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RULING AND INDENTATION TESTS USED FOR THE INVESTIGATION OF THE BRITTLE/DUCTILE TRANSITION IN BRITTLE MATERIALS

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Abstract. *The ability to machine brittle materials in a controlled manner is required to significantly improve productivity and also to reduce the sub-surface damage thus eliminating the need for lapping and polishing. Much effort has been made by researchers to investigate the feasibility of turning/grinding brittle materials and to identify criteria for ductile mode machining. For brittle materials of interest, cutting depths are generally in the sub-micron range. This requirement, among with others, is well beyond the capability of conventional turning/grinding machines and the research on brittle materials is limited to polishing, indentation and scratching when super-rigid machines are not available. Indentation and ruling tests were used to investigate the effects of elasto/plastic deformation in the micrometre scale. Material properties which affect the ductile/brittle behaviour have been identified and characterised. Brittle materials have been quasi-static indented and ruled with a range of tool conditions and speeds. A ductile/brittle transition was observed when these materials were ruled. The role of material removal mechanisms such as plastic flow, cutting, delamitation and brittle fracture were observed. In this study delamitation and plastic flow were obtained more favourable with tools with large negative rake angles and cutting. Plastic flow and delamitation however can provide lower material removal rates and worse dimensional control when compared with cutting mechanisms.*

Keywords: *PZT, indentation, fracture mechanics, ruling, ductile/brittle transition.*

1. INTRODUCTION

Ductile mode machining has been applied over the past few years to a range of brittle materials such as glass, silicon, alumina and silicon nitride and this work has been widely reported in technical literature (Chao and Gee 1989, Ker 1992).

Even though PZT compositions are used for a wide range of emerging technologies there has been little published research regarding their machining characteristics. Characterising the relationship between fracture and machinability is therefore of particular interest because this would assist in the selection of suitable machining conditions for brittle materials. Contact damage in brittle materials can be modelled in terms of elastic and elasto/plastic contact conditions (Lawn and Marshall 1979, Lawn and Wilshaw 1975). When a hard sharp indenter is pressed into the surface of a brittle material, a region of plastic deformation forms below the point of contact. During quasi-static indentation tensile stresses cause radial/median crack systems to form. When the indenter is un-loaded, a further cracking episode takes place originating from the elastic to elasto/plastic transition region. These cracks propagate and migrate towards the free surface and these are referred to as lateral cracks (Lawn and Marshall 1979, Lawn and Wilshaw 1975). Quasi-static indentation may therefore provide an insight into the fracture processes which occur during machining (Marshall et al. 1983). When the indenter moves laterally there is a greater interaction between the crack systems which result in greater brittle damage. The increased brittle damage has been attributed to changes in the form and magnitude of the residually stressed material (Chiang, Marshall and Evans 1982). Kirchner and Isaacson (1982) used a linear deformation-fracture model to demonstrate that the residual stresses reduce the strength of ceramic surfaces and this should have important implications on the machinability of brittle materials. Quasi-static indentation tests have been extensively used for this purpose to study the fracture behaviour of ceramics (Malkin and Hwang 1996, Lawn and Marshall 1979). Malkin and Hwang (1996) reported investigations into sliding indentation tests using a fracture mechanics approach which was undertaken to understand machining mechanisms. Scratching tests have also provided an important insight into the material removal mechanism for the grinding of brittle materials according to Broese van Groenou, Mann and Veldkamp (1975). Swain (1979) carried out a extensive study of micro-fracturing about scratches in brittle materials and described the phenomenon as being very similar to that which occurs beneath a quasi-static indentation. Swain suggest that if the lateral cracks occur ahead of the scratching point a three dimensional analysis can be appropriate and a material with micro-cracks and pores is required for this purpose. Chao (1991) using a diffraction-grating ruling engine investigated parameters such as stylus tool speed, load and stylus tool geometry for experimental studies into the brittle/ductile effects when ruling glass materials. Similar crack systems to these were observed by Puttick et al. (1989) in the surface of machined brittle materials. Plastic behaviour in PZT ceramics has been reported by Cheng, Lloyd and Kahn (1992). In this case a change in domain re-orientation may be the reason why such a highly brittle material responds in a ductile manner.

