

# Chemomechanical effects in ductile-regime machining of glass

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*Optical glass was subjected to controlled, low-force scratching, and ultraprecision, fixed-abrasive grinding in various environments, including a homologous series of *n*-alcohols and water. It was found that the liquid environment significantly affected the mechanism of deformation and the extent of subsurface damage. Minimum subsurface damage occurred in a heptanol environment. Observed chemomechanical effects are linked to surface charge and ion adsorption through measurements of zeta-potential between the glass and the various environments. By comparing the present results with previously reported studies of chemomechanical effects, it was found that environments that inhibit fracture in fine microgrinding enhanced fracture at higher machining rates. This finding can be used to improve efficiency and increase productivity in ultraprecision machining of brittle materials.*

**Keywords:** Grinding; optical fabrication; brittle materials; chemomechanical effects

## Introduction

Recently, it has been demonstrated that brittle materials (glasses, semiconductors, and ceramics) can be ground without inducing fracture damage. Abrasive grain cutting depths less than 50 nm are generally required, in a process called ductile-regime grinding.<sup>1-5</sup> Such ultraprecision machining processes, capable of material removal on the scale of nanometers, consist primarily of interactions between surface layers of tool and substrate. Although it has been clear since the early development of ductile-regime grinding models that chemomechanical effects can substantially alter grinding processes, to date no basic research has been directed toward an understanding of such effects in bound-abrasive ductile-regime grinding.<sup>6</sup> Chemomechanical effects in small-grain loose-abrasive grinding have recently been the subject of a study in which it was found that damage levels and material removal rate were both influenced by coolant chemistry.<sup>7-9</sup> In this article, we will describe some empirical results in scratching and microgrinding of glass in

various liquid environments. It will be shown that a possible cause of differences in damage levels when grinding in different environments is ionic adsorption at the surface of the glass, and that electrokinetic measurement of  $\zeta$ -potential for the glass and liquid environment can be correlated with this effect.

In 1928, Rebinder investigated the influence of coolant chemistry on surface properties of nonmetallic brittle materials, in a successful attempt to enhance the efficiency of rock drills.<sup>10</sup> Chemomechanical surface property modifications in brittle nonmetals are often called Rebinder effects in honor of his early work on the subject.

In the 1970s, Westwood and others at Martin Marietta completed a comprehensive study of the effects of various liquids on the hardness and wear resistance of brittle solids.<sup>11-17</sup> Their overall conclusion: the surface hardness of a brittle material is maximized when the measured  $\zeta$ -potential is zero. The  $\zeta$ -potential is a parameter of a given liquid-solid interface, describing the voltage existing at the shear plane in the electrolytic liquid doublelayer near the surface of the solid. When a solid and a liquid come into contact, the surface of the solid may become charged due to adsorption of ions from the solution. At equilibrium, this surface charge,  $\sigma$ , must be balanced by an opposite charge in the solution.  $\zeta$ -potential is one measure of the

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charge in the solution; specifically, it is the potential measured at the shear plane in the boundary layer of the solution when the solution is forced to flow past the solid surface.

The dependence of surface properties on  $\zeta$ -potential has been verified by direct measurement for various crystalline and amorphous solids (e.g., glasses,<sup>11,18</sup> quartz,<sup>14</sup> monocrystalline and polycrystalline alumina,<sup>16,19</sup> and magnesia<sup>15</sup>) in aqueous and nonaqueous solutions (e.g., water, toluene, n-alcohols,<sup>15,18,19</sup> n-alkanes,<sup>17</sup> dilute potassium chloride solutions,<sup>20</sup> dilute potassium iodide solutions,<sup>11</sup> dilute aluminum nitrate solutions,<sup>14,20</sup> dilute sodium hydroxide solutions,<sup>16</sup> and buffered sodium chloride solutions<sup>12</sup>). Again, the primary emphasis of these studies was to improve the efficiency of rock drilling. It was found that surface hardness and  $\zeta$ -potential could also be correlated to machining efficiency. For impact-drilling of very hard solids with multipoint diamond bits, it was found that environments exhibiting a smaller  $\zeta$ -potential magnitude resulted in increased drilling efficiency. Increased machining efficiency is quantitatively measured as a reduction in the specific machining energy, the energy input per unit volume of material removed. In constant-force machining, increased efficiency translates into increased material removal rate. In constant-rate machining, increased efficiency translates into lower machining forces.

