Viability Testing of Polymer Coating for Optical Cleaning Applications

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1. Abstract

The performance of optics used in high-power laser systems can be limited by surface contamination in the form of particles and films. Viability testing was performed for a commercial polymer (First Contact^{TM}) as a strip-coat cleaning process for glass substrates, multilayer dielectric (MLD) coatings, and MLD diffraction gratings. The polymer can be brushed or sprayed onto the desired optic surface; it is then mechanically removed. Contact angle measurements, particle counts, force measurements, and damage testing were all used to determine the polymer's viability as a cleaning process. A process was developed that successfully removed contamination particles from flat optics, and prevented the polymer from leaving behind a thin film of contamination. For structured films such as MLD gratings, preliminary results suggest that a different application process needs to be developed.

2. Introduction

Multi-layer dielectric (MLD) gratings are used in pulse compression and stretching, key aspects of chirped pulse amplification [1]. This process creates high-intensity ultrashort optical pulses. This technique is currently used with the OMEGA EP laser system at the Laboratory for Laser Energetics. Particle contamination on the surface of optical components has been shown to significantly decrease the laser-induced-damage threshold (LIDT), increasing the likelihood of the optics being damaged [2]. Due to diffraction gratings' fragile structure consisting of rows of pillars, cleaning is difficult without damaging their structure; therefore they are often cleaned entirely using chemical processes. The company Photonic Cleaning Technologies [3] offers a commercial mixture of polymers and solvents, First Contact^{TM}, designed for strip coat cleaning of optics, including diffraction gratings. When the mixture is applied to the surface, it engulfs contamination, such that the contamination can be removed along with the polymer after drying. Ideally, this process leaves behind no residue, and the removal is mechanically gentle enough to preserve the quality of the optical surface. Practically, any incomplete drying or incomplete removal of the polymer could potentially introduce its own film of contamination onto the optics, and localized stresses caused during the polymer removal could damage the structure of the diffraction gratings.

An optimized cleaning process that addresses the above concerns could potentially increase the operational lifetime of diffraction gratings. In this work, we take steps toward development of such a process. Using various methods such as contact angle measurements, particle counts, force measurements and damage testing, we demonstrate a process that successfully removes particle contamination from optics, prevents the polymer from leaving behind a thin film of contamination, and subsequently provides a preliminary attempt at cleaning the fragile structure of MLD gratings.

3. Instruments and Evaluation Techniques

Particle density measurements were used to evaluate the efficacy of the polymer for cleaning the surface of the samples. The particle density of a sample is determined through optical microscope imaging. The entire surface of a sample was imaged in a grid pattern; commercial software was then used to count the number of particles (>1.8 μm in diameter) in each image to determine the particle density for each sample. Cleaning effectiveness was evaluated by comparing the particle density before and after a process. The particle density from each image could be plotted to provide a particle density map of a sample, which was a useful tool in evaluating the cleaning effectiveness across a sample.

Contact angle measurements were used to evaluate the cleanliness of a sample and the degree to which the polymer coating was removed from the sample. The contact angle is defined as the angle that a liquid makes with a surface when wetted. See Figure 2(a) below for examples of the measurement. The contact angle is measured by dropping a small fixed amount of water onto the sample and using a camera and computer analysis to measure the angle. Film contamination can be detected by comparing the contact angle from before and after the polymer is used to clean an optic. For this study, clean glass is very hydrophilic with low contact angles, whereas glass coated with an organic film tends to be more hydrophobic and has higher contact angles.

It was found that the water droplets from the contact angle measurement could contribute to the particle load on the sample, thus possibly altering the particle density results of subsequent cleaning experiments. Three different water droplet drying techniques were tested and the particle density was compared before and after the water application and drying as shown in Figure 1. Figure 1(b) shows that the prompt removal of the water droplets with a nitrogen gun was highly effective in removing the particles contributed by the water and resulted in a small reduction of the particle density (number of particles per unit area on a surface): values on the order of -10 counts/mm^2 are seen in Figure 1(b). In contrast, the other methods resulted in large increases in the particle density. The nitrogen gun method for drying was therefore used in the subsequent experiments.