2. THE CONTACT MECHANICS

2.1 Static indentation

For indenters having a regular geometry the mean contact pressure is load-independent for low values and this provides a measure of yield stress expressed as hardness, H (Lawn and Evans 1977). Hardness H is calculated from the following equation:

$$\mathrm{H} = \frac{2P\sin(a/2)}{\mathrm{D}^2} \tag{1}$$

where D = length of the indentation diagonal
P = normal load
a = pyramidal indenter angle.

If the indenter is a sharp body, limited plastic deformation will tend to occur about any high stress concentration point in an otherwise elastic indentation field. The residual stress will retain a significant tensile component which is particularly undesirable when testing brittle materials. Residual stresses at the elasto/plastic boundary of a quasi-static indentation into brittle materials such as ceramics have been calculated to be 10 - 15% of H (Hirst and Howse 1969). Quasi-static indentation has also been used to assess the yield stress of materials (Lawn and Wilshaw 1975). Models have been proposed in order to express the brittle material behaviour in quasi-static indentation conditions. Johnson (1970) proposed a correction for the equation developed by Marsh and more recently Studman, Moore and Jones (1977) developed a solution which was much more appropriate for brittle materials in which:

$$\frac{P}{Y} = J + 0.5 + \frac{2}{3}\left[1 + \ln\left(\frac{E \tan \boldsymbol{b}}{3Y}\right)\right] \tag{2}$$

where β = inclination angle
J = geometry constant = -0.2 for spherical and zero for pyramidal indenters
$P = H_V$ from Tabor
Y = yield stress
E = Young's modulus

Parameters relating to the resistance of crack growth are of particular interest in this study. Irwin (1958) introduced a parameter referred to as the stress intensity factor (K_I) which material limit is the fracture toughness (K_{IC}) based on the plastic energy dissipation in the plastic zone around the crack tip. It is also possible to predict the relationship between the applied load (P) and the crack size (C). The region of residual stress caused by plastic deformation causes crack propagation as the indenter is unloaded. By modelling pyramidal indentation as an expanding plastic zone enclosed by an elastic matrix it is possible to determine fracture toughness from the following equation (Lawn and Evans 1977):

$$K_{IC} = k.\left(\frac{E}{H}\right)^{\frac{1}{2}} P.C^{-\frac{3}{2}} \tag{3}$$

where $k = 0.016 \pm 0.004$ see Beltrão (1998)
K_{IC} = fracture toughness
H = hardness
E = modulus of elasticity
P = load
C = crack length

The relationship between load and crack length is $P \alpha C^{3/2}$ and this has wide applicability above the threshold load (P_{min}). Lawn and Evans (1977) proposed a model for predicting the initiation of a median crack under sharp indenters. The critical flaw dimension C^* and the critical indentation load P^* can be calculated using the following equations:

$$C^* = \left(\frac{1.76}{c_1^{\,2}}\right)\left(\frac{K_{IC}}{H}\right)^2 \tag{4}$$

$$P^* = \left(\frac{54.47 c_2}{c_3^{\,2} c_1^{\,4}}\right)\left(\frac{K_{IC}}{H}\right)^3 K_{IC} \tag{5}$$

Where c_1, c_2 and c_3 = dimensionless constants

Modifications of these equations were used to predict the formation of the particular cracks in brittle materials. The formation of median cracks are related to P^* and C^* by the equation proposed by Lawn and Marshall (1979), whilst Hagan (1979) proposed a model for generating a critical micro-crack just beneath the indenter around the plastic boundary.

2.2 Laterally moving indenter or ruling

Scratch testing effectively involves the lateral displacement of a stylus during indentation. Generally the stylus has a sharp pyramidal type geometry though a blunt spherical stylus can also be used. Ruling is a process which is used to generate optical gratings and in ruling the indenter type tool is dead-loaded (Harrison 1973). During ruling there is an intense localised plastic deformation. Various geometries of the diamond tool can be used in a ruling equipment; for example a double-ended 'roof-edge' type tool, a double-cone 'canoe or hatchet shape' tool, a scratch diamond tool and a pyramidal indenter i.e. standard Vickers diamond indenter are all used for various applications. Tribological studies have established a relationship between quasi-static indentation and ruling (Hirst and Howse 1969). Single point diamond ruling has been used to study the machining characteristics of ductile metals and brittle materials (Hirst and Howse 1969, Chao 1991). Ruling can provide an important insight into the effects of localised deformation on the scale of single point machining, polishing and fine grinding. The lateral crack can be modelled for the ruling tests and as with quasi-static indentation a minimum threshold load is required for the onset of lateral cracking.