For all combinations of brittle solids with liquid electrolyte coolants, Westwood and his coworkers observed a direct correlation between minima in  $\zeta$ -potential magnitude and maxima in surface hardness. This correlation was observed through direct hardness testing and was verified independently through refractive index ellipsometry.<sup>18</sup> It is difficult to accurately determine the thickness of the surface hardened layer, but the ellipsometry results of Malin and Vedam<sup>18</sup> suggest that the "effective" thickness is on the order of 0.1  $\mu\text{m}$ . Effective thickness is not representative of the actual depth of damage, however. Rather, it is a measure of the dimension of an imagined layer of densified glass that, when added to the top of a bulk specimen, would produce the observed behavior in actual ellipsometric measurements. MacMillan et al.<sup>12</sup> observed that chemomechanical effects can alter the hardness of brittle solids to a depth of 30  $\mu\text{m}$ , and Westwood<sup>17</sup> suggested that 1 to 10  $\mu\text{m}$  is a reasonable definition of the surface layer for chemical adsorption. For the purposes of discussion, the surface behavior described in this article will refer to the material within 10  $\mu\text{m}$  of the actual surface.

An exact physical interpretation of the correlation between zero  $\zeta$ -potential and increased surface hardness has yet to be established. Possible mechanisms that have been described include reduced surface energy,<sup>10</sup> restricted dislocation mobility,<sup>21</sup> surface alteration through ion aggregation,<sup>20</sup> and absorbed hydrogen ions.<sup>22</sup> All of these mechanisms are possible, and any or all of them may be acting

in a given liquid-solid system. Common to all of these theories is the idea that a nonzero  $\zeta$ -potential affects the adsorption of liquid ions by the solid. Michalske and Bunker<sup>23</sup> recently described some relevant details concerning the chemistry of dissociative adsorption and its influence on fracture in glass, but they did not address electrokinetic effects such as the  $\zeta$ -potential.

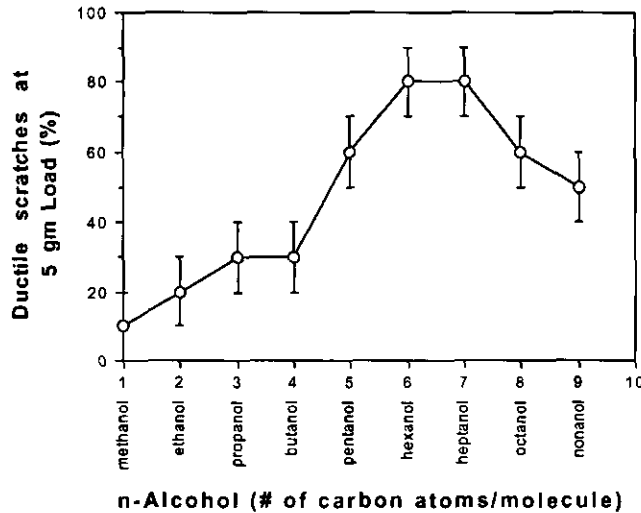
Also unexplained is the correlation between increased surface hardness and machining efficiency. In fact, Westwood was careful to point out that the effect of surface hardness on machining efficiency depends intimately on the mechanics of the machining operation. For impact drilling of glass with multipoint diamond tools, an increase in drilling efficiency was observed for chemical environments for which hardness was maximum. However, for continuous cutting of glass with diamond ball mills, the hardness maximum results in a decrease in drilling efficiency. In polishing of glass with cerium oxide, a decrease in machining efficiency also coincided with the hardness maximum and the environment for which  $\zeta \sim 0$ .<sup>18</sup>

