Manipulating mechanics and chemistry in precision optics finishing

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Received 26 October 2006; accepted 14 December 2006
Available online 12 February 2007

Abstract

Deterministic processing is critical to modern precision optics finishing. Put simply, determinism is the ability to predict an outcome before carrying out an activity. With the availability of computer numerically controlled (CNC) equipment and sophisticated software algorithms, it is now possible to grind and polish optics from a variety of materials to surface shape accuracies of ~20 nm peak-to-valley (p–v), with surface roughness values (measured on white light interferometers over 250 μm × 350 μm areas) to sub-nm root-mean-square (rms) levels. In the grinding phase the capability now exists to estimate removal rates, surface roughness, and the depth of subsurface damage (SSD) for a previously unprocessed material, knowing its Young’s modulus, hardness, and fracture toughness. An understanding of how chemistry aids in the abrasive-driven removal of material from the surface during polishing is also critical. Recent polishing process research reveals the importance of chemistry, specifically slurry pH, for preventing particle agglomeration in order to achieve smooth surface finishes with conventional pad or pitch laps. New sub-aperture polishing processes like magnetorheological (MRF) can smooth and shape flat, spherical, aspheric and free-form surfaces within a few process iterations. Difficult to finish optical materials like soft polymer polymethyl methacrylate, microstructured polycrystalline zinc sulfide, and water soluble single-crystal potassium dihydrogen phosphate (KDP) can be finished with MRF. The key is the systematic alteration of MR fluid chemistry and mechanics (i.e. the abrasive) to match the unique physical properties of each workpiece.

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Keywords: Grinding; Polishing; Magnetorheological finishing; Nanodiamonds; CeO2; PMMA; ZnS; KDP

1. Introduction

There are many viable concepts for improving precision optics manufacturing. A broad focus of this paper is to explore what is known about the conventional removal process in grinding and polishing, with a concentration on the role of mechanics and chemistry. Topics addressed include brittle fracture in shaping, subsurface damage (SSD), polishing abrasives, and slurry pH. This is followed by a more detailed examination of deterministic optics processing with magnetorheological finishing (MRF). Examples are given of problem solving, where a manipulation of mechanics and chemistry can resolve finishing issues for materials such as optical polymers, chemical-vapor-deposited (CVD) polycrystalline zinc sulfide (ZnS), and water-soluble single crystals like potassium dihydrogen phosphate (KDP).

2. Using substrate mechanical properties to predict grinding results

Knowledge of $E$—Young’s modulus, $H_k$—Knoop hardness (or $H_v$—Vicker’s hardness), and $K_c$—fracture toughness, can be used to predict important outcomes from grinding, where fracture is the key mechanism of material removal. Table 1 gives functional relationships for removal rates, root-mean-square (rms) surface roughness, and depth of SSD for either loose abrasive grinding or deterministic computer numerically controlled (CNC) microgrinding with bound diamond tools. Lambropoulos [1] has shown that, if a fabricator has experience in grinding two materials whose properties are well known, then with these relationships it is possible to predict grinding results for a material (with known properties) that has never before been processed. This gives the fabricator a competitive edge in bidding for new work. The relationships show that hard materials grind slower, smoother, but...
with less SSD, moderated by fracture toughness. Lambropoulos et al. [2] have also shown that the depth of SSD is always less than two times the peak-to-valley (p–v) surface roughness of a cleanly ground glass or crystal surface. This metric is useful for establishing controlled grinding protocols to minimize the amount of material removal required in polishing out a part.

3. Slurry pH and charge control for smooth surfaces in polishing

Oxide surfaces typically possess a high density of amphoteric hydroxyl groups that can react with either H\(^+\) or OH\(^-\), depending on the pH. Such reactions can occur at the surface of a metal oxide polishing abrasive particle, as well as at a silicate glass surface. The electric charge at either surface in flowing water can be manipulated with pH to be either positive or negative. From polishing experiments on a polyurethane lap with three glasses (fused silica, BK-7, SF-6) and three abrasives (cerium oxide, zirconia, alumina) at three pH levels (4, 7, 10), Cumbo [3] discovered his slurry charge control effect. Simply stated, he found that the smoothest surfaces are obtained for polishing of silicate glasses when charges on the glass surface and the polishing abrasive have the same sign.

