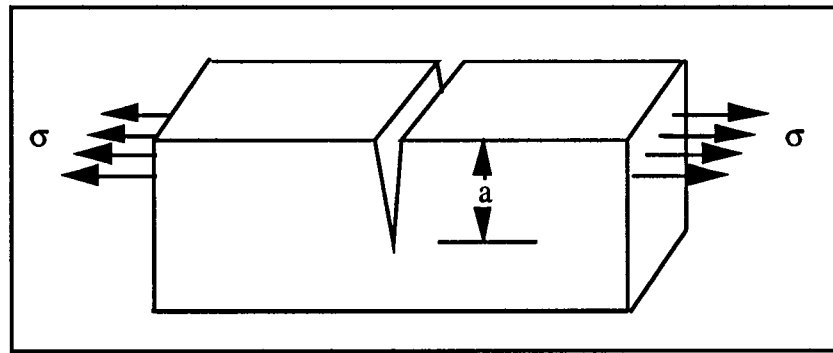


Figure 6.8 Crack Propagation from a Surface Flaw

To determine the threshold stress intensity governing the formation of cracks an additional assumption is imposed. The length of cracks is assumed to be equal to the thickness of the layer of material removed (depth of cut), i.e., it is assumed that cracks do not extend into the machined surface. Then, using the obtained stress intensities, given fracture toughness and Equation 6.1, the threshold stress intensity is calculated. Table 6.4 illustrates the procedure involved in calculating the threshold stress intensity for the two different depths of cut used.

Table 6.4 reveals that for a depth of cut = 0.254 mm and a fracture toughness of $K_{IC} = 1.5 \text{ MPa}\cdot\text{m}^{1/2}$, the critical stress intensity threshold lies between 36 MPa and 54.1 MPa. Stress intensities less than this critical value result in cracks with lengths which are larger than the depth of cut, i.e., cracks will extend into the machined surface. By using interpolation among the values represented in the Table, the critical

stress intensity thresholds for depths of cut of 0.254 mm and 0.508 mm are calculated to be $\sigma_c = 47.41$ MPa and $\sigma_c = 33.52$ MPa, respectively.

Table 6.4 Calculation of Threshold Stress Intensity for Two Different Depths of Cut

Stress Intensity [MPa]	Crack Length [mm]
0.016	2230265.851
9	7.049
18	1.762
27	0.783
36	0.441
54.1	0.195
63.1	0.143
81.1	0.087

Table 6.5 Resolved Force Components

Slot	Condition			Tn Force [N]	Tr Force [N]	Nm Force [n]	Rs Force [N]
	F	S	D	Avg	Avg	Avg	
1	-	-	+	11.49	3.64	6.66	13.77
2	-	+	+	16.84	1.98	4.94	17.66
3	+	+	+	23.49	5.43	3.89	24.42
4	+	-	+	24.29	9.22	1.85	26.05
5	+	-	-	9.91	2.34	3.51	10.77
6	-	+	-	8.77	1.83	2.89	9.41
7	+	+	-	12.65	2.44	2.99	13.23
8	-	-	-	9.19	7.82	1.47	12.16

Tn = Mean Tangential Force

Tr = Mean Transverse Force

Nm = Mean Normal Force

Rs = Resolved Force

Table 6.5 provides a listing of the tangential, transverse, normal, and resolved force components. In the table, the resolved force is defined as indicated in Equation 6.2.

$$Rs = \sqrt{Tn^2 + Tr^2 + Nm^2} \quad (6.2)$$

where Rs represents the resolved force

Tn represents the average tangential force

Tr represents the mean transverse force

Nm represents the mean normal force

Next, a the critical stress threshold intensity σ_c is used to predict the resolved cutting forces using Equation 6.3. The predicted forces are then compared to actual measured forces to examine the role of the machining parameters.

$$Rs_{pred} = \sigma_c A_c k \quad (6.3)$$

where Rs_{pred} represents the predicted resolved force

σ_c represents the critical stress threshold intensity

k represents the constant scale factor (0.25)

A_c represents the cutting area ($A_c = \pi r D$)

r represents the radius of the tool

D represents the depth of cut

6.3 Case I: Experiments with Low Depth of Cut (0.254 mm)

For the set of four experiments corresponding to the low depth of cut , a resolved force of a $Rs = 15.01$ N, corresponding to a depth of cut $D = 0.254$ mm and critical stress threshold intensity of $\sigma_c = 47.41$ MPa, is predicted. However, a comparison between the actual and predicted resolved forces indicates that the actual resolved forces for the different cutting conditions are smaller than the predicted resolved force. This indicates that both spindle speed and feed rate also effect the critical stress threshold intensity. To further quantify the role of all three machining

parameters, finite element analysis is performed. A model with coordinates listed in Table 6.1 is used. The geometry is meshed with 1432 two dimensional triangular plane stress elements. The output variable used to characterize the effects of the different machining parameters is the stress intensity. As indicated in Figure 6.9, the maximum stress concentration occurs at keypoint 5 -- the corner point on the edge of the step shown in Figure 6.4. Overall, the stress distribution patterns corresponding to the four tests remains relatively unchanged. However, as indicated in Table 6.6, the intensity of stress distribution increases as the input cutting force load is increases.