### Environmental effects in microgrinding

It will be shown in this article that in microgrinding of glass, the lowest machining efficiency correlates with minima in the absolute value of the  $\zeta$ -potential. Environments composed of one of a homologous series of alcohols or water have been used in glass scratching and glass grinding to demonstrate that reduced machining efficiency in the presence of particular coolant environments is the direct result of a chemomechanically assisted transition from brittle regime grinding to ductile regime grinding. Two important consequences of the brittle-ductile transition accompanying reduced machining efficiency are improved surface finish and reduced subsurface damage level. The reduction in subsurface damage is a newly discovered and critical chemomechanical effect in glass microgrinding, because removal of subsurface damage is the most time-consuming and expensive process in the manufacture of optical components from glass.<sup>3</sup> Finally, in this article a hypothesis and some supporting experimental data will be presented to explain concurrent changes in surface properties and grinding ductility (plastic flow) in microgrinding. The hypothesis, based on experimental observations and the ductile-regime grinding model previously reported,<sup>1</sup> is that an important chemomechanical effect accompanying the condition of  $\zeta \sim 0$  is an increase in fracture resistance in the chemically modified surface layer of the glass.

### Surface properties, machining efficiency, and subsurface damage

The ductile-regime grinding model<sup>1</sup> predicts a critical depth of cut for transition from ductile to brittle regime in microgrinding as follows:



**Figure 2** Subsurface damage evaluation in single-point scratching of ULE. 5-g load, 100  $\mu\text{m/s}$  scratching speed, 4 mm scratching length. Vertical axis is the percentage of scratches that exhibited no fracture damage under these conditions

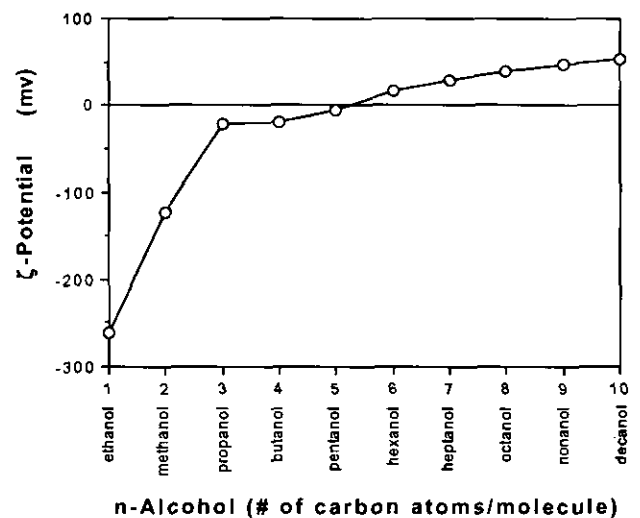
weight alcohols through hexanol. The minimum in subsurface damage level corresponds to hexanol and heptanol. Subsurface damage increases somewhat in octanol, and increases again in nonanol. Damage for ULE samples scratched in a water environment was at the same level as that observed in methanol.

We could find no existing data for the  $\zeta$ -potential of ULE glass in alcohols. To establish this database, we prepared ULE powder suspensions in each alcohol for use in an electrosonic amplitude (ESA) measurement apparatus (MATEC 8000) that can be used to measure  $\zeta$ -potential if certain attributes of the powder and liquid environments are known.<sup>29</sup> Specifically, the conversion from ESA to  $\zeta$  requires the following constants: density and mean particle size of the powder; density, viscosity, and speed of sound in the liquid; and concentration of the colloidal suspension. Sample preparation included pulverizing ULE samples into a powder using a series of ball mills, jet mills, and sieves. The particle size distribution of the powder was measured using a sedigraph, yielding a Gaussian distribution with a mean particle diameter of 3.2  $\mu\text{m}$ . All requisite property constants were obtained from the literature for these well-characterized, reagent-grade alcohols. The concentration of each colloidal suspension was fixed at 0.05% by volume. Ten measurements were made for each suspension. Results of the  $\zeta$ -potential measurements are illustrated in Figure 3 for suspensions of ULE glass in the various alcohol environments. For each data point in the graph, the standard deviation of the 10 measurements is smaller than the size of the plotted symbol.