Cumbo’s results for fused silica polished with alumina are given in Fig. 1. At pH 4, erosion of the silicate network is not chemically favored. Scraping to remove the hard silica surface gives a very rough result. Agglomeration occurs at pH 7, due to the opposite electric charges of the alumina abrasive (+) and the material coming off of the silica surface (+). Uniform loading of the polishing abrasives is prevented by these agglomerates, giving depressed removal rates and a rough surface. At pH 10, where erosion of the silicate network is optimal, both the glass surface and alumina abrasive are negatively charged. This prevents agglomeration of particles. The uniform load on polishing particles results in the highest removal rates. The resulting surface is very smooth, because the alumina abrasive is effective at scraping off the softened silica surface layer.

4. Problem solving with MRF

MRF was invented and developed by an international group of collaborators at the Center for Optics Manufacturing (COM) in the mid-1990s [4], and commercialized by QED Technologies, Inc. in 1997 [5]. MRF is based on a magnetorheological (MR) fluid consisting of nonmagnetic polishing abrasives (typically cerium oxide or nanodiamonds) and magnetic carbonyl iron particles in water. In the MRF process, this slurry is ejected from a nozzle to form a ribbon on a vertical, rotating wheel. The wheel carries the MR fluid ribbon to its apex, where the fringing field of an electromagnet stiffens the ribbon into a magnetic fluid lap. Polishing abrasives and water are driven to the top of the ribbon and into contact with the surface of a workpiece.

MRF is a sub-aperture lap removal process. Polishing and shaping are accomplished by moving the part, under CNC control, through the contact zone, or “spot” in the MR fluid ribbon [6]. Dwell time at any location determines removal. MRF is unique in that there is almost no normal load between the polishing abrasive and the part [7]. Removal is accomplished through tangential shear forces across the surface. DeGroote et al. [8] recently proposed a model for MRF removal that includes mechanics (grinding figure of merit (£/K\(_c\)H\(_v\)^2) times drag force between abrasive and MR fluid) and chemistry (single bond strength of glass composition times the chemical durability of the glass). The strong positive correlation with removal rate for six optical glasses [three silicate-fused silica (FS), FD-60, BK-7; three phosphate-EFDS-1, FCD-1, LHG-8] is excellent (see Fig. 2).

Table 1

| Material properties and relationships for predicting grinding results [1] |
|-------------------------------|-------------------------------|
| Loose abrasive grinding | Deterministic microgrinding |
| Removal rate | Goes as $E/K_cH_v^2$ | $\sim$ Grinding figure of merit |
| RMS surface roughness | Goes as $(1/H_v)^{1/2}$ | Goes as $(K_c/H_v)^2$ |
| Subsurface damage (SSD) | Goes as $(1/H_v)^{1/2}$ | Goes as $(K_c/H_v)^2$ |

Fig. 1. Adjusting slurry pH for optimal polishing results on fused silica.
5. Polishing soft optical polymers with MRF

Polymer optics are typically manufactured by injection molding (thermoplastics, high volume, economical), compression molding (thermosets, higher precision, and larger sizes) or diamond turning. Once formed with these techniques, polymer components are used “as manufactured”, usually without further cold working to improve surface finish or figure. This is because optical polymers are soft and possess high linear expansion coefficients and poor thermal conductivities [9]. Attempts to improve surface finish and figure using conventional grinding and polishing processes usually result in scratching, the embedding of abrasive particles, the formation of “orange peel” [10], and degradation to surface figure. There are circumstances when it would be desirable to perform a classical polishing operation on a polymer surface. In some instances it is desirable to drive rms surface roughness values below 2–4 nm in preparation for deposition of a coating. In other applications reduced surface form errors are required. It would be useful to eliminate diamond turning marks from polymer optics generated by this ductile machining process, to prevent optical scatter and “flare” when testing prototype imaging system designs.