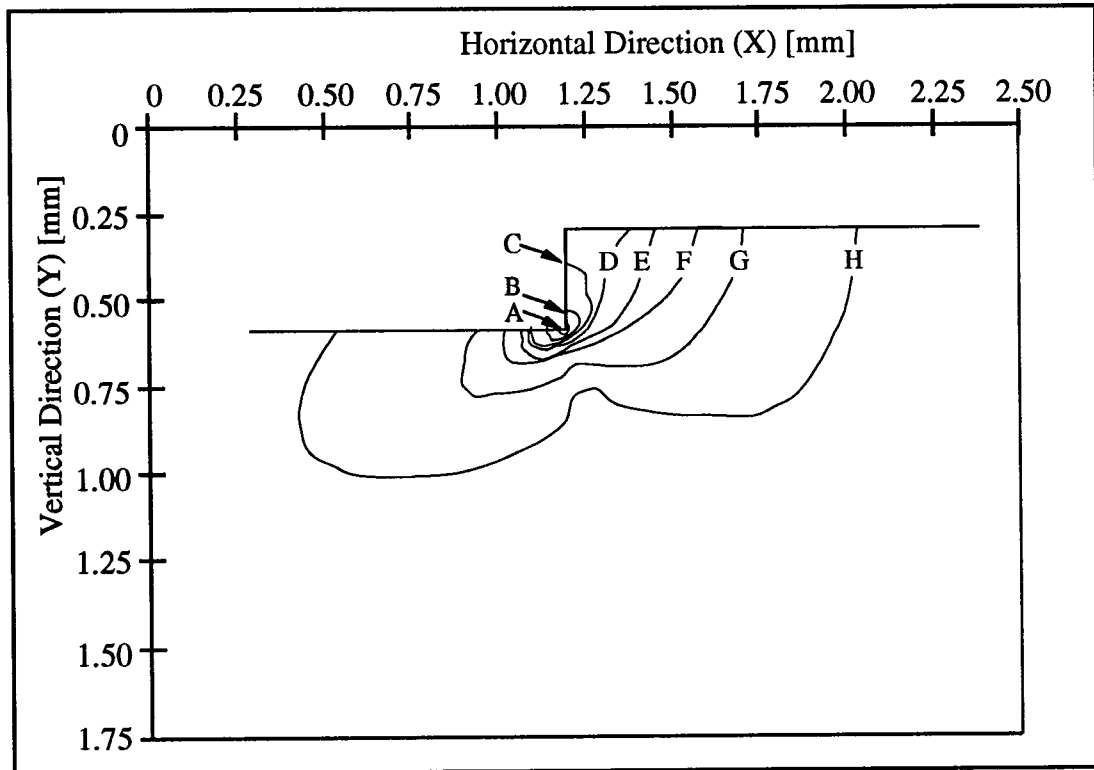


Figure 6.9 Stress Distribution Plot for Experiments with Depth of Cut = 0.254 mm
(0.01 in)

Table 6.6 Stress Distribution for Experiments with Depth of Cut = 0.254 mm

Condition*	Slot	Stress Intensity [MPa]							
		A	B	C	D	E	F	G	H
- - -	8	58.8	45.7	39.2	26.1	19.6	13.1	6.5	0.0123
+ - -	5	63.8	49.6	42.5	28.3	21.3	14.2	7.1	0.0114
- + -	6	56.3	43.8	37.5	25.0	18.8	12.5	6.3	0.0108
+ + -	7	81.1	63.1	54.1	36.0	27.0	18.0	9.0	0.0160
* Condition: Feed - Spindle Speed - Depth of Cut, "+" = High Level, "-" = Low Level									

Based on the calculated stress intensity threshold, plots of the stress distribution in the horizontal and vertical directions are constructed to examine the role of the different cutting parameters (feed, depth of cut, and spindle speed). Figure 6.10 indicates the two directions in which stress distribution plots are constructed. In the figure, the horizontal and vertical stress directions are denoted by labels X and Y, respectively. Distributions of the stress intensity variation in the horizontal and vertical directions are provided in Figures 6.11 and 6.12, respectively. In the plots, labels consisting of positive (+) and negative (-) are used to denote the test condition. The first position of a given label denotes the level of the feed (+ denoting the high level and vice versa). Likewise, the second and third positions indicate the level of the spindle speed and depth of cut, respectively. In the graphs, a logarithmic curve fit is applied to the set of data for each distribution.