A correlation exists between the measured oc-

currence of subsurface damage and the  $\zeta$ -potential of the scratching environment. The increase in damage for alcohols that are heavier (octanol-decanol) or lighter (methanol-pentanol) than hexanol and heptanol suggests that there is a definite minima in scratching damage level that corresponds to a hexanol or heptanol environment. The  $\zeta$ -potential measurements show a minimum magnitude in pentanol and hexanol environments. Although this does not correspond precisely with the hexanol-heptanol environment that produced minimum damage in scratching, comparison of the graphs in Figures 2 and 3 does suggest some correlation between the magnitude of  $\zeta$  and the likelihood of damage in scratching. It is likely that there are other factors besides  $\zeta$ -potential and ion adsorption that affect scratching damage in the different alcohols. Some of these factors (i.e., lubrication effects and water content of the different alcohols) have been shown to be less important than ion adsorption in previous studies of glass machining in alcohols.<sup>15</sup> Although the scratches made in a water environment correspond to a large negative value of  $\zeta$ -potential (60 mv), there are other, more compelling reasons to expect increased damage in water, because its embrittling effects have previously been described and explained by Wiederhorn,<sup>30</sup> Tomozawa,<sup>31</sup> and Michaelski,<sup>23</sup> among others.

Further tests involved ultraprecision machining of ULE on a two-axis diamond grinding apparatus. Samples were prepolished, mounted with epoxy to the workpiece holder, and soaked in the selected environment for at least 1 hour before grinding. Different samples, prepared from the same original piece, were used for each environment. The laboratory-scale grinder has a stiffness normal to the grinding contact of 50 MN/m and is controlled in real time to a precision of 10 nm using piezoelectric



**Figure 3** Measurements of  $\zeta$ -potential for ULE glass in various alcohol environments

Table 1 Grinding conditions

Wheel	100 mm diameter, 6.3 mm wide cup wheel
Configuration	Air-bearing spindle and cross slide
Grinding abrasive	4–8 $\mu\text{m}$ natural diamond, 50 conc., resin bond
Infeed	200 nm/pass, 100 passes per sample
Crossfeed	150 $\mu\text{m/s}$
Wheel peripheral speed	7.9 m/s
Workpiece	5 $\times$ 5 $\times$ 5 mm cube
Material removal rate	1.5 $\times$ 10 <sup>-13</sup> m <sup>3/s</sup>
Cutting environments	Water, heptanol, octanol, nonanol

actuation and capacitance gauge sensing. The entire face of each 5  $\times$  5 mm sample surface was ground in each 200 nm deep pass, and 100 passes were completed for each test, yielding a total removal depth of 20  $\mu\text{m}$  per sample per test. Wheel wear was measured and found to be negligible (less than 0.25  $\mu\text{m}$ ) for each grinding test. The wheel was trued and dressed using a diamond lapping technique<sup>6</sup> before each grinding test. The grinding conditions that were used are described in Table 1. These conditions were chosen to provide a grinding chip thickness (i.e., abrasive grain depth of cut) of approximately 180 nm in the brittle regime, but within an order of magnitude of the ductile regime calculated for ULE.

SEM photomicrographs of the ground, and subsequently etched, ULE surfaces showed effects in the different environments that were consistent with the results from scratching experiments (Figure 4). It was found that in heptanol and (to a lesser extent) octanol environments, the grinding was largely dominated by plastic flow. In the nonanol environment, grinding damage levels increased to some extent. Finally, in the water environment, the grinding regime was entirely brittle.