At scratching depths of 1 to 3.5 μm, ductile flow and scale-like cracks have been reported for some brittle materials (Malkin and Hwang 1996). The load/crack length relationships for several brittle ceramics and vitreous materials scratched by a moving indenter is $P \propto C^n$ where $1/2 < n < 3/2$ according to Beltrão P A (1998).

3. EXPERIMENTAL PROCEDURE

3.1 Material preparation

Samples of SF10 and BK7 type glass materials in the form of 50mm diameter disks, 25mm thick, were polished on one side and lapped on the other. The soda-lime glass specimens were received in the form of 65x45mm rectangles by 2mm thick and polished on both surfaces. 4mm thick disk shaped samples were diamond sawn from cylinders of PC4D and PC5K PZT materials. These were lapped and polished and the final flatness was to within better than half a wavelength of HeNe laser light (~300nm). Samples of three different conditions of each composition were prepared. The conditions were a) poled in-plane (or parallel to the surface), b) poled perpendicular to the surface and, c) unpoled. By observing the {002}/{200} X-ray diffraction (XRD) reflection doublet it could be seen that very little domain re-orientation had taken place in the near surface due to the final polishing.

A micro-hardness tester fitted with a Vickers pyramidal diamond indenter was used to make the indentations at several loads for the glass specimens and for the PZT samples. Indenter dwell was set to 15 seconds. Six indentations were made for each load value on each sample. Hardness and fracture toughness values were then calculated using Eq. 1 and 3.

Complementary to quasi-static indentation the process of single point diamond ruling as used for producing diffraction-gratings was investigated during this study. Seven different loads from 0.2109 to 1.112 N were used in the ruling machine which used a hatchet-shaped tool. All samples were machined with the groove direction parallel to the surface. For the samples poled in-plane, or parallel to the surface, two machining conditions were analysed: a) parallel to the poling direction and, b) orthogonal (90 degrees) to the poled direction. Three maximum ruling speeds of 22, 39.8 and 63.7 mm/sec were used for each load. The movement of the cross feed leadscrew was calibrated to avoid interference between adjacent grooves.

4. RESULTS

4.1. Quasistatic indentation

The size of the plastic indentation and the associated corner crack lengths were measured using the method described by Chiang, Marshall and Evans (1982). From these measurements the hardness and fracture toughness values were calculated using "Eq. 1 and 3". The hardness and fracture toughness values calculated were similar to those reported by other researchers for both glasses and PZT compositions. Different crack-patterns were observed for high and low indenter loads. A typical Vickers indentation for some glass materials takes the form of a square impression pattern with radial cracks emanating from the corners and concentric ring cracks surrounding the contact region. It was suggested that this effect is due to two different deformation mechanisms. A ring crack is shown for indenter loads of 0.1 N. Above this load value only radial and inter-granular cracks are produced.

For the PC5K two forms of crack were formed around the indentation. Firstly a larger set of well defined cracks propagated across the ceramic grains and these were used to calculate the fracture toughness. A second, more localised set of micro-cracks was observed dispersed around the plastic indentation and these propagated along the grain boundaries. This second form of crack formation was observed for indenter loads greater than 0.25 N. Grain boundary fracture could suggest the presence of a weaker grain boundary phase. For PC4D smaller radial cracks were observed for loads above 0.5 N as compared with PC5K for all the samples and poling conditions analysed and there were fewer signs of inter-granular micro-cracking. The quasi-static indentation tests have shown that the surface of PC5K degrades in the region of an indentation which is caused by the propagation of a large system of inter-granular cracks and grain pull-out. The greater amount of grain boundary fracture in PC5K may be due to its greater propensity for ferroelastic domain re-orientation. After poling parallel to the surface, strong anisotropy was observed in quasi-static indentation impressions. The K_{IC} values according to "Eq. 3" for PC5K are considered to be unreliable because there is considerable cracking away from the indentation corner. A similar phenomenon was observed with ruling at higher load values and this will be described later.

