Research Article

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Analysis of critical process parameters of ductile mode grinding of brittle materials

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Abstract: The process of ductile mode grinding has been analyzed experimentally. Besides the traditional approach, controlling the depth of a cut on feed-controlled ultra-precision machines (UPMs), two alternative approaches have been tested. By applying fluid jet polishing (FJP) using a grinding slurry, a stable ductile mode grinding could be set up. Subsequently, loose abrasive grinding processes on load-controlled traditional spindle machines have been tested to be suited for ductile mode grinding identifying the dependence of crack initiation on the type of slurry fluid being applied. In that way, substantially improved levels of surface roughness could be achieved, although local brittle cracking within the generated clear apertures still remained. Furthermore, critical process parameters were identified determining the process window of feed-controlled ductile grinding applied on state-of-the-art UPM machineries. These have been analyzed experimentally, and it was found that the critical depth of a cut significantly depends on the set of critical process parameters being applied. Finally, the critical ductile grinding process parameters could be identified determining the generated level of surface roughness achieving 0.83 nm Ra on tungsten carbide.

Keywords: ductile grinding; fluid jet polishing (FJP); SPDT; ultra-precision machining (UPM).

1 Introduction

Optical systems are tools for the manipulation of light to serve our daily life's challenges such as illuminating, imaging, communicating, measuring, or medical testing. During the generation of state-of-the-art optical elements made of glass (e.g. lenses, prisms, or beam splitters), highest quality levels are required with, e.g. shape accuracies of less than 30 nm deviation from the required shape, surface roughness levels of less than 0.5 nm rms, and without any defects below the optical surface existing. To that aim, starting from a brittle mode fine-ground surface leaving a rough surface with a cracked sub-surface layer, polishing smoothens the workpiece surface reducing roughness from dozens of nm rms down to sub-nanometer rms values while completely removing sub-surface damage (SSD). During grinding, wear is generated by pure brittle mode cracking caused by sharp abrasive grains exceeding local workpiece material limits of plastical deformation. On the contrary, polishing resembles a chemo-mechanical process, softening the workpiece surface by chemical effects enabling a plastical material movement and removal caused by sharp polishing grains scratching the workpiece surface without any crack generation.

Besides brittle mode grinding and chemo-mechanical polishing, it is possible to remove a material abrasively by plastic flow only, if the indenting grains remain within the plastically deformed top surface layers removing material. This process is called ductile mode grinding and resembles a finishing process usually applied for, e.g. IR optics, calcium fluoride crystals, and tungsten carbide molds for hotpressing of glass. It generates shiny surfaces with optical qualities.

This paper reports on experimental analyses of ductile mode grinding analyzing its process window and critical process parameters. Starting from the traditional solution of generating plastical material removal of brittle materials using feed-controlled machines, additional process parameters are identified that might enable alternative approaches for setting up ductile mode grinding processes enabling the generation of lowest levels of surface roughness.

2 Traditional ductile mode grinding

Owing to technological progresses in the development of high-precision CNC machines in the 1980s and 1990s of

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the last millennium, spindle stiffness and accuracies were achieved enabling tool-to-workpiece positioning accuracies of, e.g. less than 80 nm, underrunning critical values for local crack initiations of brittle materials, such as tungsten carbide, glass, or crystals made of calcium fluoride.

This critical cutting depth d_c below which ductile mode material removal takes place was investigated in detail and well described by Bifano et al. [1] who developed a formula enabling the determination of d_c in dependency of material properties only, such as Knoop hardness K_c and modulus of elasticity E and hardness H:

$$d_c = u \cdot \left(\frac{E}{H}\right)\left(\frac{K_c}{H}\right)^2 \quad (1)$$

Bifano's formula is widely used up until today for setting up ductile mode precision grinding and SPDT processes on ultra-precise CNC machines (UPM) with typical values for d_c of about 60 nm for BK7 glass and 160 nm for tungsten carbide. In that way, ductile mode grinding is enabled using feed-controlled machinery of highest positioning accuracies generating typically surface roughness levels of 4–15 nm rms without any brittle mode cracking leaving a highly tensioned sub-surface layer of less than 1-µm depth.

3 Ductile mode grinding analysis

During abrasive machining of brittle materials with grains (e.g. diamonds), scratching the top workpiece surface layers locally, the process of plastic material flow without any brittle cracking depends a.o. on the following parameters: pressure between grain's tip and workpiece surface, temperature, chemical reactions, environmental conditions such as the coolant between grain and workpiece determining transport mechanisms into potential crack tips being initiated and the molecular workpiece material structures of its top surface layers determining E, H, K_c.

Besides controlling the grain's indentation depth by feed control applying UPMs, in the following, alternative approaches to achieve a ductile mode grinding on tungsten carbide and on glass are tested. To that aim, two finishing processes were tested: fluid jet polishing (FJP) and traditional load-controlled loose abrasive grinding. Finally, the identified critical process parameters were tested and verified within a standard UPM ductile mode machine leading to a better process control of ductile mode grinding process.

3.1 Kinetic ductile grinding

Analyzing the ductile grinding process, its limitation to be only applicable on high-precision CNC machinery was investigated. To that aim, the possibility to guarantee indentation depths of less than 60 nm applying other finishing methods such as FJP was experimentally tested. FJP [2] is a sub-aperture polishing method where a premixed polishing slurry is accelerated through a nozzle within a computer-controlled polishing machine (see Figure 1). In that way, usually the chemo-mechanical process of traditional polishing is being applied without any polishing pad being present. Typically, slurry pressures below 20 bar are used accelerating the 1-µm-diameter CeO_2 grains through a 0.5-mm-diameter nozzle at velocities of about 30 m/s onto the glass to be polished.