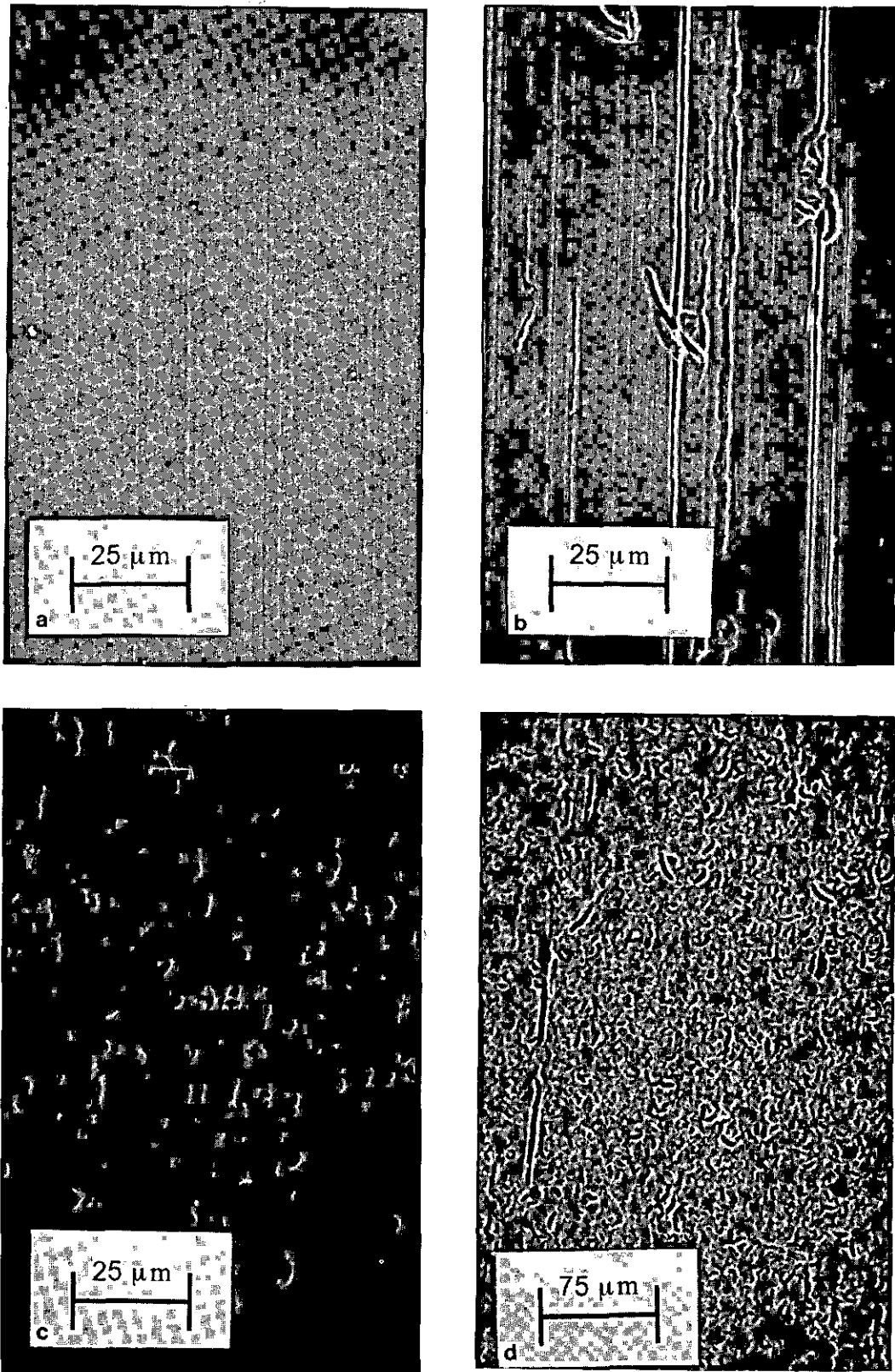
The specific grinding energy (which is inversely proportional to machining efficiency) also showed a significant dependence on environment. For the simple grinding geometry used in these experiments, in which the wheel speed is much greater than the crossfeed speed, the specific grinding energy is given by