Table 1 - Yield Stress of Some Glass Materials

Material	Yield Stress (GPa)
SF10	2.31
BK 7	3.36
Soda-lime glass	3.23

The radial crack lengths measured from the centre of the indentation impression were plotted as functions of load. In each case it was observed that the relation $P \alpha C^{3/2}$ was approximately followed as predicted by Lawn and Evans (1977) "Eq. 4 and 5". It was also observed that the PC4D is consistently tougher than PC5K. The yield stress was calculated using "Eq. 2" and the results considering the stresses either parallel (E_{33}) or (E_{11}) and perpendicular to the polar axis are presented in Tables 1 and 2.

Table 2 - Yield Stress of PC4D and PC5K Ceramics

Poling direction relative to sample surface	Stress direction relative to polar axis	Yield Stress PC4D (GPa)	Yield Stress PC5K (GPa)
Unpoled	N/A	1.46	1.12
Parallel	E_{33}	1.46	1.16
Perpendicular	E_{11}	1.57	1.32
Parallel	E_{11}	1.62	1.39

As for the fracture toughness values the yield stress values show that the material behave in an anisotropic manner. When comparing the yield stress values with the maximum contact stress values it is possible to notice that there are a good agreement between these values.

Materials poled parallel and ruled parallel to the surface are the ones in which less surface degradation was observed. In these case the fracture toughness has its higher values in the direction were the tool is moving and the yield stress presents its higher value in the direction orthogonal to the tool movement which is approximately the same direction were the lateral cracks propagate. Using the measured values of plastic indentation diagonal from the centre of the indentation impression to one corner and the crack length measured from the same centre to the crack tip and plotting these results it was possible to predict the P^* and C^* values for each PZT composition and poling condition using the quasi-static results. The critical median crack or a micro-crack will grow or propagate to a lateral or medium crack respectively when the load reaches a critical value P^* defined by Lawn and Marshall (1979).