To adjust FJP for ductile mode grinding, SiC grinding grains were used in a water-based slurry, and their kinetic energies were adjusted so that the indentation depths remain below d_c.

Consequently, the ability to measure the indentation depth is mandatory; a matter that was solved by setting up the following 'process testing' procedure: a slurry featuring a grain concentration of less than 0.1% by mass (instead of the usually applied ~10% mass) was used to abrade a BK7 surface using 7-µm SiC grains. The test was stopped after some seconds, and single particle

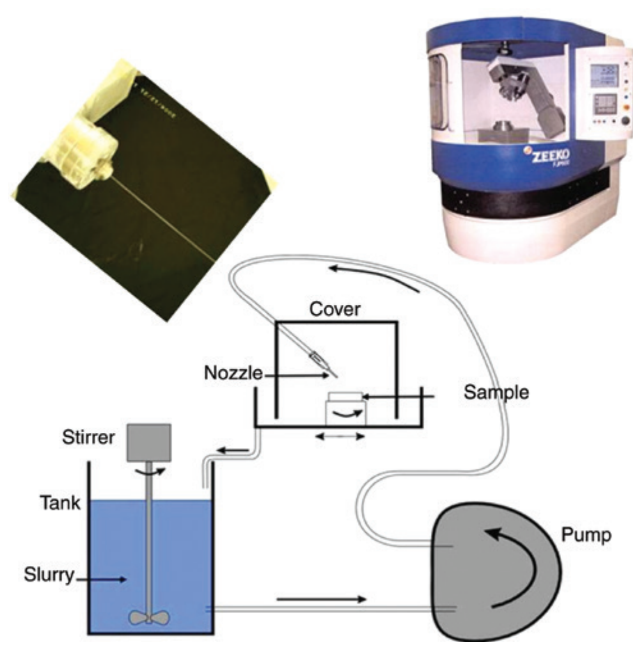

Figure 1: Functioning principle of FJP: a premixed slurry is accelerated by a pump through a nozzle and is being used for zonal polishing within a CCP machine.

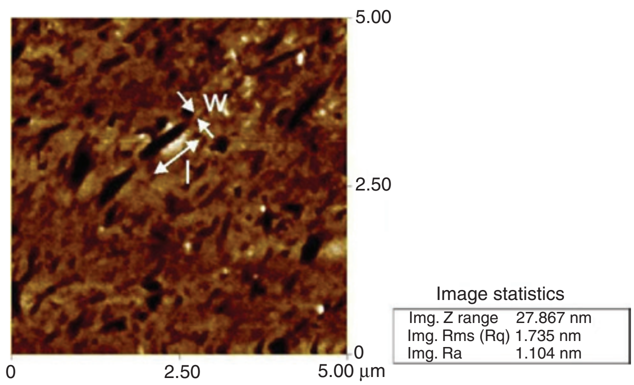

Figure 2: AFM measurement of indentation depth of a single FJP grain in ductile mode: 28 nm.

interactions were detected by atomic force microscopy (AFM). Figure 2 shows a typical result of a FJP grain with an indentation depth of about 30 nm removing BK7 glass material in a ductile mode. This process was subsequently applied to finish a BK7 surface leaving 8-nm rms surface roughness (see Figure 3) without any cracking underneath the surface (measured by etching).

3.2 Load-controlled ductile grinding

Contrary to feed-controlled CNC grinding on UPMs, within loose abrasive load-controlled grinding on traditional spindle machines, the grain's cutting depths can hardly be controlled to remain below Bifano's d_c; this is because of spindle inaccuracies, machine resonance vibrations, and grinding grains agglomerating in the loose abrasive slurry.

Nevertheless, Bifano et al. and Meeder et al. could demonstrate a dependency of crack initiations on the type of coolant used in loose abrasive load-controlled grinding of glass [3, 4].

In the following, scratching experiments are described verifying the dependency of the brittle to ductile mode transition on the coolant fluid used. To that aim, single diamond scratches of 15-mm length (using a diamond tip radius of 40 µm and a tip angle of 45°) were carried out on previously polished flat B270 glass surfaces applying constant load. All scratches were generated using the same scanning speed, and for each tested coolant, several scratches have been generated increasing tool load from scratch to scratch detecting the transition from brittle to ductile mode. Figure 4 shows the setup used for the scratching experiments. Subsequently, each scratch was measured by Nomarski microscopy and judged to be either ductile or brittle mode. For each load applied, 30 scratches have been made, and the probability for brittle mode was determined. Figure 5 shows three typical scratches: a ductile mode scratch and two brittle mode scratches, one with 100% cracking and one being only partially cracked.