$$U = \frac{F_T V_w}{MRR} \quad (2)$$

where  $F_T$  is the tangential grinding force,  $V_w$  is the grinding wheel peripheral speed, and  $MRR$  is the material removal rate. All of these variables were measured in the grinding tests.  $V_w$  and  $MRR$  were

measured directly using displacement and velocity sensors.  $F_T$  was measured indirectly through the motor torque applied to the grinding spindle. This measurement includes, besides tangential cutting force, hydrodynamic resistance between the workpiece and the grinding wheel and air friction of the grinding spindle. The "measured" specific grinding energy, based on  $F_T$ , will be larger than the actual specific grinding energy due to the influence of these additional resisting forces. In all of the environments, the specific grinding energy increased gradually with continued grinding, indicating some dulling of the grinding abrasive grains with use. However, for a given grinding depth,  $U$  was always largest in an environment of heptanol, followed by octanol, nonanol, and finally water. At a grinding depth of 10  $\mu\text{m}$  the measured specific grinding energy for each of these environments is shown in Table 2. These results (reduced machining efficiency and reduced subsurface damage in heptanol and octanol, with  $\zeta \sim 0$ ) are consistent with Westwood's observations in cutting tests on glass with a noncascading ball mill. Later work by Cuthrell on multipoint diamond machining of glass also showed a decrease in machining efficiency with increased surface hardness. A recent study by Golini and Jacobs using loose abrasives showed a brittle-ductile transition for ULE glass in octanol and heptanol environments.<sup>7-9</sup>

It is important to note that all of these experiments were performed near the ductile-brittle transition in abrasive grain depth of cut. In very brittle regime machining of glass by multipoint diamond tools, Westwood found that the correlation was reversed: an increased machining efficiency accompanied chemomechanical environments in which  $\zeta \sim 0$ . That a particular environment can enhance efficiency and fracture at high machining rates (brittle regime grinding) and then inhibit efficiency and fracture at low machining rates was demonstrated by Westwood in a series of experiments on polycrystalline alumina.<sup>14</sup> For diamond grinding of glass, the implications of this phenomenon are significant: the same environment can be used to enhance material removal rate in coarse figuring operations and to inhibit fracture in finishing operations. This phenomenon was first observed in loose abrasive grinding of glass by Phillips, Crimes, and Wilshaw.<sup>32</sup> In this study, machining rate was measured for constant pressure, constant area loose abrasive grinding in both octanol and water. These experiments were performed with various sizes of abrasive, from 4.5 to 105  $\mu\text{m}$  in diameter. It was found that an octanol environment enhanced machining efficiency by up to 100% (compared with a water environment) for large grain sizes ( $\sim 100 \mu\text{m}$ ), but "[for particle diameters] below 10  $\mu\text{m}$  the effect is reversed and water becomes a more efficient environment."<sup>32</sup> Recent research on loose abrasive microgrinding processes has demonstrated both theoretically and experimentally that a decrease in



**Figure 4** SEM photographs of surface damage on ULE microground in various environments. Grinding is from top to bottom for each photo. All surfaces were etched to reveal damage. Ground in (A) heptanol; (B) octanol; (C) nonanol; (D) water