Because of low normal forces, MRF should be capable of polishing optical polymers to smooth away diamond turning marks, and to correct figure errors without embedding abrasives in the soft surface. DeGroote et al. [11] carried out a series of experiments with different abrasives and carbonyl iron (CI) powders to find MR fluid formulations that could polish four different optical polymers. Standard MR fluids based upon CeO$_2$ or nanodiamond abrasives were tried, along with SnO$_2$, Al$_2$O$_3$, TiO$_2$, and SiO$_2$. Removal rates determined by MR spotting were high compared to those seen on glasses, but roughness levels achieved for three of the four commercial polymers tested (polystyrene, polycarbonate, and a cyclic olefin polymer) were no better than 1.5 nm rms [12]. Many surfaces could not be smoothed to below the 3–5 nm rms level.

The best results are given in Table 2 for experiments conducted on 15 mm thick by 40 mm diameter pucks of polymethyl methacrylate. Using an MR fluid formulation consisting of a low 36 vol% concentration of small ~2 μm (median size) diameter CI powders with ZrO$_2$ abrasives (1.8 μm initial median size of agglomerates), surface roughness levels of 0.5 nm rms were achieved. These values are comparable to those obtained on optical glasses. In a separate experiment, DeGroote and co-workers were able to correct the surface figure error of a diamond turned PMMA puck from 4.45 μm p–v (initial over 95% of the clear aperture) to 0.35 μm p–v (final). This 12× improvement was accomplished with two figure correction runs (run #1 2.8 h, run #2 0.7 h) on a commercial Q22Y MRF machine [13]. Surface roughness levels evaluated at five sites over the part surface were reduced from 35±3 nm p–v/3.8±0.3 nm rms to 18±7 nm p–v/0.47±0.05 nm rms [12], and the diamond turning grooves (35 nm high with a period spacing of 3 μm) were completely removed. This work confirms that MRF can be made gentle enough for the polishing of optical polymers.

### Table 2

| Abrasive (vol%-CI) | Peak removal rate$^a$ (μm/min) | Lowest rms roughness$^b$ (nm) |
|-------------------|--------------------------------|-------------------------------|
| CeO$_2$ (36)      | 4.0                            | 1.6 ± 0.1                     |
| Diamond (45)      | 3.7                            | 4.8 ± 2.1                     |
| SnO$_2$ (36)      | 6.3                            | 1.4 ± 0.4                     |
| ZrO$_2$ (36)      | 3.6                            | 0.50 ± 0.06                   |
| Al$_2$O$_3$ (45)  | 4.0                            | 1.5 ± 0.1                     |
| TiO$_2$ (45)      | 14.6                           | 3.3 ± 0.2                     |
| SiO$_2$ (44)      | 11.6                           | 2.8 ± 0.2                     |

$^a$Std. deviation estimated @ ±2% for an average of 4 MRF spots.
$^b$After uniform ~0.5 μm removal runs.
$^c$Smaller CI particles used in the MR fluid.

[12].

6. Eliminating nodular imprints from CVD polycrystalline ZnS

Millimeter-size nodular structures exist inside infrared-material CVD polycrystalline ZnS grown on graphite mandrels. These “hillocks” may be exposed on the part surface after polishing off microns of material with MRF with either the conventional CeO$_2$-based or the nanodiamond-based magnetorheological (MR) fluid. Kozhinova et al. [14] found that it was possible to eliminate the decoration of nodules and achieve and/or maintain an rms surface roughness of 2 nm on previously ground, diamond turned, or pitch-polished surfaces through mechanics and chemistry. The two-step solution is shown in Fig. 3: a switch from hard CI to soft CI was made that reduced the decoration problem, and an adjustment to the MR fluid pH eliminated it. With the modified MR fluid, it
is possible to polish out microns of material without bringing out the nodule texture on the surface of CVD polycrystalline ZnS.