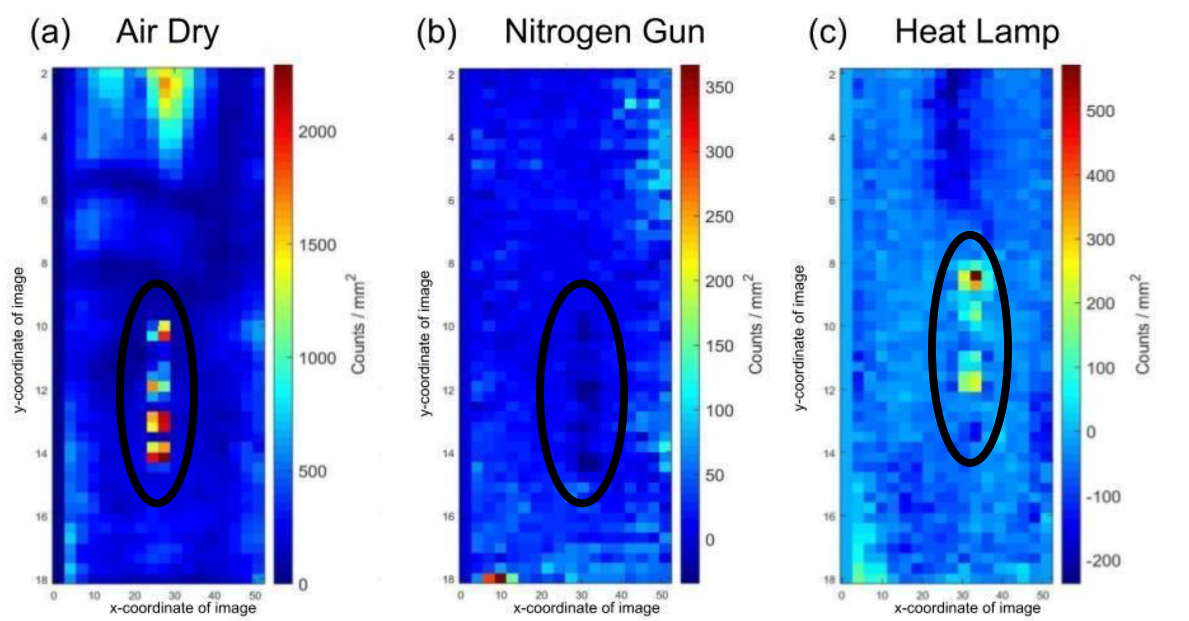

Figure 1: Maps of particle density changes on glass slides for three different methods of removing contact-angle water droplets: (a) allowed to air dry at room temperature, (b) blown off with a nitrogen gun, or (c) air dried with a heat lamp. The black ovals indicate the regions where the water droplets were applied. The X and Y axes correspond to coordinates on the glass slide. The color indicates the difference in particle count per mm^2, counts after minus counts before the contact angle measurement.

Laser damage testing of the MLD mirrors was performed using a laser system operating at 1053 nm and having a tunable pulse duration between 0.6 ps and 100 ps. This system has been described in detail elsewhere [4]. By comparing results before and after polymer use, the laser damage testing characterizes the polymer's impact on optical performance. If the polymer mixture is not fully removed in the processing of a nominally pristine sample, the damage threshold would be anticipated to decrease.

4. Developing an application and removal process

During initial testing of the polymer, the manufacturer's recommended procedures were used. However, measurements performed on the treated samples showed an increase of water contact angle after polymer treatment, suggesting a thin film of residual material from the polymer solution. Given the sensitivity of the intended application (high-powered lasers) to surface contamination on the optics, experiments were performed to attempt to eliminate this contamination by modifying the process parameters.

In order to effectively develop and implement a cleaning process with the polymer solution, glass microscope slides were used as test subjects. These slides, which possess a less uniform surface and a higher degree of contamination than clean MLD optics, were used to develop a procedure for applying, drying, and removing the polymer.

The general procedure starts with application of the polymer coating onto the samples via brushing or spraying. When brushed onto the optics, the optic was laid flat, polymer was poured onto the optic, and the surface tension between the polymer on the optic and the brush was used to spread the polymer. When sprayed onto the optics, the optic was held vertically, and the polymer was sprayed in multiple light coats to prevent the polymer from dripping. The coating was applied to a chosen thickness, such as, for example, to the appearance of the pink color of the manufacturer's visual aid that corresponded to approximately 5-10 spray coats. Tape was applied around the outer edges of the optic when spraying, to prevent the polymer from getting onto the side edges of the samples, because this caused an increase in the difficulty in fully removing the polymer from the optical surface. In order to later remove the polymer, a mesh or floss peel tab was embedded into the wet polymer coating. Then the sample was allowed to dry for a period of time, after which the mesh or floss tab was used to peel the polymer from the optic.