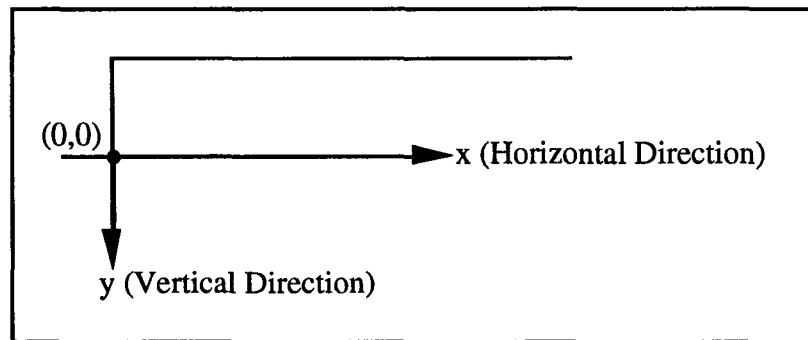


Figure 6.10 Definition of Directions for Stress Distribution Plots

Examination of the stress distribution (Figure 6.11) in the horizontal direction corresponding to the different milling tests reveals that the most severe condition in terms of generation of cracks is condition ++- (feed = 0.4 in/min, spindle speed = 900 rpm, depth of cut = 0.01 in). The next test of significant corresponds to condition +-+ (feed = 0.4 in/min, spindle speed = 600 rpm, depth of cut = 0.01 in). Hence, reducing the spindle speed, when a high feed rate and low depth of cut are used, results in a corresponding decrease in the stress intensity. The two remaining tests, condition -+- (feed = 0.2 in/min, spindle speed = 900 rpm, depth of cut = 0.01 in) and condition --- (feed = 0.2 in/min, spindle speed = 600 rpm, depth of cut = 0.01 in), have approximately equivalent stress distributions.

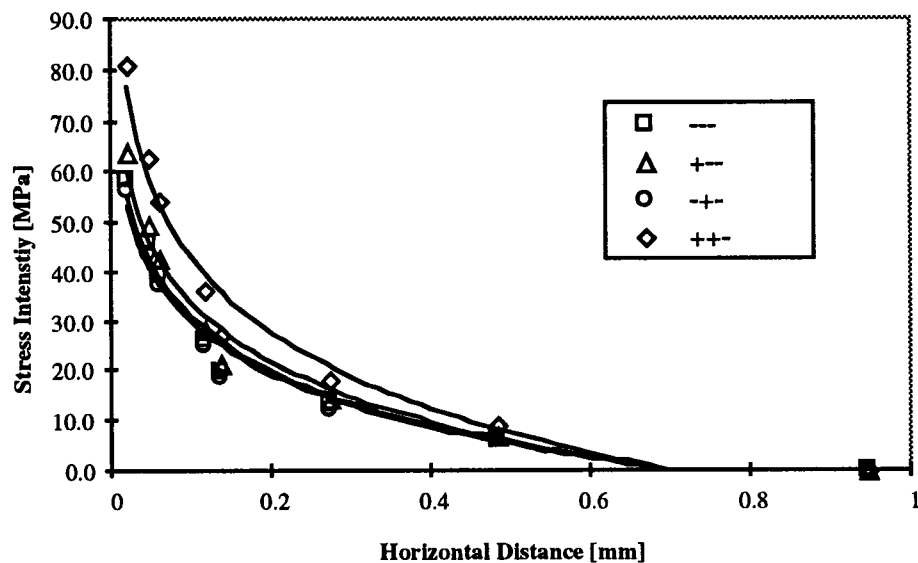


Figure 6.11 Intensity of Stresses in Horizontal Direction (Depth of Cut = 0.254 mm)

Figure 6.12 provides a distribution of the stress distribution in the vertical corresponding to the four milling experiments. Overall, as before, the most severe milling test corresponds to condition ++-. However, for the vertical direction, the three remaining conditions (+--, -+- and ---) do not vary significantly. Furthermore, a

comparison between Figures 6.11 and 6.12 reveals that the stress distribution in the feed direction dissipates more gradually than that in the normal direction. This observation results from the applied loading conditions. In all of the experiments, the tangential force component is significantly larger than the normal force component.