**Table 2** Specific grinding energy for ULE glass microgrinding in various environments

Environment	Specific grinding energy
Heptanol	$9.5 \times 10^{13} \text{ J/m}^3$
Octanol	$5.6 \times 10^{13} \text{ J/m}^3$
Nonanol	$2.8 \times 10^{13} \text{ J/m}^3$
Water	$2.2 \times 10^{13} \text{ J/m}^3$

abrasive size is accompanied by a reduction in fracture damage depth and ultimately leads to a transition from brittle regime grinding to ductile regime grinding.<sup>7-9</sup> The critical abrasive size for transition is proportional to the critical depth of cut [Equation (1)]. For glass the critical abrasive size is about 1  $\mu\text{m}$ .<sup>33,34</sup> The results by Phillips et al. can be interpreted as follows: if material removal processes are operating in the very brittle regime, octanol results in enhanced fracture and greater machining efficiency. If, on the other hand, material removal processes are operating near the brittle-ductile transition, octanol results in inhibited fracture and lower machining efficiency.

### Chemomechanical surface modification

The grinding and scratching results indicate that fracture initiation and/or propagation can be directly influenced by the grinding environment. The empirical evidence shows that increased critical depths of cut (i.e., less fracture damage in scratching and grinding and larger specific grinding energy) correlate with particular environments in glass microgrinding. These results suggest that the ratio of fracture propagation energy to plastic deformation energy in microgrinding and scratching must increase in these environments. The material property characterizing resistance to fracture propagation energy is  $K_{CS}$ , and the material property characterizing resistance to plastic deformation is  $H$ . From Equation (1), it is clear that a chemomechanically induced increase in fracture resistance would have the opposite effect of a chemomechanically induced increase in surface hardness on the critical depth of cut. Moreover, one expects to find decreased fracture resistance in environments for which ion adsorption is large (i.e.,  $|\zeta| \gg 0$ ), because the adsorption of ions in the region of the crack tip is likely to increase the internal stresses near the crack tip, reducing the magnitude of externally applied stresses that would be required to propagate the crack.

In an attempt to measure these surface properties ( $K_{CS}$  and  $H$ ) directly for the glass, microindentation tests were performed on the ULE samples. Polished samples were lightly etched in HF acid before

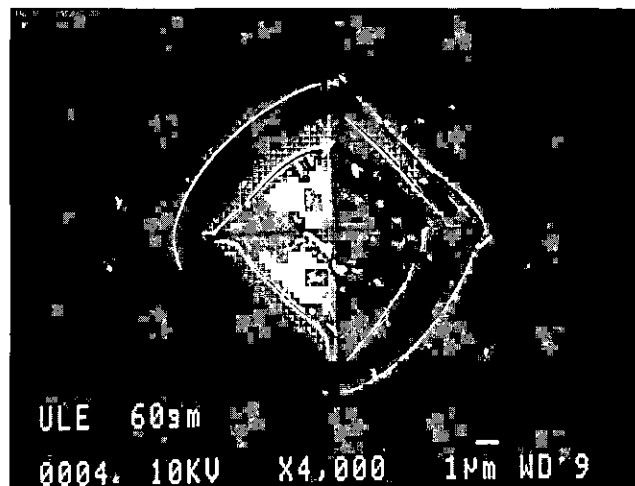
indentation to relieve any residual stresses in the ULE surface. A different sample was used for each environment. Vickers diamond pyramid hardness was measured for ULE samples in four different environments with a 5-g normal load. Ten measurements were made in each environment. Several hours after indentation, the samples were examined under an SEM. The diagonal of the indentation was measured from SEM photograph records, and hardness was computed from the standard formula. Results are shown in *Table 3*.

Also included in this table are the measured  $\zeta$ -potential magnitudes for each of the selected environments. The average hardness increases a small amount for environments with larger  $\zeta$ -potential, a trend opposite from all previously reported correlations of hardness and  $\zeta$ -potential on glasses in alcohols and water.<sup>11-17</sup> It should be noted that some fracture was propagated near the indentation by these hardness tests, and that the measured data for hardness may be affected by energy release through fracture. In any case, the measured increase in hardness is small enough to be within the standard deviation of the measurements, and is probably not large enough to explain the observed differences in grinding and scratching behavior.