The initial experiments, which used the manufacturer-recommended instructions, showed that the contact angle increased after the polymer treatment (brush and spray section of Figure 2). Before removal, the polymer coating has a contact angle of approximately 90 degrees, much greater than the baseline contact angle of the glass substrates. Therefore, an increase in contact angle indicates that a film of contamination is left behind by the polymer because the surface of the optic is more similar to the polymer than it was before. We hypothesized that this was caused by incomplete curing of the polymer. This was tested by applying a spray coating, then allowing the polymer to cure overnight (approximately 24 hours) instead of 1 hour, and alternatively by applying heat with a heat lamp as the polymer cured. However, no significant changes were found with these alternate curing methods (overnight and heat lamp section of Figure 2). Furthermore, there was a significant decrease in particle density after the polymer was used to clean the glass substrates for all cases (Figure 3). This indicates that the polymer was reducing the particle contamination load for the samples, and that the longer or hotter curing provided no advantages. Therefore, subsequent experiments maintained the 1 hour drying time at room temperature.

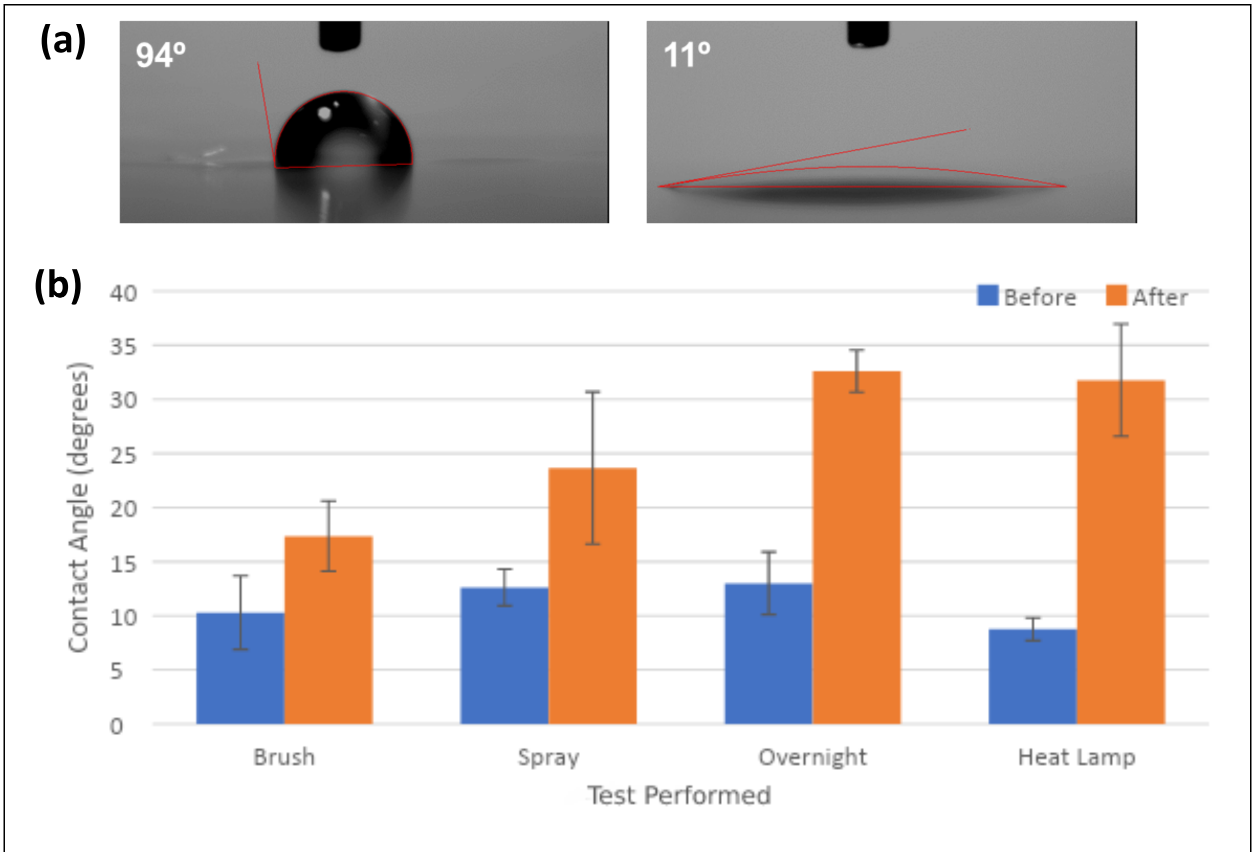

Figure 2: (a) Images from contact angle measurements of the polymer coating (left) and a glass slide (right). (b) Contact angle measurements for different polymer application and drying techniques. The

