Critical parameters for grinding large sapphire window panels

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ABSTRACT

Advances in optical manufacturing and testing technologies for sapphire material are required to support the increasing use of large-aperture sapphire panels as windscreens for various electro-optical system applications. Single surface grinding is a crucial process step in both the figuring and finishing of optical components. Improper grinding can make subsequent polishing operations more difficult and time consuming. Poor grinding can also lead to the introduction of surface stress and sub-surface damage which can affect critical opto-mechanical performance characteristics such as strength and durability. Initial efforts have been completed at Exotic Electro-Optics under the funding of the Office of Naval Research and the Air Force Research Laboratory to investigate a number of process enhancements in the grinding of a-plane sapphire panels. The information gained from this study will ultimately provide a better understanding of the overall manufacturing process leading to optimized process time and cost. EEO has completed two sets of twelve-run Plackett-Burman designs of experiment (DOE) to study the effects of fundamental grinding parameters on sapphire panel surfaces. The relative importance of specific process parameters on window characteristics including surface roughness, stress, sub-surface damage are reported.

Keywords: sapphire, window, IR, grinding

1. INTRODUCTION

Large aperture sapphire panels are currently used as the exterior windscreen for the Electro-Optical Targeting Systems (EOTS) on the Sniper Advanced Targeting Pod (ATP) and the Joint Strike Fighter (JSF). As these programs move into routine production, there is an ongoing need to reduce product cost while increasing product performance. Reduction in fabrication process time is the largest practical factor available to lower overall cost. Improved system performance could come from (a) reduced surface roughness to increase panel transmittance and (b) reduced sub-surface damage and surface stress to strengthen panels.

This research program investigated the critical parameters in the grinding operation for large sapphire window panels, with an ultimate goal to achieve: (a) improved final surface roughness; (b) reduced surface stress; (c) decreased sub-surface damage; and (d) increased material removal rate. Improvement in these areas will yield technological progress allowing an increased level of production for high strength, low cost, large sapphire window panels.

2. EXPERIMENT

Two rounds of experiments are presented: one with sub-scale samples for initial process evaluation and the second with larger panels to identify size dependencies. Each experiment was organized as a twelve-run Plackett-Burman DOE. Plackett-Burman designs are fractional factorial experiments used to reduce the amount of runs necessary to study large numbers of parameters. They are generally utilized as screening designs, which serve as the most efficient means to reveal the most vital parameters for the regulation or modification of a process. However, they are not able to elucidate second and higher-order interactions between coupled parameters. For this, Resolution V fractional factorials or a full factorial study is necessary.

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2.1 Experimental Round 1

In Round 1, two sets of specimens were used: (a) twelve sub-scale 4.0" x 4.0" x 0.2” a-plane sapphire panel samples, sliced from larger 9” x 13” panels, and (b) a set of bonded-interface samples (described in Section 3.3). Each of the samples was processed in one of twelve carefully specified experimental runs, allowing one to measure the panel response characteristics due to a specific set of process variables. A Plackett-Burman DOE is a two-level fractional factorial. This means that in each experimental run, each process parameter is set at one of two levels: a low level and a high level. A list of parameters along with a description of the low/high levels used is given in Table 1. An identical list of parameters, excluding Starting Grit Size, was used for the Round 2 experiment.

Table 1. Plackett-Burman DOE Matrix for Round 1.

Process Parameter	Low / High Value
Weight Added	Light / Heavy
Water Type	Deionized / Tap
Spindle Speed	Slow / Fast
Starting Grit Size	Small / Large
Arm Oscillation Speed	Slow / Fast
Slurry pH	Acidic / Alkaline
Slurry Viscosity (Baumé)	Thin / Heavy
Tooling Groove Type	Square / Circular

The sample response to these parameters was measured via preselected output variables. These output variables were chosen to quantify the output of the process with the aforementioned goals of lowering surface roughness, increasing removal rate, reducing stress, and reducing sub-surface damage. The output variables chosen for both Round 1 and Round 2 experiments, along with the measurement techniques employed to quantify them, are given in Table 2.