Three different coolants were tested: water, glycerin, and octanol (with the latter two containing less than 2% of water), and in addition, dry scratching in air was tested. Vertical loads ranging from 15 g (always causing ductile scratching) to 200 g (always causing brittle scratching) were applied. The results of the scratching experiments are shown in Figures 6 through 9. All graphs show the chance on a brittle scratch versus the load on the diamond tip in grams. Each data point represents between 20 and 30 scratches done under identical circumstances. The corresponding value is the average of all brittle (assigned value: 1) and ductile (0) scratches. Based on the assumption that for an increase in load the probability of a brittle scratch must increase, the cumulative distribution

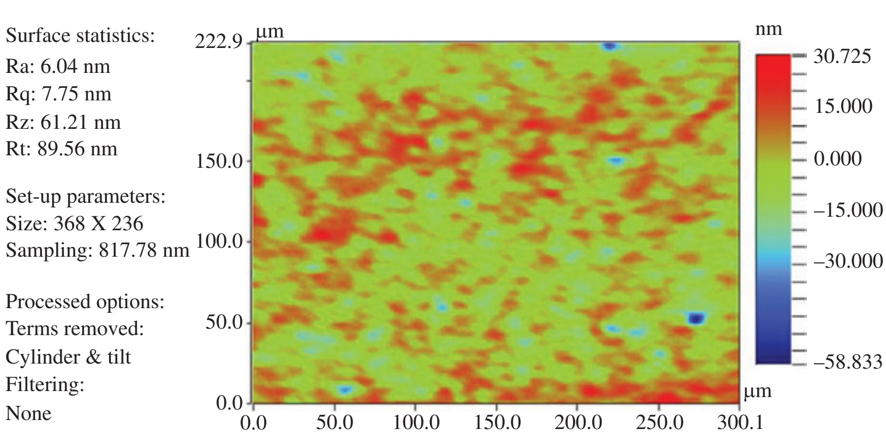

Figure 3: Surface roughness of a fluid jet ductile mode ground BK7 surface measured by white light interferometry featuring 8 nm rms.

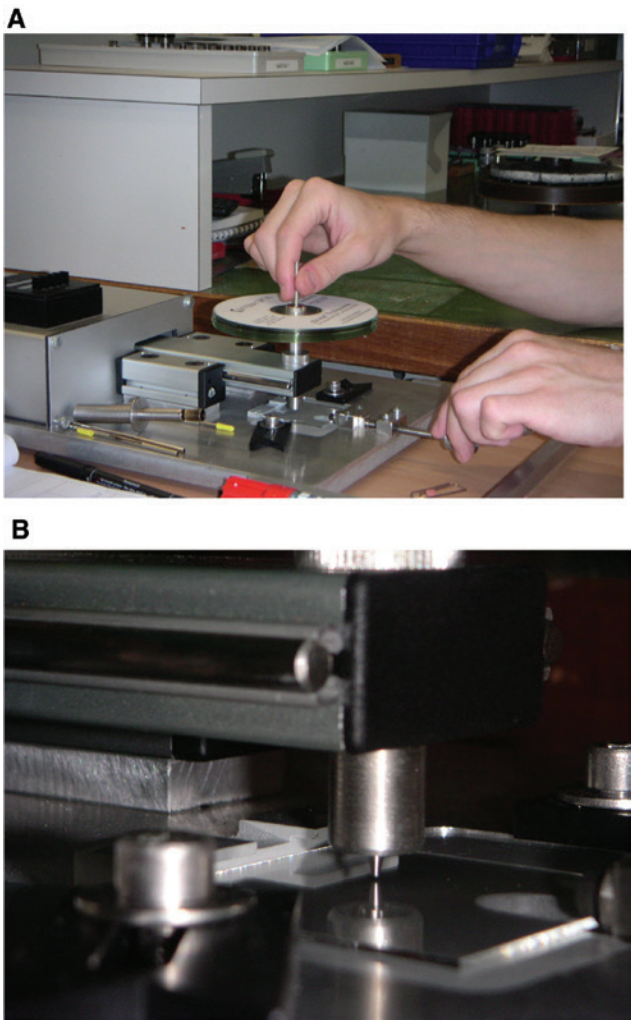

Figure 4: (A) Setup used for scratching using a translational table and a diamond tip loaded by a certain number of CDs: (A) translation stage including the weighted diamond tip above the glass plate being scratched, (B) diamond tip in contact with the glass surface without any coolant being present.