Overnight and Heat Lamp drying methods were performed with spray-applied polymer coatings. The blue bars are the measurements before application and the orange bars are the measurements after removal.

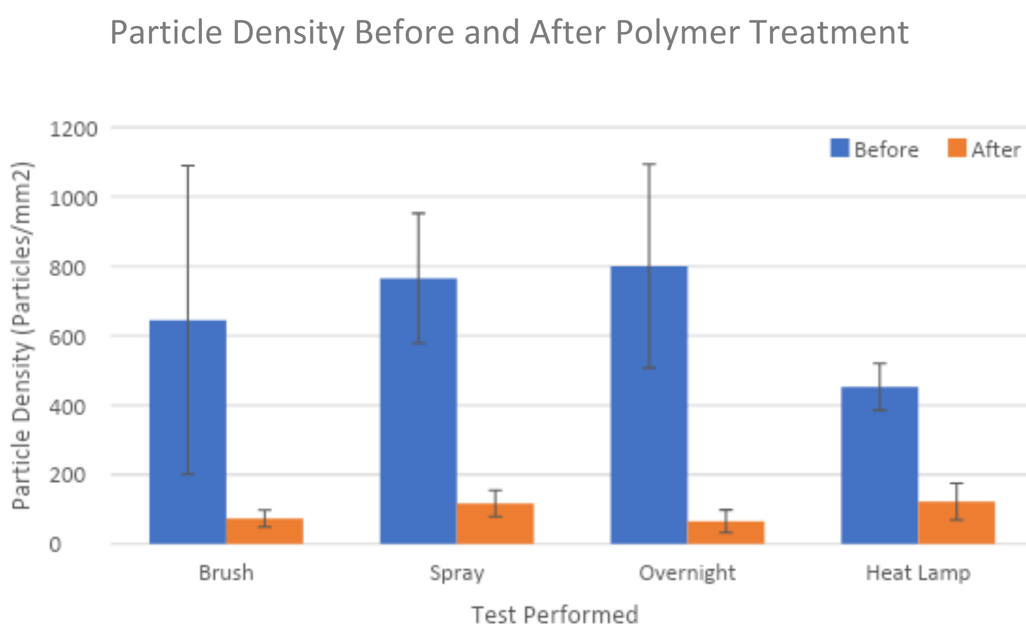

Figure 3: Cleaning efficacy for various application and drying techniques, reporting measured particle density in particles/mm^2 before (blue) and after (orange) the polymer treatment was used to clean the optics. The Overnight and Heat Lamp drying methods were performed with spray-applied polymer coatings.