Table 2. Measurement Techniques.

Data Obtained	Technique Used
Surface roughness	Optical and contact profilometry
Surface stress	Raman spectroscopy
Sub-surface damage	Bonded-interface specimens and Nomarski microscopy
Removal rate	Thickness and time measurements

The panel samples were first back-side ground and polished, using the current EEO manufacturing process parameters. The front (experimental) side was left unprocessed. Photomicrographs of the panel surface were taken at each of three points: a point at the center, a point 20 mm diagonally from the corner, and a point 15 mm from the edge along the centerline of the part. The thickness of each sample was measured using a micrometer at the center of the four edges of the sample in order to gauge removal for subsequent experimental grinding steps. The pre-experimental surface roughness of the front of each panel was then measured by a contact stylus profilometer. Measurements of each sample were taken along four lines that traversed the entire length of the sample. Two lines were parallel to the edge of the specimen that corresponded to the c-plane, with one line at the center, and one 0.3" from the edge. The other two lines were parallel to the edge of the specimen that corresponded to the m-plane, with the same positioning. Once the data were collected into a spreadsheet, the average of the four measurement lines was used to quantify the average surface roughness of the sample. This approach ensured that variations in surface roughness across the surface, as well as any directional dependence of the surface irregularity on the sapphire surface were taken into account.

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The grinding process proceeded in a stepwise manner, using a sequence of progressively finer grits and removing a minimum specified amount of material. After each step the removal time and change in thickness were recorded. It is from these measurements that the removal rate data were derived. The surface roughness was then measured and photomicrographs were taken at the three previously defined points on the surface. Afterwards, the panel was subjected to the next step of experimental grinding.

2.2 Experimental Round 2

For Round 2, twelve 9.0" x 13.0" x 0.2” panel samples were used. As in Round 1, the panels were first measured for thickness using a micrometer, and photomicrographs of the as-grown surface were collected. After preliminary Raman / fluorescence spectroscopic characterizations for surface stress, the specimens were double-side ground. Thickness was measured in order to set a baseline for subsequent removal measurements. The front surface of each panel was measured using an optical profilometer. Optical photomicrographs and surface roughness measurements were taken at the three previously defined points on the sapphire panel surface. The three surface roughness measurements were averaged together to gauge the average surface roughness across the panel.

As previously mentioned, the Round 2 experiments did not incorporate the starting grit parameter into the design. This is because the double side grinder makes use of a smaller grinding grit than the initial grits used for single side grinding; therefore rough grinding is not practical for this process. The removal of this parameter necessitated a redesign of the Plackett-Burman matrix used to define the experimental runs. Thus the Round 2 grinding experiment was not a replicate experiment, but a distinct one.

As with Round 1, each grinding step removed a pre-defined minimum amount of material and the exact amount of material removed and the time this operation took was recorded. After the first step, the experimental panel was ground using a progressively finer sequence of grits. Due to the blocking of the large panels, it was not possible to measure surface roughness or acquire photomicrographs of the panel surface in between grit steps. Thus, measurements of surface roughness and photomicrographs were taken before and after the complete experimental grinding sequence. Lastly, the panels were sent out for stress characterization via Raman / fluorescence spectroscopy.

3. RESULTS

3.1 Surface Roughness

A plot of the average root-mean-square (RMS) surface roughness of each Round 1 sample through each step of the experiment is given in Figure 1. This plot shows that although there is large variation in the surface roughness of each panel during rough grinding phases, once fine grinding starts at experimental step 3, these values start to converge, and by step 4, they are essentially equal.

At grinding step 1 in Figure 1, a clear grouping containing two sets of six samples emerges. The high-RMS group consists of all the samples ground using slurry with the larger diameter grit and the low-RMS group consists of all the samples ground using the smaller grit. This is as expected. It is clear that the larger grit grinding induces higher RMS surface roughness due to its larger particle size. What is surprising is how quickly the elevated RMS is decreased by the subsequent grinding step. Once the panels undergo grinding in step 2, the grouping has practically disappeared. This suggests that one may be able to employ a relatively high initial removal rate by using larger grit without affecting the final surface roughness of the panels.