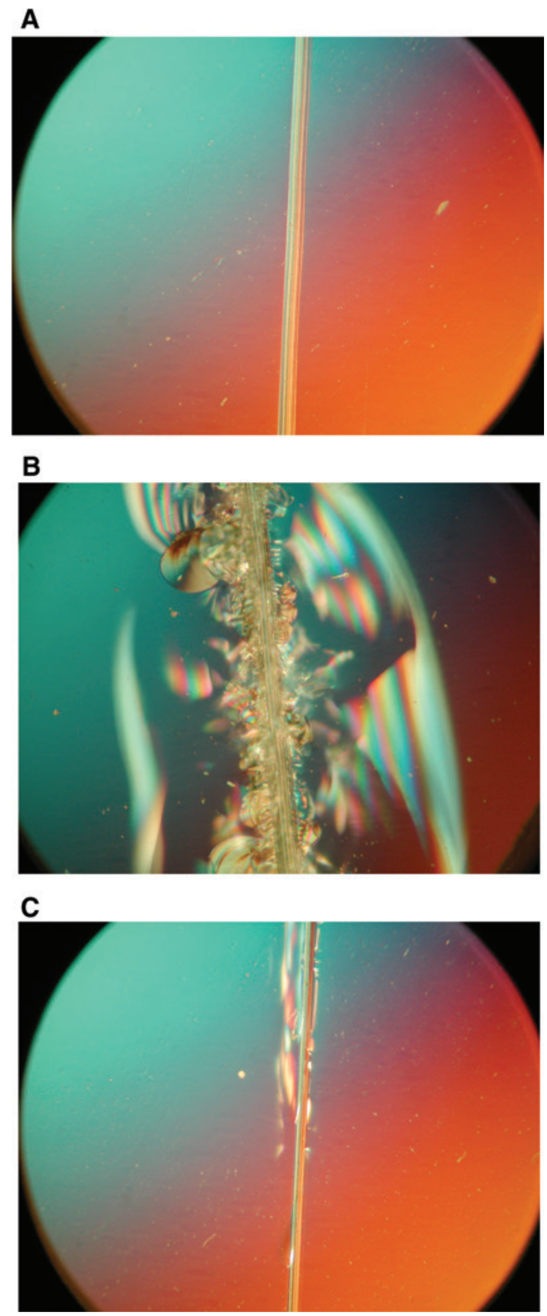

Figure 5: Three scratches: (A) ductile mode scratch without cracking, (B) brittle mode scratch with 100% cracking, and (C) brittle mode scratch only partially cracked.

function (cdf) is fitted to the measurement data in Figures 6 through 9. In terms of a normal distribution with mean μ and standard deviation σ, the cdf is written as:

$$F(x)=\frac{1}{\sigma\sqrt{2\pi}}\int_{-\infty}^{x}\exp-\frac{(u-\mu)^2}{2\sigma^2}du \qquad (2)$$

Higher loads always yield 100% brittle mode scratching. The normal distributions associated with the fitted functions can now be used to characterize the measurement data. The mean value μ for each experiment is the load in grams for which the probability of a brittle scratch is 0.5. One can regard this as the ductile to brittle transition point. The standard deviation σ can be seen as a measure for the width of the ductile-brittle transition region (again, measured in grams of applied load). All experimental results are presented in Table 1 and Figures 6 through 9.

Subsequently, loose abrasive grinding experiments on a load-controlled lapping machine were done to verify the findings of the scratching experiments. The same abrasive solvents (water, octanol, and glycerine) were used to make diamond powder slurries, corresponding to the diamond tip used for scratching. In each experiment, a 1% solution

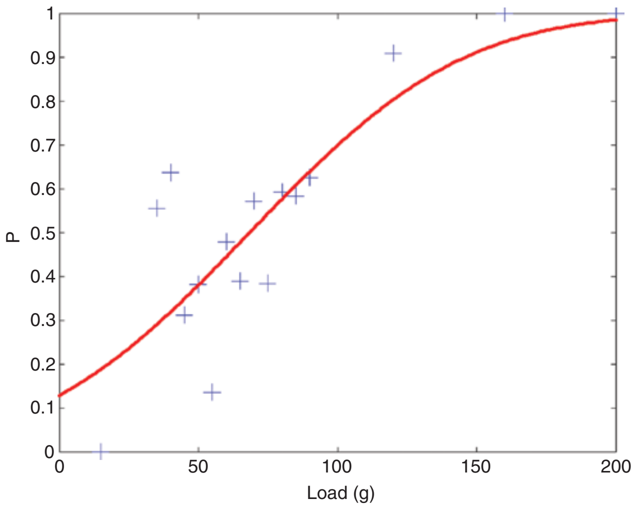

Figure 6: Measurement results for scratches made in water. The graph shows the chance P on a brittle scratch vs. the load on the diamond tip in grams. Fit data for the cumulative distribution function: mean value $\mu = 68.34$, standard deviation $\sigma = 60.49$, sum of squared errors (sse) = 0.39.

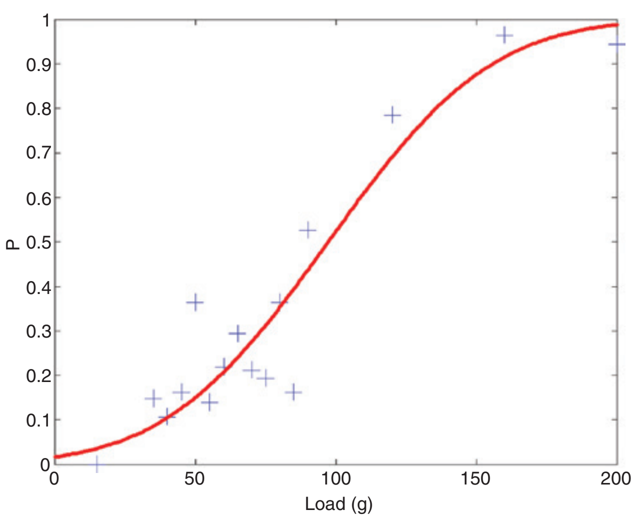

Figure 7: Measurement results for scratches made in glycerin. The graph shows the chance P on a brittle scratch vs. the load on the diamond tip in grams. Fit data for the cumulative distribution function: mean value $\mu = 97.21$, standard deviation $\sigma = 45.61$, sum of squared errors (sse) = 0.15.

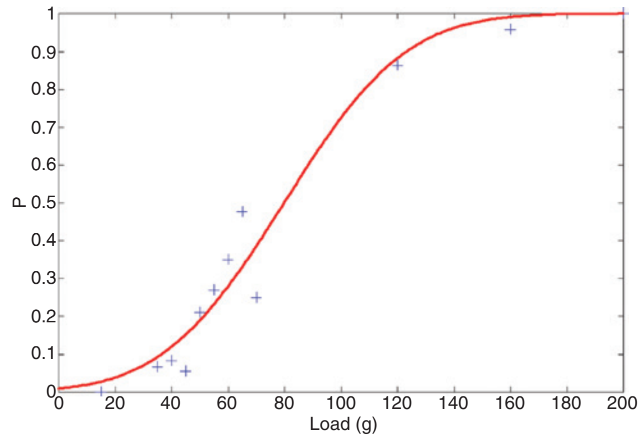

Figure 8: Measurement results for scratches made without the use of a fluid. The graph shows the chance P on a brittle scratch vs. the load on the diamond tip in grams. Fit data for the cumulative distribution function: mean value $\mu = 97.76$, standard deviation $\sigma = 33.85$, sum of squared errors (sse) = 0.060.