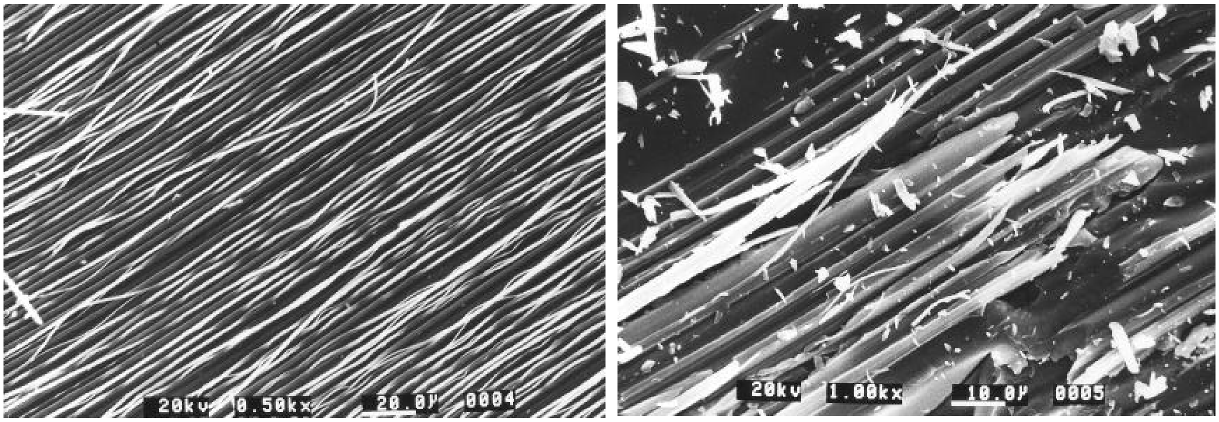
4.2. Ruling

The ruled surface was assessed and the area with brittle damage and grain loss was quantified and divided by the plastically deformed stylus track area in both sides of the stylus tool for the average ruling speed of 39.8 mm/s. From these results it is possible to observe that only the material poled parallel and ruled colinear for both compositions can have the results resolved as the constants are out of the zero intersection region. For the other conditions the range of the loads used for the ruling tests was to high to achieve only ductile behaviour, although in some PC4D samples poled parallel and ruled orthogonal only ductile behaviour was observed. The gradients are consistent higher in the PC5K than PC4D for the same poling condition, indicating that the ceramic surface degrades in a higher rate in PC5K. For samples poled parallel and ruled orthogonal the gradient is smaller than the other conditions probably due to the PZT anisotropy. In a sample poled parallel, the cracks grow less in the poled direction (relevant modulus E_{33}) as observed in the quasi-static indentation tests. For a sample poled parallel and ruled orthogonal the direction in which the lateral cracks will propagate is the poling direction and therefore cracks will tend to propagate less and that was observed during these tests. A ductile/brittle transition was observed as the stylus load was increased above 0.21 N and this load is similar to the results reported for glass ”Fig. 1 a and b”, although the ductile/brittle transition of ferroelectric ceramic is not as well defined.

Some lateral cracks were formed similar to those observed in scratching tests on alumina (see Beltrão 1998). It is therefore suggested that material detachment takes place after the tool has passed and with material loss through crack propagation driven by residual stresses. A similar material removal process was reported by Puttick et al. (1989) for glass materials. The contact pressure necessary for a ductile/brittle transition was calculated for each material condition (Beltrão 1998). A linear relationship between the ruling loads and crack lengths was observed for an average ruling speed of 39.8 mm/s. At a low stylus load of 0.21 N a completely ductile response was observed for PC5K and PC4D when the poling and ruling conditions were colinear.

PC4D ceramic was least affected by ruling at higher load values and also exhibited the least domain re-orientation. One explanation concerns the size effect which explains that for some materials when the depth of cut is under 1 μm, the deformed region is small and for a process such as grinding the resisting shearing stress reaches the theoretical strength which corresponds to the atomic lattice bonding energy for shearing. (Taniguchi 1996).

The respective crack lengths were measured from the centre of the groove to the extremity of the crack for both asymmetric flanks of the tool. These results suggest that there is an optimum tool flank angle to achieve minimal cracking of the ruled material. The ruling experiments demonstrated that poling conditions had a significant effect on the material strength and this was especially evident for high load values. A similar procedure used to predict C^* and P^* in the quasi-static indentation tests was used for the ruling tests. The applied load divided by the area of the stylus contact during scratching will be equal to the contact stress.



a) b)

Figure 1 a) Ductile Behaviour of Soda lime Glass Specimens; b) Ductile/Brittle Transition in Soda lime Glass Specimens

In these tests it was observed that there was an increase in contact stress values between 0.21 - 0.42 N when these had been expected to decrease due to the size effect as the contact stress should be proportional to the hardness according to those researchers. This increase in contact stress values might be attributable to a soft hydrated layer on the PZT samples caused by the polishing process in the same manner as that described for glass by Izutami (1979).

Variation between the behaviour of PC4D and PC5K ceramic materials was also observed. The differences observed between these two PZT compositions may be also explained by other mechanisms such as the formation of micro-cracking which is more pronounced in PC5K as stated previously.

5. CONCLUDING REMARKS

Quasi-static indentation and ruling tests provided important insights into the machining response of PZT ceramics and other brittle materials. Ductile behaviour was observed in some glasses and in two commercial PZT compositions one ‘hard’ and one ‘soft’ and some glasses for a particular range of conditions. The more ductile conditions were observed with loads of 0.21 N and with the PC5K poled parallel and ruled co-linearly and with PC4D poled parallel and ruled colinearly and orthogonal. PC4D behaves in a more stable manner with less degradation of the surface with higher loads and speeds, suggesting that a higher range of ductility could be achieved when machining with a fixed depth of cut. Poling prior to machining enabled a change of the response of both PZT compositions and produced anisotropic behaviour. If only the domain switching mechanism is to be considered as a means for accommodating the strains and consume energy generated by machining the unpoled PC5K material is the one with more favourable conditions. This was demonstrated by the use of unidirectional ruling which showed the possibilities of ductile machining of the PZT ceramics. Additional research needs to be carried out to comprehensively understand the mechanisms associated with PZT plasticity and its influence on machining conditions.

REFERENCES

Beltrão P A, 1998, *Analysis of the Potential of Ductile Mode Machining of Ferroelectric Materials*, PhD Thesis, Cranfield University, Cranfield, UK.