Once all of the experimental runs were complete, it was possible to perform a parametric analysis of the data using Minitab, a commercial software tool used for such analyses. The Main Effects plots for Final RMS are given in Figure 2. Note that the Final RMS surface roughness is the value achieved after all grinding steps have been completed. According to Figure 2, the input variables Water Type, Slurry Baumé and Tooling Groove Type only marginally affect the Final RMS surface roughness of a panel undergoing the grinding process. Slurry pH and spindle speed were slightly more important. Improved Final RMS surface roughness was obtained through higher Added Weight, larger Starting Grit size and higher Arm Oscillation Speed.

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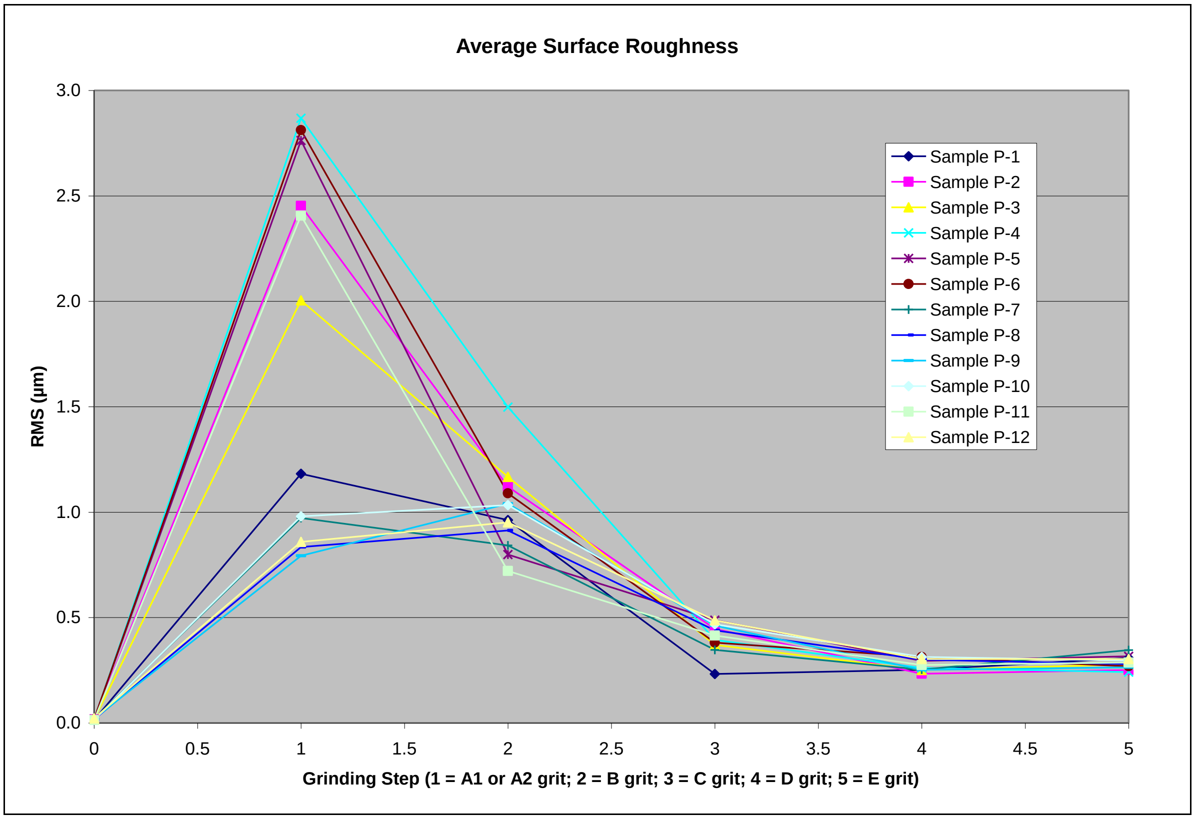

Fig. 1. Average RMS surface roughness of each Round 1 sample for each grit size. Notice that although there is great disparity in the RMS surface roughness of each sample early in the process, later grinding stages normalize these disparities. Grit size: A1 > A2 > B > C > D > E.