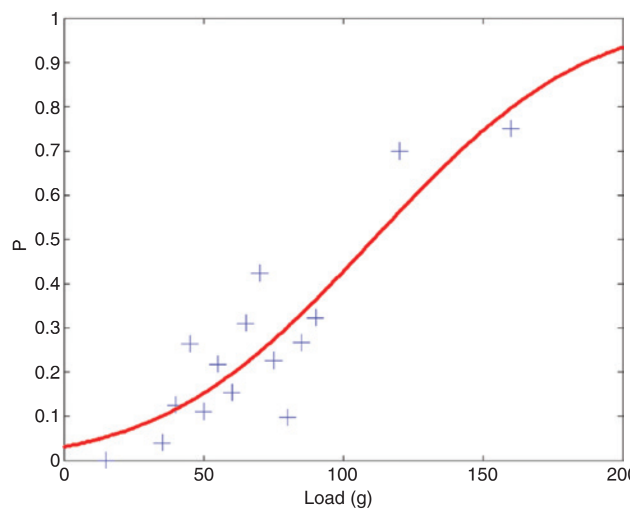

Figure 9: Measurement results for scratches made in octanol. The graph shows the chance P on a brittle scratch vs. the load on the diamond tip in grams. Fit data for the cumulative distribution function: mean value $\mu = 110.70$, standard deviation $\sigma = 59.22$, sum of squared errors (sse) = 0.14.

Table 1: μ and σ for different coolant fluids applied (see Eq. 2).

Coolant	μ (g)	σ (g)
Water	68.3	60.5
Air	97.8	33.9
Glycerin	97.2	45.6
Octanol	110.7	59.2

is used to machine a round piece of Schott K5 optical glass for 2 h using a brass tool and 3-µm grain size. The results are shown in Figures 10 through 12.

Of the tested coolants, octanol is best suited for ductile grinding, whereas water is least suited. The graphs in Figures 6 through 9 show that in the case of octanol, scratches still have a high probability of being ductile for relatively large loads. When translated to loose abrasive grinding, this means that a larger wear rate can be achieved while remaining in the ductile regime. Water-based slurry will fill holes and cracks and speed up crack propagation without having the beneficial chemical

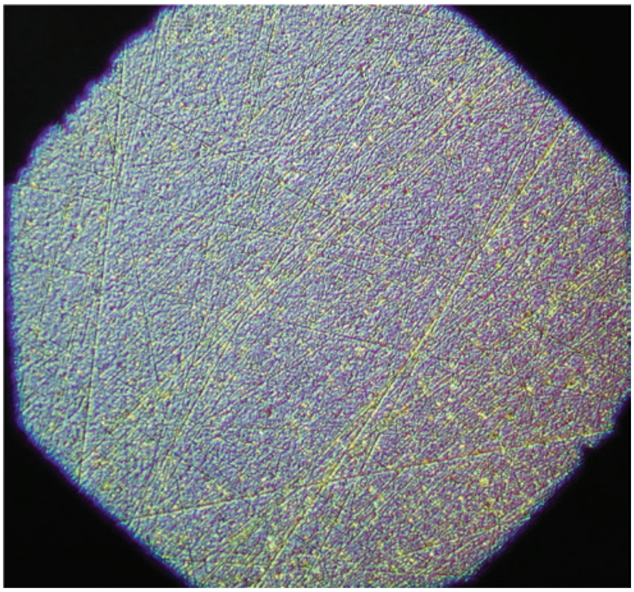

Figure 10: Microscopic images (Nomarski mode) of loose abrasive ground Schott K5 surfaces using water as coolant.

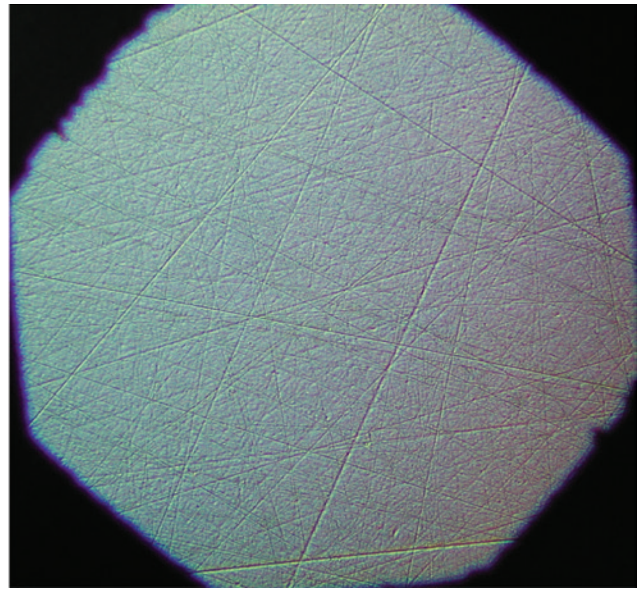

Figure 12: Microscopic images (Nomarski mode) of loose abrasive ground Schott K5 surfaces using octanol as coolant.