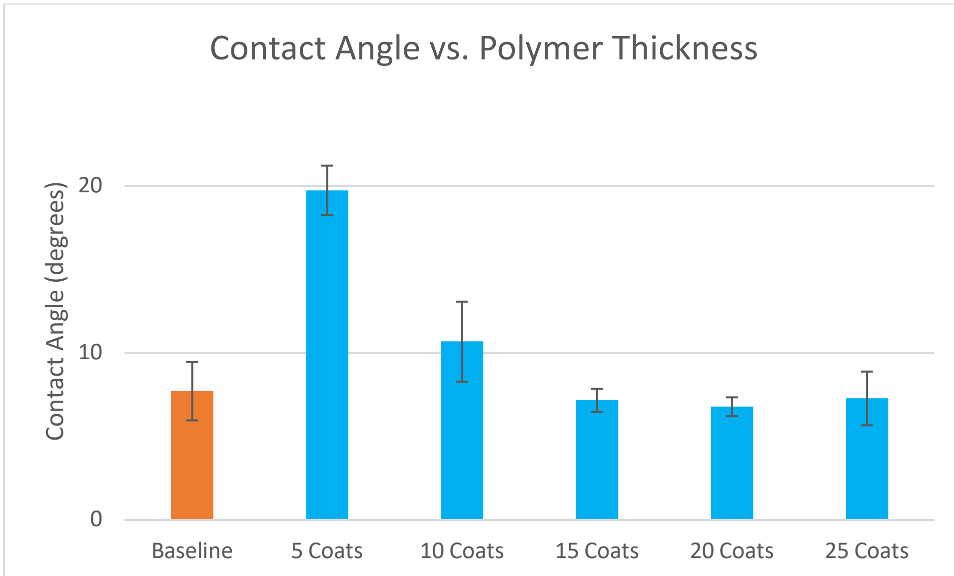

Figure 4: Measured contact angle in degrees after polymer treatment as a function of spray-coat thickness. Baseline shows the average contact angle of all glass substrates before treatment.

Since contact angle measurements indicated that alternate polymer curing methods still resulted in incomplete removal, it was alternatively hypothesized that the thickness of the polymer affected its ability to peel without leaving a film of contamination. This was tested by coating glass substrates with 5, 10, 15, 20, and 25 coats of the spray polymer. One coat was 2-3 pumps from the vendor-provided bottle sprayed at a distance of approximately 12 inches away. The polymer was given 1 hour to dry. As shown in Figure 4, it was found that as the thickness of the polymer increased, the contact angle decreased until around 15 coats when it was the same as or lower than the baseline. With the increased polymer thickness, the polymer was able to remove particle contamination without leaving a film of contamination.

In summary, the final application process resulting from this work with glass slides used 20 spray-coats of polymer, each coat being 2-3 pumps from approximately 12 inches away using the vendor-provided bottle, with at least 1 minute between each coat. The peel tab was embedded in the final 2 coats. The polymer was then given 1 hour to cure at room temperature in horizontal orientation. The polymer was removed using a mesh peel tab in an apparatus using a force gauge and motorized stage.

5. MLD Mirrors and Gratings

Once a process for using the polymer was established, the process was tested on few-square-inch optical component samples that are representative of the optics used in the OMEGA EP laser system, such as MLD mirrors and gratings. The objective was to demonstrate that the developed process effectively cleans particles on actual optics, without leaving its own film of contamination. The MLD mirrors are manufactured by coating multiple layers of dielectric materials (typically silica and hafnia in high-LIDT applications) onto glass substrates. The MLD gratings are MLD mirrors with a thick top layer (typically silica) that has sub-micron lines or grooves etched into it. The MLD mirrors were tested before gratings due to the simpler surface geometry.

5.1 MLD Mirrors

Figure 5: Contact angle before (blue) and after (orange) the polymer was used to clean the MLD mirrors.

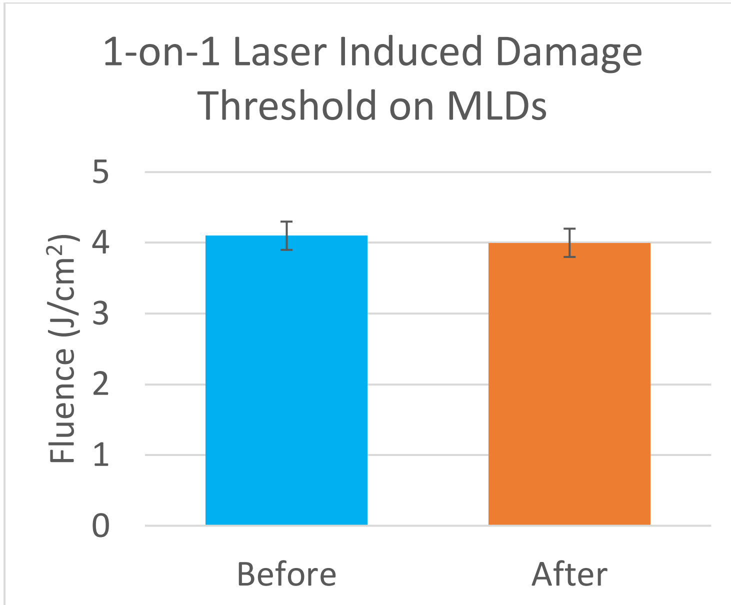

Figure 6: Fluence that the optics could withstand before sustaining damage. This was measured before and after the polymer was used to clean the optics. These measurements were taken in a vacuum with a 42-degree angle of incidence, 10 ps pulse duration, S-polarization, and a wavelength of 1053 nm. These parameters are based on the MLD mirrors' design and conditions the mirror would be exposed to in OMEGA EP.