The fact that increased Arm Oscillation Speed results in decreased Final RMS surface roughness is extremely beneficial to the production process, because increased grinding speeds are associated with increased removal rates. Thus, an initial aggressive process incorporating high removal rates will result in higher quality surface finishes. Also, it is expected that decreased surface roughness in grinding will ultimately lead to lower levels of sub-surface damage and higher panel strength after polishing.

The behavior of the Added Weight and Starting Grit Size parameters both suggest that additional process improvements could be made by increasing both Added Weight and Starting Grit Size in the process. The results of this experiment show that increasing these grinding parameters would decrease Final RMS surface roughness. It can be assumed via Preston's wear equation that increasing each of these parameters would have the net effect of decreasing cycle time through increased grinding removal rates.

The remaining input parameters do not follow a consistent trend. Pareto charts were produced in order to determine the statistical relevance of each parameter to the process. However, these analyses indicated that none of the input parameters produced a significant first-order response in the surface roughness output variables. This tells one that interaction effects may be causing aliasing in the results. A subsequent experiment considering second-order effects must be done to see if some of these second-order effects can be extracted.

Although an analysis of final surface roughness did not show any significant first-order effects in Round 1, the statistical analysis conducted in Round 2, which instead considered the change in surface roughness, showed two main first-order parameters: Arm Oscillation Speed and Water Type (see Figure 3). This confirms the findings of Round 1, which showed that, on average, heightened Arm Oscillation Speed produced lower surface roughness panels, although the

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statistical analysis did not yield this as a significant parameter. The discrepancy between these results further suggests that first-order analyses are being confounded by second-order interactions.

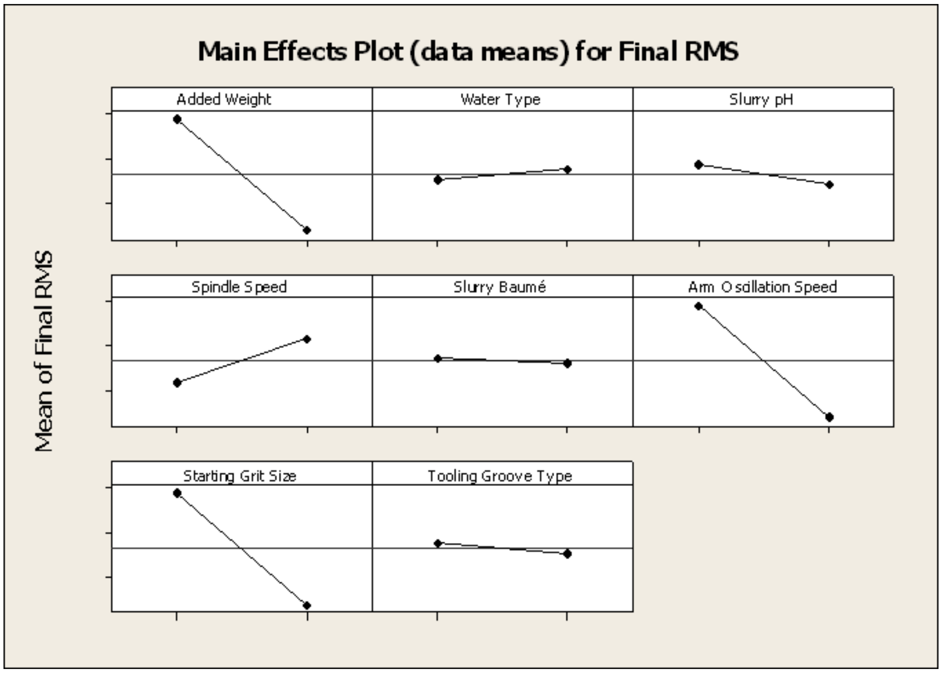

Fig. 2. Main Effects plots for final average RMS surface roughness of all Round 1 experimental samples that underwent processing for a given parameter value. Round 2 data were consistent with these results.