7. Polishing water-soluble KDP with MRF

KDP is the only nonlinear single-crystal electro-optical material that can be grown in sizes large enough for use as a switch or as a frequency converter in solid state lasers investigating inertial fusion. Large high-peak power lasers like OMEGA and OMEGA EP at the Laboratory for Laser Energetics (LLE) of the University of Rochester, Gekko XII at Osaka University, or the NIF at Lawrence Livermore National Laboratories, require large quantities of KDP plates up to 42 cm × 42 cm in diameter by 1 cm thick. The only commercial process currently available for finishing these plates to specified thickness tolerances, required levels of surface flatness, and rms surface roughness levels of between 1 and 3 nm is single point diamond turning in a mineral oil bath [15]. The material is five times softer than fused silica, and KDP’s solubility in water is high (33 g/100 g). MRF would be valuable if it were capable of finishing this crystal, especially since it could be used to figure the KDP surface to correct for internal inhomogeneities that otherwise could degrade the transmitted wave front quality of the optic [16].

Arrasmith et al. [6] evaluated seven nonaqueous liquids (glycerol, ethylene glycol, polyethylene glycol, liquid paraffin, decahydronapthalene, ethanol, and dicarboxylic acid ester) for use in formulating a new, water-free MR fluid compatible with both the viscosity requirements of an MRF machine and with KDP. Although liquid paraffin was compatible with the KDP surface, a dicarboxylic acid ester (DAE) was chosen for its less hazardous and more eco-friendly nature. Using CI and nanodiamond abrasives in the DAE carrier fluid, a 50 mm × 50 mm diameter, diamond turned KDP block was processed on a Q22X MRF machine [13] to remove 0.5 μm of material. The resulting surface was found to have an rms surface roughness of ~2 nm, and all diamond turning marks were removed. Pulsed IR and UV laser damage testing was performed at LLE on diamond-turned and MRF-processed KDP surfaces [17]. The results are given in Table 3 for UV and IR wavelengths with two different laser-testing protocols. UV damage thresholds of 14 J/cm² for N-on-1 testing were obtained for both surface-finishing processes. A similar result was measured in the IR for the MRF-processed surface. Because of the very high-thermal expansion coefficient for KDP (200–400 × 10⁻⁶/°C), extension of this work to large, single-crystal plates is a significant challenge.

8. Concluding remarks

Through an understanding and application of mechanics and chemistry to the grinding and polishing process, it is possible to develop deterministic techniques for precision finishing of a variety of materials of interest for optical applications. Here, it has been shown that, through modifications to the fluid used in MRF, it is possible to polish soft optical polymers like PMMA, CVD polycrystalline ZnS, and water soluble, single-crystal KDP. Similar advances can be anticipated in the future for other materials. Specifically, a nonaqueous MR fluid should enable the processing of barium metaborate, lithium triborate, nickel sulfate, copper sulfate, and other difficult to process optical crystals.

Acknowledgments

The author is grateful to a number of colleagues, current students, and former students for their technical support in carrying out the research summarized here. These include Jessica DeGroote, Ed Fess, Irina Kozhinova, John Lambropoulos, Alex Mal'tsev, Ansgar Schmid, Semyon Papernov, Henry Romanofsky, John Schoen, Shai Shafir, Steven Arrasmith, Mike Cumbo, Aric Shorey, William Kordonski, Sergei Gorodkin, Marc Tricard, and Don Golini. This paper is dedicated to Harvey Pollicove (1945–2004), founder and director of the Center for Optics Manufacturing (COM) of the University of Rochester.
This work was supported by the US Department of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-FC52-92SF19460, the University of Rochester, and the New York State Energy Research and Development Authority. The support of DOE does not constitute an endorsement by DOE of the views expressed in this article.

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