The results of contact angle measurements, laser-induced damage testing, and particle counting suggest that the process developed with glass microscope slides applies well to MLD mirrors (with no surface structure). Contact angle measurements show no change before and after the polymer was used to clean the optics (Figure 5). Additionally, the laser-induced damage threshold was measured before and after the polymer was used to clean the optics. The LIDT corresponds to the laser pulse energy per unit area that the optics can withstand before sustaining damage. Since there was insignificant change in the LIDT before and after the polymer treatment (Figure 6), this provides an independent and practically relevant result indicating that the polymer left no relevant film of contamination on the optics.

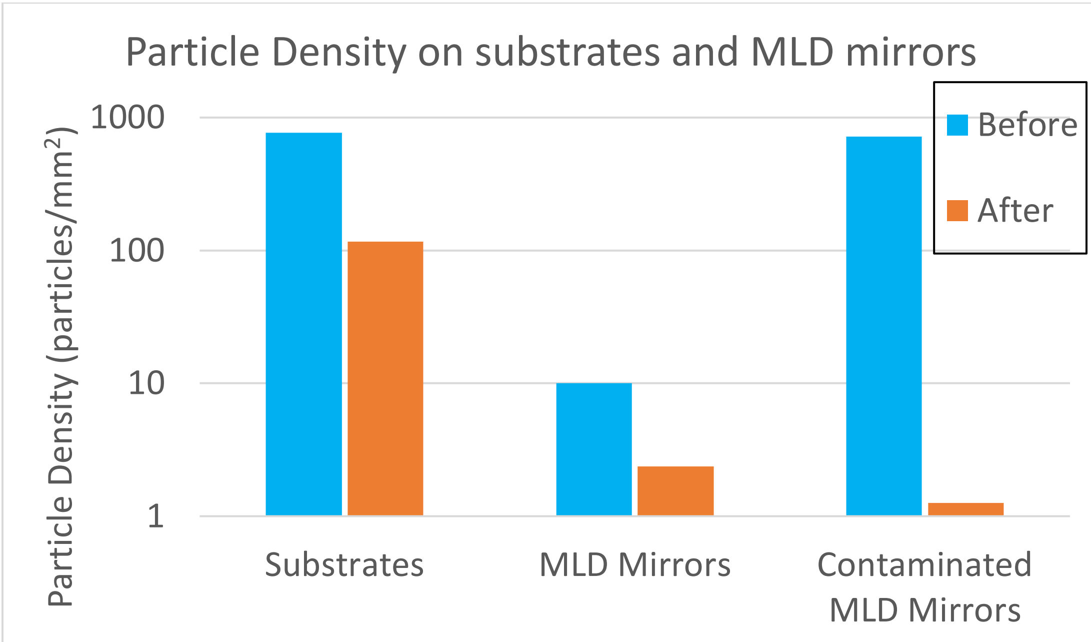

Figure 7: Measured particle density in particles/mm^2 before and after the polymer was used to clean glass substrates, MLD mirrors, and contaminated MLD mirrors.

Particle density was measured on clean glass substrates and on clean and intentionally contaminated MLD mirrors. The "contaminated" MLD mirrors had 325-mesh stainless steel particles gently poured onto the glass while held horizontally; the glass was then tilted vertically and shaken gently until the particles were not visible to the naked eye. In all cases the particle density decreased significantly, indicating that the polymer was cleaning the optics of particle contamination (Figure 7). It is worth noting that some small fraction of the "particles" identified by the microscope image analysis may not actually be particles at all, but may correspond to other defects such as scratches or digs. This implies that the actual cleaning efficacy may be higher, since the after-treatment value of particle density may include such non-particle defects. Furthermore, the extremely low particle density of the cleaned MLDs suggests that the polymer treatment is mechanically gentle on the surface, since any new scratches/digs generated during the processing would be recorded as particles.