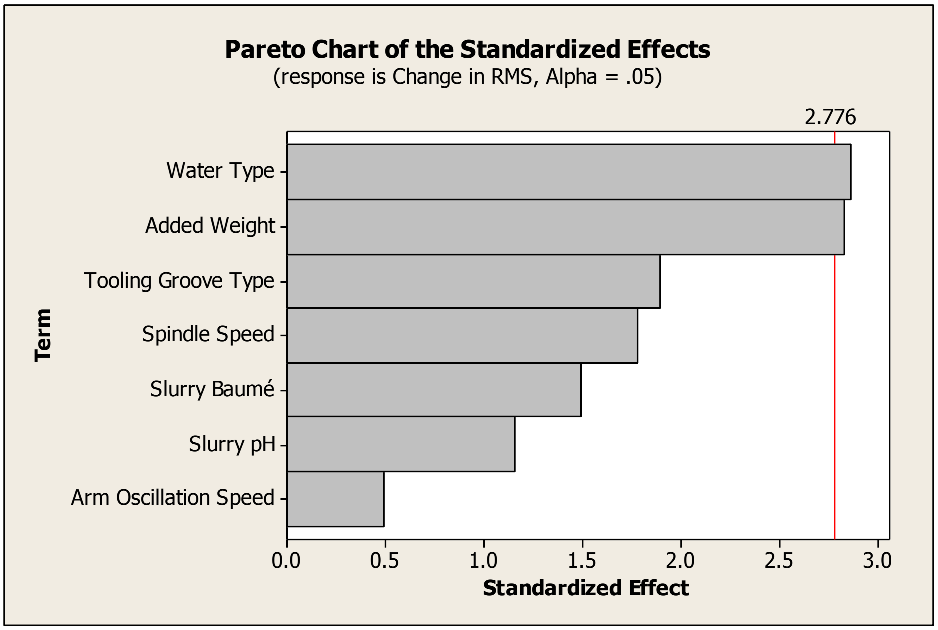

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Fig. 3. Pareto Chart for the change in RMS surface roughness for Round 2 experiments. Surprisingly, Arm Oscillation Speed is not found to be a significant effect. This is probably due to aliasing, as it was shown in Round 1 that this parameter is indeed significant.

3.2 Surface Stress

Raman spectroscopy is a well known optical method of characterizing materials. A slightly alternative method of measuring stress in sapphire is by exploiting the piezospectroscopic effect as applied to the fluorescence from trace Cr^{3+} impurities. Like Raman, the basis of this method is that the characteristic fluorescence lines, commonly known as the R1 and R2 lines, shift under applied stress. This technique is described fully in the associated paper in these proceedings.[1]

The observed frequency shifts due to the grinding processes are shown in Figure 4. The line shift is dependent only on the process-induced stress applied along the crystallographic a-axis and not on previous residual stress. In general, the results show that different processing parameters affect the R-line frequency. In these experimental results, the R-line shifts are analyzed in terms of the difference between the R1 and R2 line shifts, that is, the change in R1-R2 separation. The resolution of the individual measurements was ±0.04 cm^{-1}. Comparing that value to the observed peak shifts, it appears that a measurable shift associated with fabrication process induced stress is observed. Depending on the specific process parameters and the position on the sample where the measurements were made, the shift/stress can either be compressive or tensile. This result is dominated by tensile shift, characteristic of micro-fracturing expected in grinding operations. The random nature of localized grinding action is the likely cause of the apparent randomness in the peak shifts. This result is consistent with observations reported by Molis and Clark who measured the complex stresses around indentations in chromium-doped sapphire.[2] While the results suggest some specific operating parameters that can lead to reduced residual stress levels in sapphire windows, the Minitab parametric analysis of the data showed no clear correlation with the controlled grinding parameters probably due to the randomness of the result.