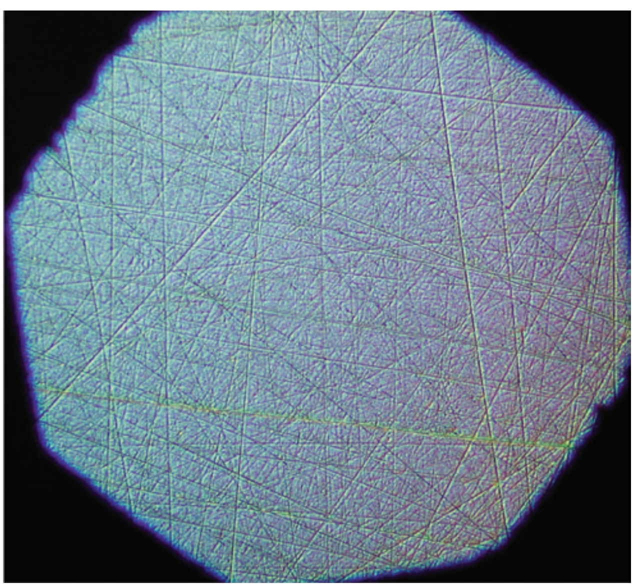

Figure 11: Microscopic images (Nomarski mode) of loose abrasive ground Schott K5 surfaces using glycerin as coolant.

effects of other fluids. The corresponding μ values confirm this: for octanol, the ductile to brittle transition point μ is at 110.7 g load; for water, it is at 68.3 g.

Diamond without a coolant has the best ductile results at low press forces but a quicker fall-off at higher loads. This is, of course, no realistic manufacturing situation, but, nonetheless, interesting. An explanation for this phenomenon is that micro cracks do not propagate as quickly because no fluid can seep in, but at higher loads where cracks are inevitable, there is no chemical influence to 'soften' the glass (thus, making it less 'crack-resistant').

Related to this, an age-old 'secret trick' of master opticians is to let the polishing process 'run dry' when reaching the final surface form. This slightly increases the final surface quality. The earlier statement about the experiments without any fluid explains this trick. According to our data, the optician should let his process 'run dry' under a minimal polishing load for the best results. Another practical implementation of the scratching experiments is found in a well-known method for cleaving glass plates, namely, making a scratch with a diamond pen along the line of cleavage. The glass plate can then be broken cleanly. Our experiments show that applying a fluid – ideally water – before breaking the glass plate should make a cleaner and less force-dependent cleaving.

Finally, the load-controlled loose abrasive lapping results show that the scratching experiments can be translated to a practical loose abrasive grinding situation. The images in Figures 10 through 12 show that octanol is, indeed, the best solvent of the tested fluids, followed by glycerine and water. Controlled environment scratches apparently are a good simulation of a loose abrasive grinding process. Unfortunately, caused by inaccuracies of load-controlled spindle machines and existing mechanical vibrations as well as by grain's tendency to agglomerate in the loose abrasive machining process, load-controlled ductile mode grinding is not stable along the whole clear aperture of the optical surface being

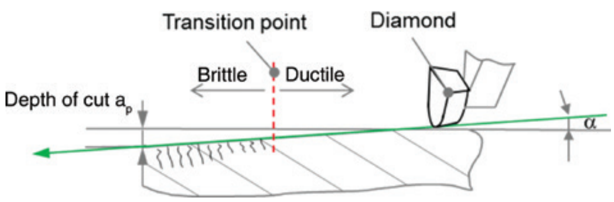

Figure 13: Layout of scratching experiments generating linear grooves of increasing cutting depths using a SPDT machine. In that way, the transition point at d_c from ductile to brittle mode scratching is detected.

ground. Consequently, brittle cracking is present in Figures 10 through 12.

3.3 Feed-controlled ductile grinding process

The results obtained from analyzing kinetic (see paragraph 3.1) and load-controlled (paragraph 3.2) ductile grinding processes were transferred to feed-controlled grinding and tested on UPMs at the Fraunhofer Institute for Production Technology IPT in Aachen, Germany. In Section 3.3.1, the influence of critical ductile grinding process parameters onto the ductile grinding process window is being analyzed, and in Section 3.3.2, the ductile grinding process *per se* is being analyzed experimentally, and process parameters affecting the generated level of surface roughness are identified.

3.3.1 Critical process parameters and process window

In the following, the influence of critical process parameters, such as, e.g. type of coolant, on the ductile grinding process window is experimentally analyzed. To that aim, scratching experiments on a single point diamond turning machine (SPDT) were carried out, scratching linear grooves into the material under test while increasing the cutting depth from zero to values way beyond Bifano's d_c ensuring that the critical cutting depth at which the transition from ductile to brittle mode material removal takes place is detectable (see Figure 13). That way, the influences of various process parameters onto d_c are measurable.

Two materials have been tested: BK7 glass and CTN01L, a binderless tungsten carbide suited to be used as a mold material for precision glass molding (PGM). Using Bifano's formula, d_c yields 62 nm for BK7 and 165 nm for CTN01L.