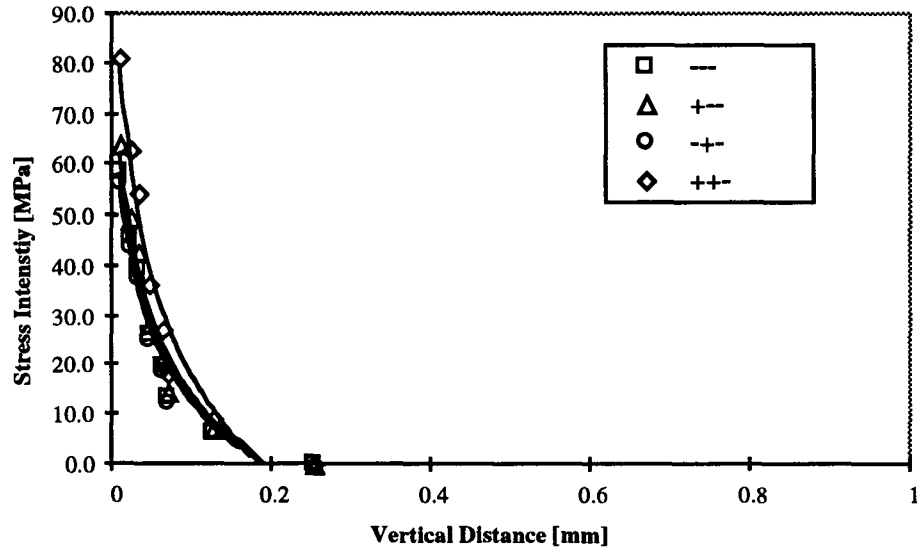


Figure 6.12 Intensity of Stresses in the Vertical Direction (Depth of Cut = 0.254 mm)

6.4 Case II: Experiments with High Depth of Cut (0.508 mm)

A resolved force of a $R_s = 21.23$ N, corresponding to a depth of cut $D = 0.508$ mm and critical stress threshold intensity of $\sigma_c = 47.41$ MPa, is predicted for remaining four experiments. As before, a comparison of the actual resolved forces for the different cutting conditions deviate from the predicted resolved force. When a low feed rate is used, the actual resolved forces are lower than the predicted resolved force. However, when a large feed rate is used, the actual resolved forces measured are larger than the predicted resolved force. This indicates that the feed plays a significant role in the determination of the critical stress intensity threshold.

Finite element analysis provides further insight about the role of the machining parameters on the stress distributions. For the four experiments corresponding to a large depth of cut, the model described in Table 6.1 is slightly modified to accommodate the set of four experiments corresponding to the high depth of cut ($D = 0.02$ in). In this model, the vertical axis coordinate (Y coordinate) of keypoints 5 through 9 is changed from $Y = 6.746$ mm to $Y = 6.492$ mm. Altogether, the new geometry is meshed with 1290 two dimensional triangular plane stress elements. As before, the output variable used to quantify the role of the machining parameters is the stress intensity. Figure 6.13 indicates, as before, that the maximum stress concentration occurs at keypoint 5 -- the corner point on edge of the step shown in Figure 6.2. The stress distribution pattern does not significantly vary among the four tests.