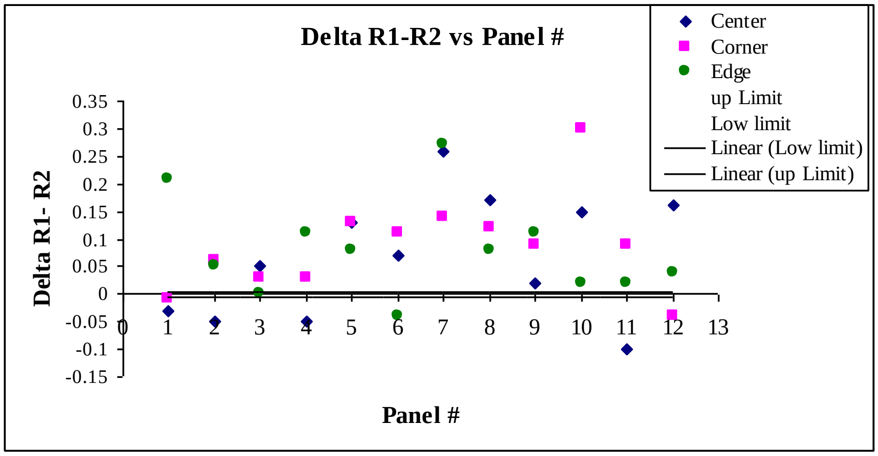

Fig. 4. Δ R1-R2 line shifts of ground sapphire versus Round 2 specimen (panel) number.

3.3 Sub-surface damage

The bonded interface technique has been previously employed to study sub-surface flaws in polished sapphire.[3] In these experiments, two 1.5” x 0.22” x 0.24” bars of sapphire were glued together at polished m-plane or c-plane faces to make a compound sample. Each sample was bonded as either a dual m-plane sample or a dual c-plane sample. Both m and c-plane faces were used to increase the experimental data set, giving one a view into the distinct processes which control fracturing relative to each plane.

Pre-experimental photomicrographs of these samples were taken along the interface edge of each sample piece at pre-determined locations. After pre-inspecting, the bars were bonded into the compound samples. The glue and fixturing were optimized in order to minimize the gap distance between the bars.

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After the experimental grinding on the a-plane face, the bars were separated and the chip and fracture defects with the greatest depths were tabulated for each sample. This depth is referred to as the maximum defect depth. The maximum defect on many samples was often an order of magnitude greater than the next largest defect. Therefore the second largest chip and fracture defects on each sample were also tabulated. This depth was referred to as the characteristic defect depth as it was much more representative of the magnitude of the majority of defects on the sample. The characteristic depth should not be thought of as an average but rather as the representative depth of the vast majority of defects.

The depths of damage sites were tabulated by first finding the maximum chip depth and the maximum fracture depth for each of the four samples (two m-pane and two c-plane) for a given set of processing parameters. The maximum defect depths from the samples were averaged to determine the maximum defect depth reported for each set of processing parameters. Although the data are reported here for the deepest defect regardless of type, data for chips and fractures were collected separately as it is unknown whether the same mechanism produced both the chips and the fractures. Ultimately, both defects should be minimized and certain process parameters might favor the creation of one defect over the other. That analysis is ongoing. For now, both types of defects were considered in aggregate.