In the following, experiments were conducted testing the influence of four different process parameters onto d_c

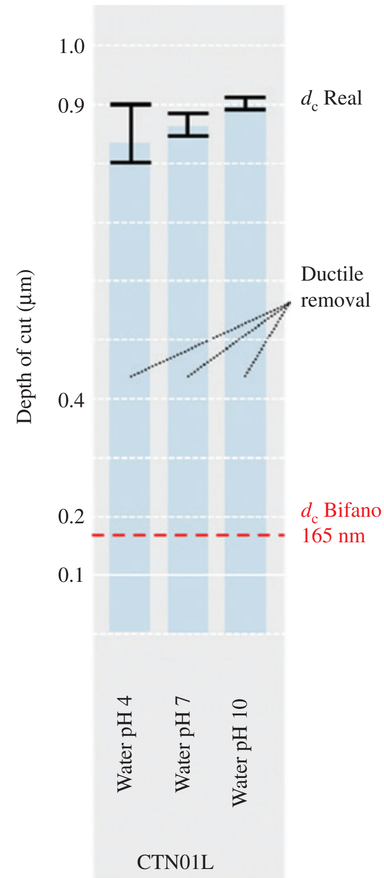

Figure 14: Dependence of d_c for tungsten carbide on the applied pH value of water, which was used as coolant. Note that the experimentally determined d_c value differs by a factor of six from the calculated value using Bifano's formula.

by generating grooves as described above (and sketched in Figure 13) measuring d_c's position within the groove.

It was found that the transition point from ductile to brittle material removal depends significantly on the following critical process parameters:

1. the type of coolant used
2. the pH value of the coolant
3. the tool tip radius of the applied diamond tip and
4. whether ultrasonic assistance (US) is being switched on or off:

$$\begin{aligned}&\textbf{\textit{critical depth of cut}}\\ &\boldsymbol{d_c} = f(\textit{coolant type},\\ &\quad \textit{pH of coolant},\\ &\quad \textit{tool tip radius},\\ &\quad \textit{US (on or off)})\end{aligned} \tag{3}$$

In addition, it was found that the measured d_c values differ significantly from the values obtained from Bifano's formula: e.g. for CTN01L: d_c [water, pH 10, tool tip radius

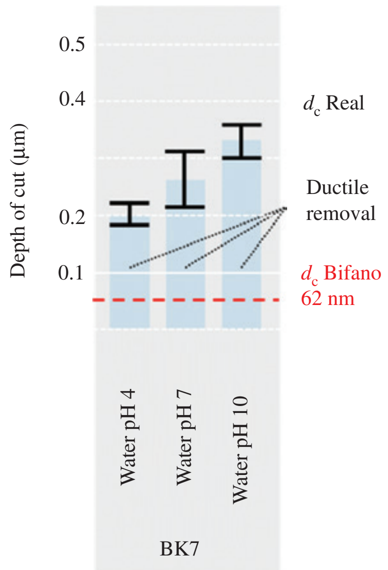

Figure 15: Dependence of d_c for BK7 glass on the applied pH value of water, which was used as a coolant. Note that the experimentally determined d_c value differs by a factor of 1.75 from the calculated value using Bifano's formula.

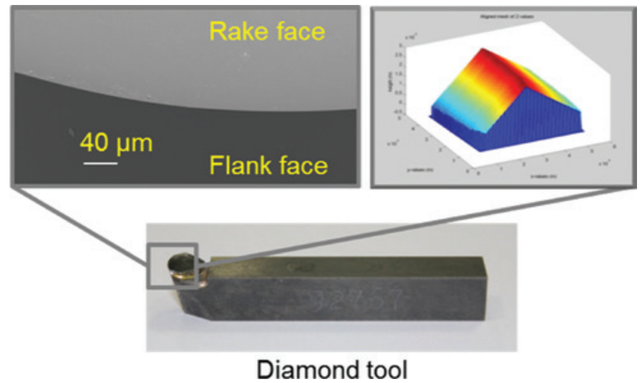

Figure 16: Diamond tool tip radius used for the experiments conducted as sketched in Figure 13.

Table 2: Tool tip radius.

Tool tip radius	d_c
0.36 mm	0.5 µm
0.52 mm	0.9 µm

Coolant = KSSA coolant, US = off, pH = 8.5, CTN01L.

Table 3: US.

US	d_c
On	1.6 µm
Off	1.07 µm

Coolant = water, tool tip radius = 0.53 mm, pH = 7, CTN01L.

Table 4: Coolant.

Coolant	d_c
KSSA	0.5 µm
Water	0.9 µm

pH = 8, tool tip radius = 0.53 mm, US = off, CTN01L.

0.36 mm, US (off)] = 1000 nm, whereas Bifano's formula gives a value of 165-nm depth depending on material parameters only (see Figures 14 and 15).

Figures 14 and 15 sketch the experimental results for the influence of different pH values using water as a coolant onto d_c for CTN01L and BK7 glass, demonstrating that, e.g. for BK7, changing the pH value from 4 to 10 increases the d_c value from 0.2-µm depth up to 0.35-µm depth (a factor of 1.75).

Table 2 shows the dependence of d_c on the applied tool tip radius (see Figure 16) with all other parameters of Eq. (3) being frozen. Note that d_c increases with increasing tool tip radius.

Table 3 shows the dependence of d_c on ultra sound being switched on or off. US switched on shifts d_c to higher values.

Table 4 gives the dependence of d_c on the coolant being used for all other critical process parameters being frozen.

Based on the data received from the experiments carried out, the highest ductile mode material removal rate detected was d_c max = 1600 nm (see Table 3) and is achieved by the following set of critical process parameters: coolant = water, tool tip radius = 0.53 mm, pH = 7, material = CTN01L, US switched on.

In conclusion, the process window of ductile grinding significantly depends on four critical process parameters [see Eq. (3)], and by adjusting these, the transition from brittle to ductile mode grinding can be shifted to substantially higher values of the critical cutting depth d_c.