For many brittle materials, surface fracture toughness  $K_{CS}$  can also be measured using a diamond pyramid indenter. It has been demonstrated that the surface fracture toughness is given by:  $K_{CS} = \xi(E/H)^{0.5} P/c^{1.5}$ , where  $P$  is the indentation load,  $\xi$  is a geometric constant, and  $c$  is the radius of the "half-penny" cracks that extend from the corners of the indentation.<sup>6,25,26</sup> Measuring toughness from indentation cracks requires the assumption that the cracks "extend well beyond the deformation zone on near-circular fronts,"<sup>24</sup> and that these are the only cracks caused by the indentation. Unfortunately, for indentation of ULE at loads below 100 g, neither of these assumptions is valid. *Figure 5* is an SEM photo of a typical indentation of ULE glass at 60 g normal load. Only the precursors to half-penny cracks are evident at the corners of the indentation. The surface fracture that dominates this indentation is a peripheral crack running along

**Table 3** Microhardness of ULE in various environments (5-g load)

Environment	Hardness (GPa)	Standard error (GPa)	$\zeta$ -potential ( mv )
Hexanol	4.18	0.34	17
Heptanol	4.44	0.37	30
Nonanol	4.62	0.28	47
Water	4.91	0.25	60



**Figure 5** SEM photomicrograph of a typical indentation on ULE glass. Environment, water; load, 60 g

the edges of the indentation and a larger crack encircling the indentation. This behavior may be due to the tendency for ULE (a doped fused silica compound) to densify rather than to flow under the influence of a compressive stress, making artifact-based hardness and toughness measurements anomalous for this material. This phenomenon and its relation to microhardness measurement in fused silica has been described previously by Marshall and Lawn.<sup>25</sup>

The anomalous indentation fracture observed in ULE makes it difficult to assess potential chemomechanical effects on the material's fracture toughness,  $K_{CS}$ . It is conceivable that one could measure the fracture resistance of the glass in a more qualitative way for ULE glass, by noting the occurrence of median and lateral cracks in indentation, without regard to the exact relation of those cracks to the value of  $K_{CS}$ . To that end, we indented ULE in each of the selected environments 10 times at loads of 5, 25, 50, 150, and 300 g. Each indentation was subsequently photographed in an SEM, and an evaluation of indentation cracks was made. In every environment, at every load, there were cracks around the periphery of the indentation. Notably, there were no median cracks in any of the environments for the 5-g loading condition. At 25 g, the environments that did not result in the formation of any median indentation cracks were heptanol and octanol. Also at 25 g, 20% of the nonanol indentations did not exhibit median cracks. All other alcohol environments tested exhibited median crack formation for 25-g indentations. These measurements of indentation median crack formation correlate extremely well with the grinding and scratching results. The environments that produced the most damage-free grinding result on ULE also produced the most limited amount of crack formation in in-

dentation. It is reasonable to conclude that the fracture resistance is larger in heptanol and octanol than in the other environments.

## Conclusions

Chemomechanical effects in alcohols and water have been explored for microgrinding of glass. It has been shown that significant changes in the surface properties of the glass can be made by changing the machining environment. The material effect of such an environmental change is to alter hardness and fracture toughness in the surface layer, resulting in a net increase in the critical depth of cut for a ductile-brittle transition. Although the correlation is not perfect, it appears that surface charge and ion adsorption may play an important role in chemomechanical effects. In scratching, grinding, and indentation tests, the minimum damage observed in ULE glass corresponds to a heptanol environment. Chemomechanical increases in the critical depth of cut can be used to facilitate transition from brittle regime grinding to ductile regime grinding, without changing other machining conditions and without increased machine precision. This has been demonstrated through models and experiments in this article. Additionally, chemomechanical environments that enhance ductility in fine microgrinding can be used to enhance fracture at higher machining rates, thereby improving efficiency and increasing productivity.

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