The depths of defects measured in each Round 1 grinding sample are depicted in Figure 5. The characteristic defect depth of the samples indicates that P-8, P-7, and P-4 are the least damaged grinding samples. This partially agrees with the results of the maximum defect depth findings data which indicated that P-7 and P-8 are the least damaged samples. Representative photomicrographs of a characteristic defect on sample P-8 and the maximum defect on sample P-10 are shown in Figure 6. In brittle materials, the reliability of the material is of critical importance in manufacturing and in the ultimate application. The presence of one critical flaw can lead to catastrophic failure in large single crystal materials. Therefore, the maximum defect depth, while typically uncharacteristic of the overall grind, is necessary to consider in evaluating processing parameters. P-7 and P-8 are the favored processing parameters as they have the lowest characteristic and maximum defect depths. Although the data supports the conclusion that the maximum defects are loosely related to the characteristic depth, it is the characteristic depth data which is most seriously considered in this analysis, as the repeatability of the maximum depth of damage measured is still not firmly established.

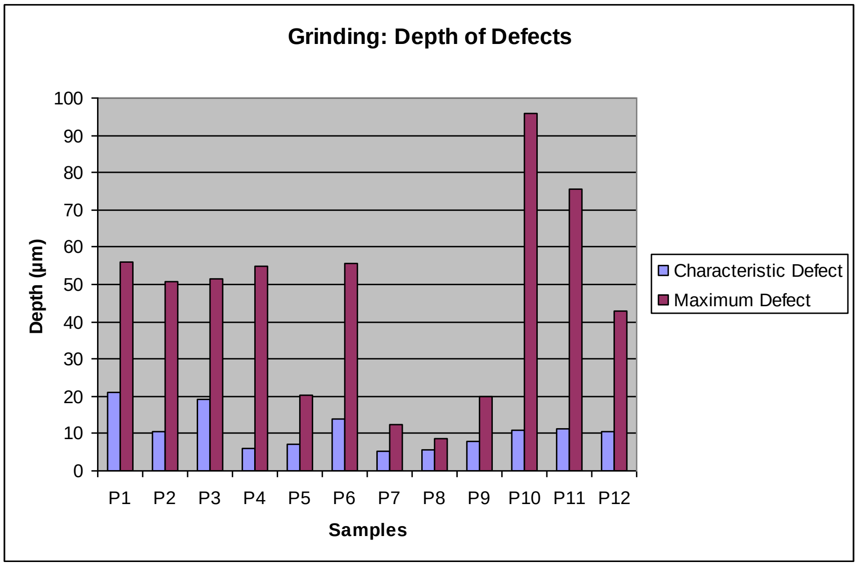

Fig. 5. The characteristic and the maximum depth of defects in the ground samples for Round 1 experiments. The light bars represent the characteristic depth of defects, and the dark bars represent the maximum depth of defects.

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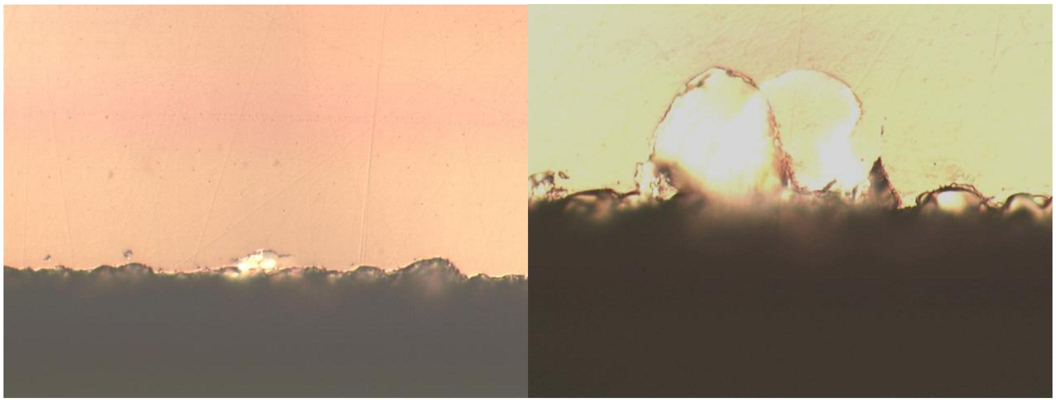

Fig. 6. Representative photomicrographs (1000x magnification) of damage sites: (a) a small defect characteristic of the P-8 sample; (b) the maximum defect of the P-10 sample.