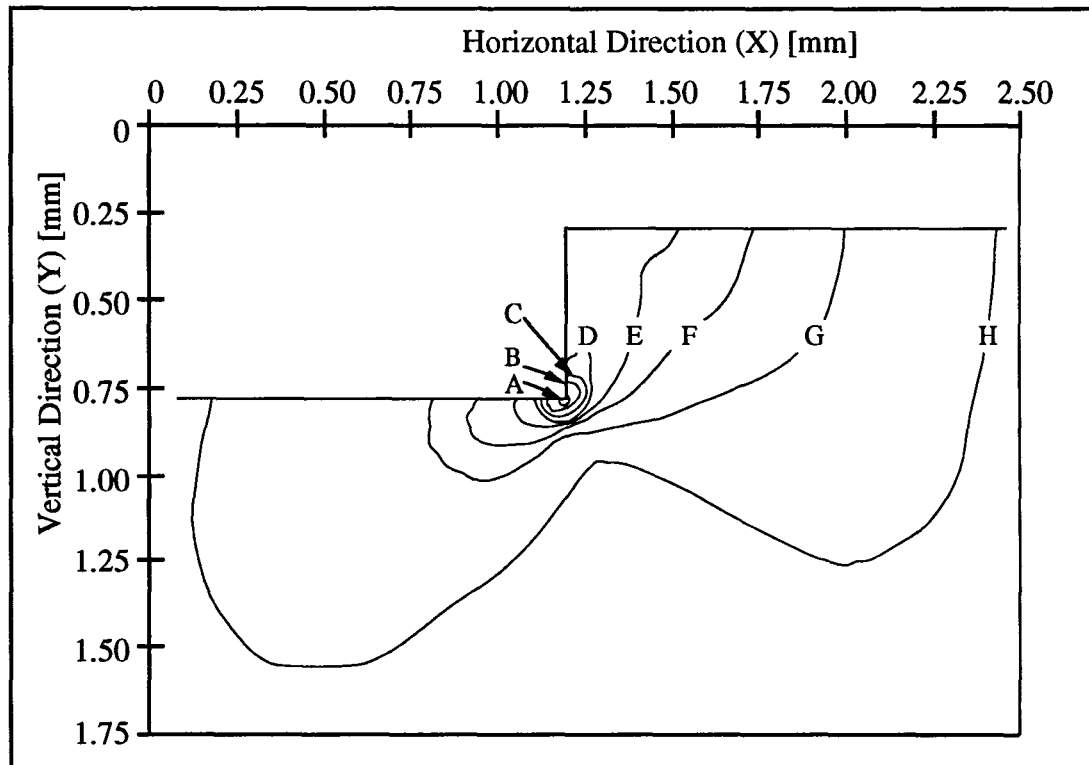


Figure 6.13 Stress Distribution Plot for Experiments with Depth of Cut = 0.508 mm
(0.02 in)

However, a comparison of Figure 6.9 (depth of cut = 0.254 mm) to Figure 6.13 (depth of cut = 0.508 mm) reveals that the stress intensities corresponding to the large depth of cut experiments have noticeably higher magnitudes than those corresponding to the low depth of cut. Furthermore, the intensity dissipates a slower rate for the large depth of cut experiments, i.e., the stress intensity remains high over a greater area for the large depth of cut experiments when compared to the low depth of cut experiments. Table 6.7 tabulates the intensity of the stress distribution corresponding to the labels denoted in Figure 6.13. As before, the stress intensity is proportional to input forces.

Table 6.7 Stress Distribution for Experiments with Depth of Cut = 0.508 mm

		Stress Intensity [MPa]							
Condition*	Slot	A	B	C	D	E	F	G	H
-- +	1	50.9	39.6	34.0	22.7	17.0	11.3	5.7	0.0174
+ - +	4	104.0	81.2	69.6	46.4	34.8	23.2	11.6	0.0306
- + +	2	73.4	57.1	48.9	32.6	24.5	16.3	8.2	0.0263
+ + +	3	102.0	79.0	67.7	45.2	33.9	22.6	11.3	0.0269
* Condition: Feed - Spindle Speed - Depth of Cut, "+" = High Level, "-" = Low Level									

Figure 6.14, a plot of the stress distributions in the horizontal direction, reveals that two conditions result in high stress intensities. First, condition +++ (feed = 0.4 in/min, spindle speed = 900 rpm and depth of cut = 0.02 in) results in high stress intensities. This observation matches that the finding from the previous set of experiments in which high stress intensities were observed for condition ++-. However, when a large depth of cut is taken, the high stress intensities are also observed for condition +-+ (feed = 0.4 in/min, spindle speed = 600 rpm and depth of

cut = 0.02 in). Hence, spindle speed does not seem to have a large effect on the stress distribution in the horizontal direction when feed and depth of cut are set at their high levels. Second, the remaining two conditions result in noticeably different distributions. After conditions +++ and ++-, condition -++ results in high stress intensities. Finally, condition --+ seems to result in the least amount of damage.

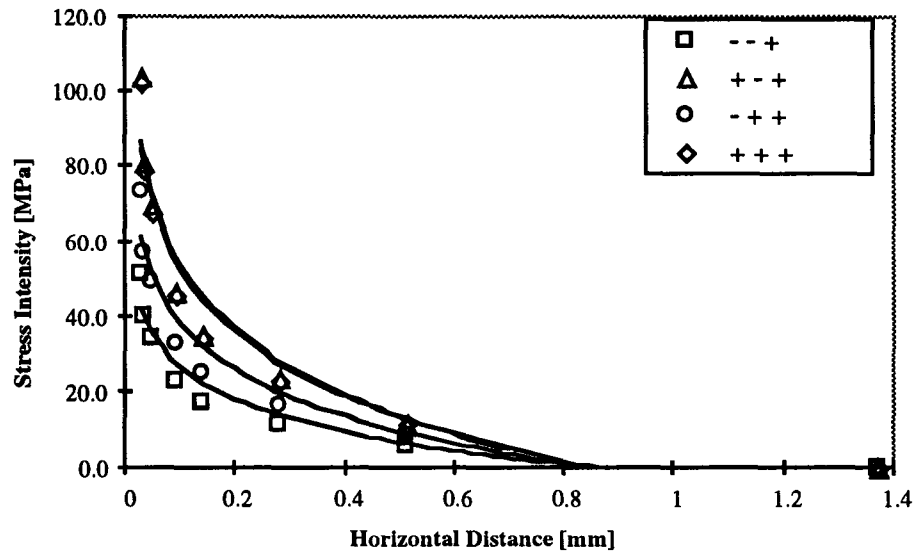


Figure 6.14 Intensity of Stresses in the Horizontal Direction(Depth of Cut = 0.508 mm)

Figure 6.15 provides a distribution of the stresses in the vertical direction. As before, the intensity of stresses for the different milling experiments in the vertical direction dissipates more rapidly than the intensity of stresses in the horizontal. Furthermore, for both the directions, the highest and lowest stress intensities are observed with conditions +-+ and +++, and conditions -++ and --+ , respectively.