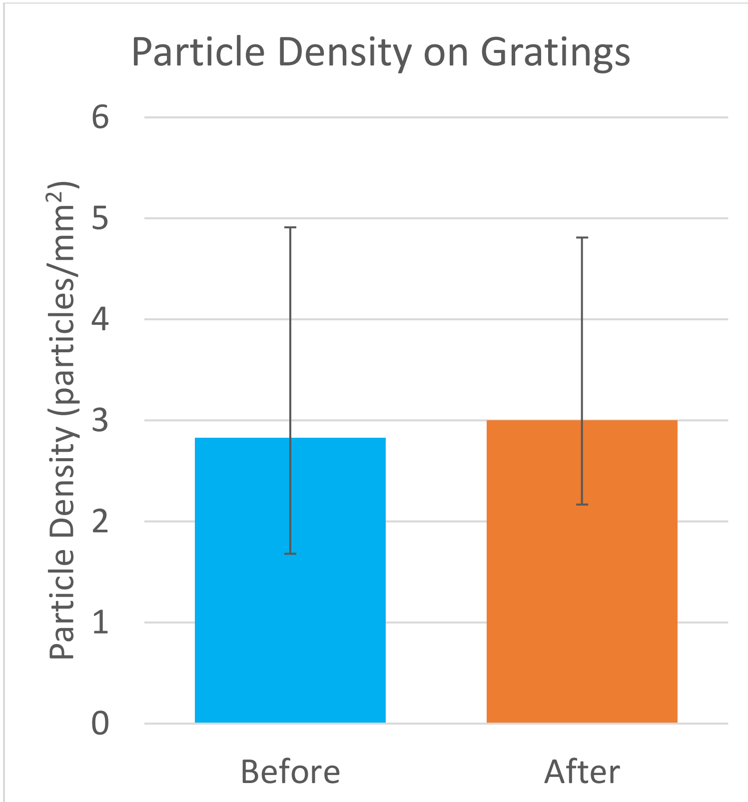

Figure 8: Measured particle density in particles/mm^2 before and after the polymer was used to clean an MLD grating.

5.2 Gratings

The concluding set of experiments in this work involved preliminary testing of the developed treatment process on MLD diffraction grating samples. A grating's structure consists of lines of pillars. For the gratings used, the grating spacing period was 574 nm with 575 nm tall by 200 nm wide pillars. Because of this, the polymer requires a greater cure time to get into the gratings' structure. First Contact™ recommends letting the polymer cure overnight. Aside from this, the procedure for applying the polymer was the same as for the MLD mirrors. When peeling the polymer from the grating, the polymer was peeled parallel to the pillars of the grating to minimize the stress on the pillars.

The initial state of the grating samples was nominally clean, and the particle density did not change after the polymer was used to clean the optic (Figure 8). Since damaged pillars could be counted as particles, as described previously, this indicates that the polymer didn't significantly damage the structure of the gratings. However, in this preliminary work the surface of the polymer after removal did not look to be a replica of the grating surface, which would have generated an appearance of structured coloration. This indicates that the polymer may not have penetrated into the grooves of the grating to properly clean it. Nonetheless, the lack of mechanical damage of the pillars is an encouraging result at this preliminary stage of the research with polymer cleaning of grating samples.

6. Future work

Due to time constraints, it was only possible to successfully peel the polymer from a grating once. Therefore, additional trials are necessary to ensure that the process is repeatable. Another important set of future experiments would involve adjusting process parameters to better clean the structured surface of the gratings. The First Contact™ product line includes a thinner which can be added to the polymer to decrease its viscosity. This could potentially help the polymer to penetrate between the pillars better.