The Minitab parametric analysis of the data showed no clear correlation with the controlled grinding parameters. Again, interacting second-order effects are likely at work. Although factorial analysis did not yield any significant uncoupled first order results, trends in the data suggest that deionized water was influential in minimizing sub-surface damage. Samples P-7, P-8, and P-4 all used deionized water in their process. Further, the two worst samples, P3 and P1, both used tap water in their process. This again suggests that slurry chemistry is involved.

3.4 Material Removal Rate

The total average removal rates of Round 2 samples are shown in Figure 7. The Round 1 results are consistent with these data. Because complete removal rate information was not collected, a factorial analyses using the Minitab software was not possible. However, using the available information, it is apparent that one process was the best at increasing removal rate. Not surprisingly, the parameters yielding the highest removal rate included Heavy Added Weight, High Arm Oscillation Speed and High Slurry Viscosity. Interestingly, these parameters are also favorable for minimizing surface roughness.

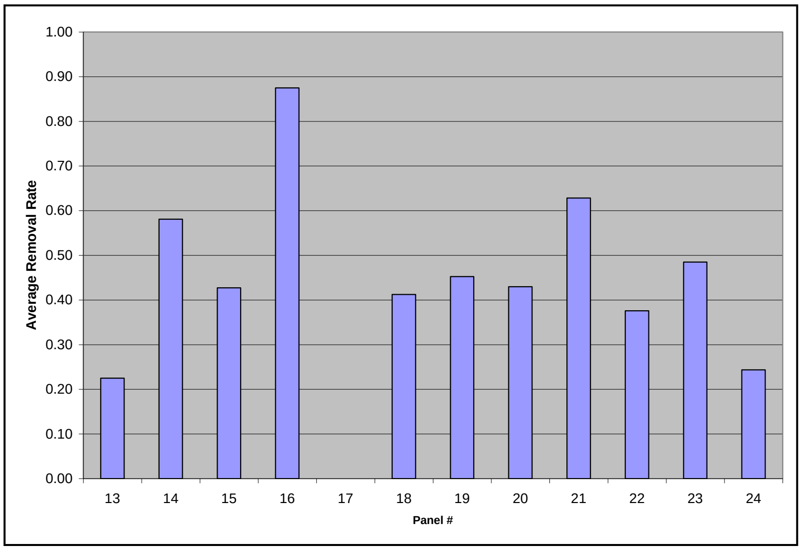

Fig. 7. Total average removal rates of Round 2 samples. No data is available for sample 17.

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4. CONCLUSION

This research program investigated the critical parameters in the grinding operation for large sapphire window panels in order to develop a better understanding of the process with an ultimate goal to achieve improvements in four output properties. The parametric trends observed in each of those characteristics are as follows.

4.1 Final surface roughness

- The grinding parameters that most improved the final surface roughness were:
  - heavier added weight;
  - larger starting grit size;
  - deionized water type.
- Higher initial removal rate does not result in a degraded final surface roughness because initial differences in surface roughness of ground panels are quickly normalized through the successive reduction of grit size during grinding.

4.2 Surface Stress

- The results with respect to surface stress were inconclusive. There were no clear correlations observed.
- Randomness of local grinding action is the likely cause of apparent randomness in peak shifts.

4.3 Sub-surface damage

- Water Type appears to be a significant factor in induced sub-surface damage.
- Parametric analyses yielded no clear correlation between sub-surface damage depth and grinding parameters.

4.4 Material removal rate

- The grinding parameters that most improved the removal rate were:
  - heavier added weight;
  - higher slurry viscosity;
  - faster arm oscillation speed.

Further investigations will be focused at optimizing the settings of the parameters identified as most influential. At present, those parameters are: Water Type, Arm Oscillation Speed, Added Weight and Slurry Baumé. In addition, the data suggest that interacting second-order effects may play an important role. Further experiments are required to allow extraction of those effects in order to fully optimize the sapphire grinding process.

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