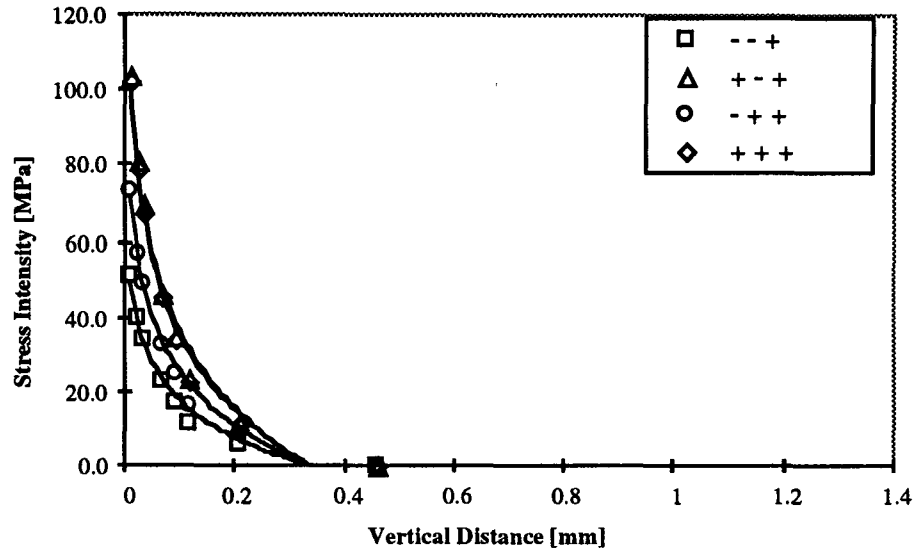


Figure 6.15 Intensity of Stresses in the Vertical Direction (Depth of Cut = 0.508 mm)

6.5 Summary

In this chapter, a fracture mechanics approach is first used to determine a critical stress intensity threshold. Based on a given fracture toughness and depth of cut, the critical stress intensity is defined. Next, the calculated critical stress intensity threshold is used to theoretically predict the resolved cutting force. This predicted force is then compared to the actual resolved force measured during the milling process. Overall, the predicted forces vary from the measured forces. The effect of feed seems to be of particular importance in calculation of the critical stress intensity threshold. Specifically, when a large depth of cut is taken, the measured resolved force is less than the predicted resolved force when a low feed rate is used. However, if a high feed rate is used, the measured resolved force is larger than the predicted resolved force. This indicates that the critical stress intensity threshold is not a static, unchanging parameter.

Next, two dimensional (2-D) finite element analysis is performed to determine the internal stress field induced when DICOR/MGC material is milled. In the proposed model, triangular plane stress elements are used to mesh the geometry. The boundary conditions are set based on the loading constraints. Next, the measured tangential (feed direction) and normal force components are used as input parameters. Depending on the depth of cut simulated, two different geometries are used.

In set of experiments corresponding to the low depth of cut, the largest stress intensities in the horizontal direction are observed for machining condition ++- (feed = 0.4 in/min, spindle speed = 900 rpm, depth of cut = 0.01 in) followed by condition +-+ (feed = 0.4 in/min, spindle speed = 600 rpm, depth of cut = 0.01 in), respectively. The stress intensities for remaining two conditions (-+- and ---) do not significantly vary between each other. The same trends are observed for the stresses in the vertical for each of the milling experiments corresponding to the low depth of cut. However, the stresses in the vertical direction are dissipated more rapidly than the stresses in the horizontal stresses.

For experiments corresponding to the large depth of cut, large stresses in the horizontal direction are observed for two machining tests -- condition +++ (feed = 0.4 in/min, spindle speed = 900 rpm and depth of cut = 0.02 in) and condition +-+ (feed = 0.4 in/min, spindle speed = 600 rpm and depth of cut = 0.02 in). Furthermore, there is a visible difference in the magnitude of the stresses observed for the remaining conditions. In terms of magnitude, larger stresses are observed for condition -++ followed by that for condition --+. As before, the stresses in the vertical dissipate more rapidly than the stresses in the horizontal direction.

Finally, several of the observations cannot be adequately explained. First, when a low depth of cut is taken, only one experiment (condition ++-) results in high stress intensities. On the other hand, when a large depth of cut is taken, two experiments (conditions +++ and +-+) result in high stress intensities. This may result from the design criterion used to define the stress threshold. Specifically, the fracture toughness K_{IC} was treated as a static constant which remains unchanged. This may not be true, the fracture toughness may vary as a non-linear function of temperature, loading conditions and other variables.