Broese van Groenou A, Maan N and Veldkamp J D B, 1975, *Scratching Experiments on Various Ceramic Materials*, Philips Res. Repts., Vol. 30, pp320-359.

Chao C L, 1991, *Investigations of Machining of Glasses and Other Normally Brittle Materials in Ductile Regime*, PhD Thesis, Cranfield University.

Chao C-L and Gee A E, 1989, *Investigation of Brittle/Ductile Transition in Single Point Machining of Glassy Materials Using a Ruling Engine* Proc. IPES5/ASPE Ann Mtg, Monterey, CA, Sept 1989, pp259-63 (Cranfield: CUPE & Raleigh, NC: ASPE).

Cheng S, Lloyd I K and Kahn M, 1992, *Modification of Surface Texture by Grinding and Polishing Lead Zirconate Titanate Ceramics*, J. Am. Ceram. Soc., Vol. 75, pp2293-6.

Chiang S, Marshall D B and Evans A G, 1982, *The response of Solids to Elastic/Plastic Indentations I. Stresses and residual Stresses*, J. Appl. Phys., Vol. 53, pp298-311.

Hagan J T, 1979, *Micromechanics of Crack Nucleation During Indentations*, J. Mat. Sci., Vol. 14, pp2975-80

Harrison G H, 1973, *The Diffraction Gratings - An Opinionated Appraisal*, J. Appl. Opt., Vol. 12, pp2039-49.

Hirst W and Howse M G J W, 1969, *The Indentation of Materials by Wedges*, Proc. Roy. Soc. London A, Vol. 311, pp429-44.

Irwin G R, 1958, Fracture, in: Fluegge S. (ed.) Handbuch der Physik, Vol. 6, Springer-Verlag, Heidelberg, Germany, pp551-90.

Izutami T, 1979, *Polishing, Lapping and Diamond Grinding*, in: Herman H (ed.), Treatise on Material Science and Technology, Academic Press, New York, USA, Vol. 17, pp115-70.

Johnson K, 1970, *The Correlation of Indentation Experiments*, J. Mech. Phys. Solids, Vol. 18, pp115- 26.

Ker M K, 1992, *The Strength Enhancement of Conventionally Ground Silicon Nitride Ceramics*, PhD Thesis, Cranfield University, Bedford.

Kirchner H P and Isaacson E D, 1982, in: R. C. Bradt (ed.), Fracture Mechanics of Ceramics, Plenum Press, New York, NY, USA.

Lawn B R and Evans A G, 1977, *A Model for Crack Initiation in Elastic/Plastic indentation fields*, J. Mat. Sci., Vol. 12, pp2195-9.

Lawn B R and Marshall D B, 1979, *Hardness Toughness and Brittleness: An Indentation Analysis*, J. Am. Ceram. Soc., Vol. 62, pp347-50.

Lawn B R and Wilshaw R, 1975, *Review of Indentation Fracture: Principles and Applications*, J. Mat. Sci., Vol. 10, pp1049-81.

Malkin S and Hwang T W, 1996, *Grinding Mechanism for Ceramic*, Ann. CIRP, Vol. 45, pp 569-79.

Marshall D B, Evans A G, Khuri Yakub B T, Tien J W and Kino G S, 1983, *The Nature of Machining Damage in Brittle Materials*, Proc. Roy. Soc. London A, Vol. 385, pp461-75.

Puttick K E, Rudman M R, Smith K J, Franks A and Lindsey K, 1989, *Single-point Diamond Machining of Glasses*, Proc. Roy. Soc. London A, Vol. 426, pp19-30.

Studman C J, Moore M A and Jones S E, 1977, *On the Correlation of Indentation Experiments*, J. Phys. D: Appl. Phys., Vol. 10, pp949-56.

Swain M V, 1979, *Microfracture about Scratches in Brittle Solids*, Proc. Roy. Soc. London A, Vol. 366, pp575-97.

Taniguchi N, 1996, *Nano-Machining or Processing Systems with Nanometre Accuracies and Sub Nanometre Scattering Widths or Processing Resolutions* in: Taniguchi N (ed.), Nanotechnology Integrated Processing Systems for Ultra-precision and Ultra-fine Products, Oxford University Press, Oxford, pp15-62.