3.3.2 Surface roughness

Having identified critical process parameters affecting the transition point from brittle to ductile mode grinding, d_c can be shifted to much higher values than predicted by Bifano's formula, e.g. for CTN01L from 165-nm cutting depth up to more than 1500 nm.

This opens the door to analyses of the ductile grinding process itself by, e.g. testing the surface quality within the ductile part of the grooves that were generated by single diamond scratching using a SPDT machine as described in Figure 13.

It was found that the level of surface roughness, Ra, Rt, and Sq, generated by ductile mode material removal depends significantly on the following critical process parameters:

1. the type of coolant used
2. the pH value of the coolant
3. cutting depth d (with $0 < d < d_c$)

$$\begin{aligned} &\textbf{\textit{level of surface roughness}} = \\ &\textbf{\textit{Sq}} = f(\textit{coolant type}, \\ &\quad \textit{pH of coolant}, \\ &\quad \textit{cutting depth } d)) \end{aligned} \tag{4}$$

Table 5 shows the dependence of the generated level of surface roughness (Ra and Rt) within the ductile zone of the groove on the coolant being applied.

Table 6 shows the dependence of the generated level of surface roughness (Ra and Rt) within the ductile zone of the groove on the pH value of the coolant being used. Note that the level of surface roughness decreases with decreasing pH values.

Table 7 shows the dependence of the generated level of surface roughness (Ra and Rt) within the ductile zone of the groove on cutting depth (with $d < d_c$). Note that the level of surface roughness increases with increasing cutting depths.

In conclusion, the generated level of surface roughness of ductile grinding significantly depends on three critical process parameters [see Eq. (4)]. Based on the data received from the experiments carried out, Ra can be adjusted to yield between 0.83 nm and 13.2 nm, resembling a factor of about 16 (see Table 8).

Table 5: Coolant type.

Coolant	Ra	Rt
pH≈7, *d*≈0.7 µm, CTN01L		
KSSA	2.5 nm	20 nm
Water	8.2 nm	70 nm
pH≈7, *d*≈0.1 µm, CTN01L		
KSSA	0.83 nm	4.8 nm
Water	1.5 nm	8 nm

Table 6: pH value of coolant.

pH value	Ra	Rt
4	6.7 nm	50 nm
7	8.2 nm	70 nm
10	13.1 nm	170 nm

Coolant = water, *d* ≈ 0.7 µm, CTN01L.

Table 7: Cutting depth.

d	Ra	Rt
Coolant = water, pH = 7, CTN01L		
129 nm	3 nm	16 nm
285 nm	4.4 nm	32 nm
460 nm	6.1 nm	54 nm
770 nm	8.2 nm	70 nm
Coolant = KSSA, pH≈8, CTN01L		
112 nm	0.83 nm	4.8 nm
205 nm	0.94 nm	7.2 nm
625 nm	2.47 nm	19.75 nm
887 nm	3.75 nm	39 nm

Table 8: Range of surface roughness within ductile mode material removal.

Level of surface roughness	*d*	Ra	Rt
Minimum (coolant = KSSA, pH≈8)	112 nm	0.83 nm	4.8 nm
Maximum (coolant = water, pH≈10)	770 nm	13.1 nm	170 nm

CTN01L.

4 Conclusions

The process of ductile mode grinding has been analyzed experimentally. Besides the traditional approach controlling depth of cut on feed controlled UPMs, two alternative approaches have been tested. Applying FJP using a grinding slurry featuring 7 µm SiC grains, ductile mode grinding could be achieved by controlling the grain's kinetic energy not exceeding a critical value. Subsequently, loose abrasive load-controlled grinding has been tested identifying the dependence of crack initiation on the type of slurry fluid being applied. In that way, substantially improved levels of surface roughness could be achieved by applying, e.g. octanol instead of water-based slurries. Nevertheless, due to agglomeration and unstable and inaccurate traditional spindle machines, each generated surface contained within the clear aperture is also a local zone of brittle fracture.

Finally, critical process parameters determining the process window of feed-controlled ductile grinding on state-of-the-art ultra-precise machineries have been analyzed experimentally. In that way, four critical process parameters could be identified determining the transition between brittle and ductile mode grinding: the critical depth of cut depends substantially on (a) the type of coolant used, (b) the pH value of the coolant, (c) the tool tip radius of the applied diamond, and (d) whether US is being switched on or off. Depending on the applied set of process parameters and for the experimental data collected, maximum ductile mode material removal rates could be achieved with d_c max = 1600 nm. Finally, the level of the generated surface roughness was analyzed identifying three critical parameters affecting it: within the ductile process window of ultra-precise machining, the level of surface roughness generated depends strongly on (a) the type of coolant used, (b) the pH value of the coolant, and (c) the cutting depth d (with $0 < d < d_c$). Depending on the applied set of process parameters and for the experimental data collected, the minimum level of surface roughness on tungsten carbide (CTN01L) generated by ductile mode material removal was Ra = 0.83 nm, a value usually obtained by fresh feed polishing.

Currently, the experimentally gained results are being further analyzed developing an extended Bifano formula including the influence of the critical process parameters onto the critical depth of cut d_c as well as to enable a prediction of surface roughness levels to be generated depending on the chosen set of critical ductile grinding process parameters.

Besides that, experimental studies are being conducted on the trail toward larger values of the critical depth of cut d_c possibly enabling the use of standard CNC grinding machines for a ductile mode grinding instead of being restricted to the use of ultra-precision machineries only.

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