Chapter 7

Conclusions and Recommendations

7.1 Conclusions

The work presented in this thesis represents a two year effort in investigating the surface texture formed during the machining of ceramic material. Special attention has been given to the micro-mechanism of material removal, characterization of surface texture, and establishment of the interrelation between the machining parameters and surface texture formation. Main contributions of this thesis work can be summarized as follows:

1. A new methodology to perform surface characterization has been developed in this thesis work. The methodology employs image processing to capture micro-scale details of the surface texture and utilizes computer graphics to visualize the surface topographical conditions. This non-destructive evaluation of surface integrity represents a significant contribution in characterization of surface integrity.
2. A special effort has been made to investigate the capability of using scanning electron microscopy to detect the cracking developed on the machined surface. The high resolution of micrographs attainable by the developed methodology ensures a coverage of rich information on surface height variation at an accuracy level that conventional profilometry methods can never achieve. Results from investigating the penetration depth of electron beams indicate that cracks formed on the surface layer not exceeding 2 μm , as the case study of DICOR/MGC material, can be revealed, showing great promise of further

improving the developed methodology for detecting surface and sub-surface damage.

3. Finite element method has been successfully used to predict the internal stress field induced by machining. The cutting force data obtained during machining have been used as the loading conditions to the study. The mesh generation takes consideration of obtaining the internal stress distribution on micro-scale level. Analysis using fracture mechanics indicates that the competition between the stress intensity factor and crack toughness of the material dominates the material removal during machining. Varying parameter settings, such as depth of cut, feed and cutting speed, have intrinsic effects on the stress threshold for the pullouts of individual grains and micro-cracking on the grain boundaries.
4. A new performance index -- cavity density -- is introduced in this thesis work to quantify the combinational effect of material microstructure and the dynamic loading during machining on the micro-mechanisms of the material removal process. Meaningful explanations have been obtained using this parameter to characterize the surface texture in micro-scale. It is important to note that this parameter is newly introduced in this study and further study is needed to clearly understand the physics behind this parameter.

7.2 Recommendations for Future Work

The research for the thesis work has generated new concerns which need to be addressed. The following directions are recommended for future research.

1. Efforts are needed to expand the current work on the integration of image processing and computer graphics. Accuracy of calibration between the obtained reflection intensity and the height elevation actually represented will be studied. Any errors introduced during the calibration stage have enormous influence on the derivation of meaningful conclusions.
2. Finite element analysis in three-dimensional space is needed. The additional dimension will provide a third direction along which crack propagation may follow. This addition permits a comprehensive study on the intergranular cracking occurred during machining. It should be pointed out for 3D finite element analysis that additional computational efforts are noticeable. Special care has to be taken to ensure that a compromise between memory requirements and computational time is achieved.
3. Interdisciplinary efforts are called for continuing this research. Knowledge and intelligence from the mechanical engineering, material engineering, signal processing should be applied in an integrated way so that the assessment of surface integrity of machined ceramics can be studied using an integrated systems engineering approach. In this effort, collaboration with industry and research institutions is of vital importance.

Appendix A

MATLAB Script Files

A.1. Reconstruction of Surface Topography

```
% topography.m
clear
load ESEM.m

% Invert gray levels
ESEM=abs(ESEM-256);

% change this constant to calibrate height
Microns_to_Pixels= 0.0596479

ESEM=Microns_to_Pixels*ESEM;

% N = Number of elements in file ESEM.m
% N has to be an even positive integer

N=360

% Selection of Section, uncomment irrelevant sections

%Upper left section (1,1) to (N/2,N/2)
%Upper left section (1,1) to (5,5) N = 10 for this example

for i = 1:N/2
    for j = 1:N/2
        ZUL (i,j) = ESEM(i,j);
    end
end

% Upper Right Section (1,1+N/2) to (N/2,N)
% Upper Right Section (1,6) to (5,10)

for i =1: N/2
    for j =1+N/2: N
        ZUR(i,j-N/2)=ESEM(i,j);
    end
end

% Lower Left Section (1+N/2,1) to (N,N/2)
% Lower Left Section (6,1) to (10,5)
```

```

for i = 1+N/2:N
    for j = 1:N/2
        ZLL(i-N/2,j)=ESEM(i,j);
    end
end

% Lower Right Section (1+N/2,1+N/2) to (N,N)
% Lower Left Section (6,6) to (10,10)

for i = 1+N/2:N
    for j = 1+N/2:N
        ZLR(i-N/2,j-N/2)=ESEM(i,j);
    end
end

% End selection of levels

% Definition of centerplane CP

CL1=(sum(sum(ZUR)))/((N/2)^2);
CL2=(sum(sum(ZUL)))/((N/2)^2);
CL3=(sum(sum(ZLL)))/((N/2)^2);
CL4=(sum(sum(ZLR)))/((N/2)^2);

% Scale ZUR, ZUL, ZLL, ZLR

ZUR = ZUR-CL1;
ZUL = ZUL-CL2;
ZLL = ZLL-CL3;
ZLR = ZLR-CL4;

%mesh plot

mesh(ZUL);
grid
xlabel('Pixels (0.746μm/pixel)')
ylabel('Pixels (0.746μm/pixel)')
title('Slot 8, Ra =0.21 μm')
axis([0 180 0 180 -50 50])