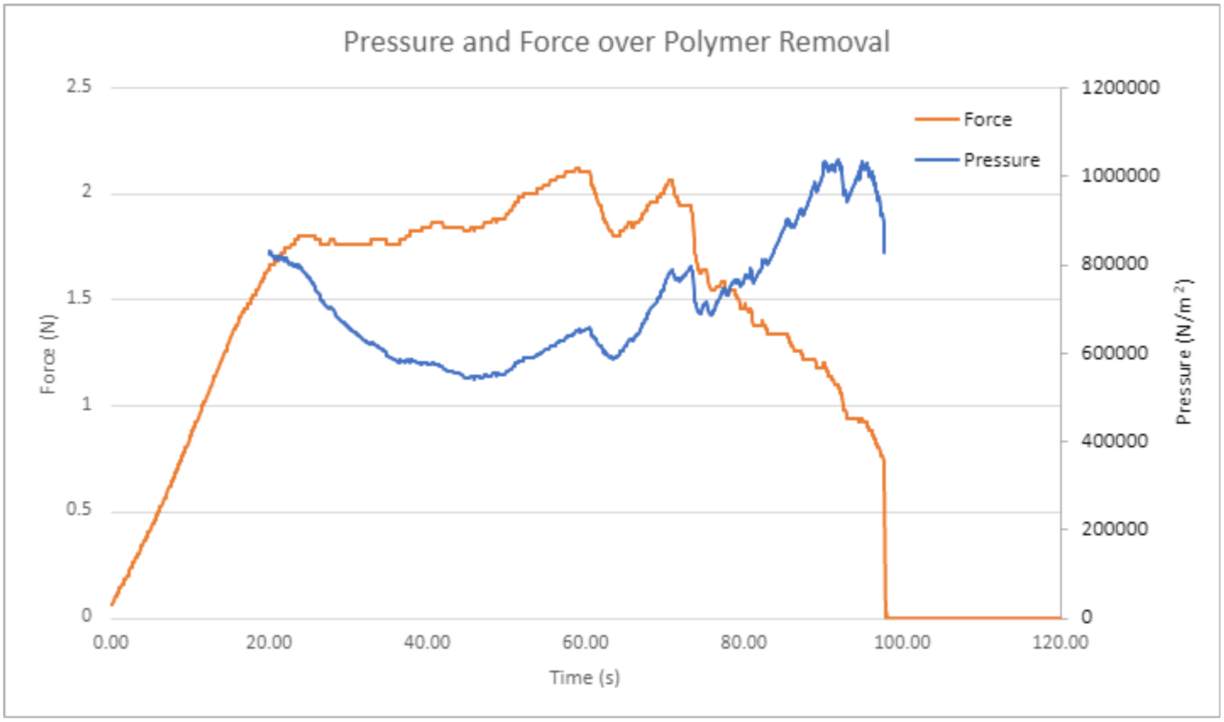

Figure 9: Force and pressure as a function of time during polymer removal from a MLD grating. The pressure, determined using the force, is only plotted over the duration of the polymer coating being peeled.

For structured surfaces such as diffraction gratings, future research could be valuable in the optimization of the polymer removal procedure. Specifically, surface structures change the adhesion of the polymer coating, and the force required may also be a function of the peeling geometry. In our preliminary experiments, the polymer was only peeled parallel to the grating's pillars. Peeling the polymer perpendicular to the grating's pillars may impact the cleaning effectiveness and/or the resistance to mechanical damage of the pillars, due to change of local stresses. While the measurement or calculation of the localized stresses in the pillars would require significant effort and resources, measurement of the total force during the peeling of the polymer may be a useful metric for this purpose. An apparatus was developed using a motorized stage and a force gauge attached to the peel tab, thereby measuring the total force applied as the polymer was being peeled.

An example set of force data during removal of the polymer from a MLD grating is shown in Figure 9. The force data shows that as the polymer is peeled, the force remains relatively consistent between 1 and 2 newtons. Though this data does not directly represent the localized stresses on the pillars, we may be able to estimate the average "pressure" P by dividing the measured force F by the approximate area A that is actively being peeled:

$$P = F/A$$

Note that both *F* and *A* can be functions of time, especially since the samples are not generally rectangular. The active peeling area was approximated as a long thin rectangle, with the short side aligned with the peeling force vector and the long side determined by the width of the sample (determined through video analysis due to the gratings' sector shape). For simplicity, the short dimension of the active area was assumed to have a constant characteristic length of 0.1 millimeters.

By measuring the pressure exerted on the optics during the removal of the polymer, we can potentially establish a practical threshold that diffraction gratings can endure without incurring damage. This information would enable us to make informed decisions on the most appropriate peel tab method, whether it be mesh or floss, and the optimal angle for peeling the polymer in order to prevent damage to the gratings.

7. Conclusions

The commercial polymer First Contact^{TM} was tested as a strip-coat cleaning process for glass substrates, multilayer dielectric coatings, and diffraction gratings for high-power laser systems. Contact angle measurements, particle counts, force measurements, and laser damage testing were used to evaluate the effectiveness of the polymer treatment as a cleaning process. The results showed that the polymer was successful in removing contamination particles from flat optics and that the developed process prevented the treatment from leaving behind its own thin film of contamination. However, preliminary results from the treatment of MLD gratings suggest the need for development of a modified process for treatment of structured surfaces.

8. Acknowledgements

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9. References

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