```

A.2 Calculation of Roughness Average

```

%Ra.m
clear
load ESEM.m

% Invert gray levels
ESEM=abs(ESEM-256);

```

```

% change this constant to calibrate height

Microns_to_Pixels= 0.4075191

ESEM=Microns_to_Pixels*ESEM;

% N = Number of elements in file ESEM.m
% N has to be an even positive integer

N=360

% Selection of Section, uncomment irrelevant sections

%Upper left section (1,1) to (N/2,N/2)
%Upper left section (1,1) to (5,5) N = 10 for this example

for i = 1:N/2
    for j = 1:N/2
        ZUL (i,j) = ESEM(i,j);
    end
end

% Upper Right Section (1,1+N/2) to (N/2,N)
% Upper Right Section (1,6) to (5,10)

for i =1: N/2
    for j =1+N/2: N
        ZUR(i,j-N/2)=ESEM(i,j);
    end
end

% Lower Left Section (1+N/2,1) to (N,N/2)
% Lower Left Section (6,1) to (10,5)

for i = 1+N/2:N
    for j = 1:N/2
        ZLL(i-N/2,j)=ESEM(i,j);
    end
end

% Lower Right Section (1+N/2,1+N/2) to (N,N)
% Lower Left Section (6,6) to (10,10)

for i = 1+N/2:N
    for j = 1+N/2:N
        ZLR(i-N/2,j-N/2)=ESEM(i,j);
    end
end

% End selection of levels

```

% Definition of centerplane CP

```
CL1=(sum(sum(ZUR)))/((N/2)^2);  
CL2=(sum(sum(ZUL)))/((N/2)^2);  
CL3=(sum(sum(ZLL)))/((N/2)^2);  
CL4=(sum(sum(ZLR)))/((N/2)^2);
```

% Calculation of Ra

```
ZUR = abs(ZUR-CL1);  
ZUL = abs(ZUL-CL2);  
ZLL = abs(ZLL-CL3);  
ZLR =abs(ZLR-CL4);
```

```
RaZUR = (sum(sum(ZUR)))/(N/2)^2  
RaZUL = (sum(sum(ZUL)))/(N/2)^2  
RaZLL = (sum(sum(ZLL)))/(N/2)^2  
RaZLR = (sum(sum(ZLR)))/(N/2)^2
```

A.3 Calculation of Cavity Density

% cslice.m

% crack density for UL

```
NN=180;  
altitude=-1;  
matrix1 = zeros (NN,NN);  
matrix1=matrix1+altitude;  
densityul=0;  
for i = 1:NN,  
    for j = 1:NN,  
        if (ZUL(i,j) - altitude) < .00000000001;  
            matrix1(i,j) = ZUL(i,j);  
            densityul=densityul+1;  
        end  
    end  
end  
densityul=densityul/NN^2
```

% crack density for UR

```
NN=180;  
altitude=-1;  
matrix = zeros (NN,NN);  
matrix=matrix+altitude;  
densityur=0;  
for i = 1:NN,  
    for j = 1:NN,  
        if (ZUR(i,j) - altitude) < .00000000001;  
            matrix(i,j) = ZUR(i,j);  
            densityur=densityur+1;  
        end  
    end  
end
```

```

        end
    end
    densityur=densityur/NN^2

    % crack density for LR
    NN=180;
    altitude=-1;
    matrix = zeros (NN,NN);
    matrix=matrix+altitude;
    densitylr=0;
    for i = 1:NN,
        for j = 1:NN,
            if (ZLR(i,j) - altitude) < .00000000001;
                matrix(i,j) = ZLR(i,j);
                densitylr=densitylr+1;
            end
        end
    end
    densitylr=densitylr/NN^2

    % crack density for LL
    NN=180;
    altitude=-1;
    matrix = zeros (NN,NN);
    matrix=matrix+altitude;
    densityll=0;
    for i = 1:NN,
        for j = 1:NN,
            if (ZLL(i,j) - altitude) < .00000000001;
                matrix(i,j) = ZLL(i,j);
                densityll=densityll+1;
            end
        end
    end
    densityll=densityll/NN^2

    contour(matrix1)
    xlabel('Pixels (0.746μm/pixel)')
    ylabel('Pixels (0.746μm/pixel)')
    title('Slot 8, CD = 0.21%')

```

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