

Laser Polishing of Micro-Machined Microfluidic Molds

ICOMM/4M
2010
No. 75

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ABSTRACT

The objective of this paper is to explore the effectiveness of using a pulsed 1064 nm wavelength Nd:YAG laser to polish micro-machined metal molds. Polishing is desired to reduce the surface roughness of Polydimethylsiloxane (PDMS) devices cast with these molds. Reducing the roughness on polymer microfluidic devices is desirable in order to reduce fluidic resistance, control surface friction, improve optical transparency, and improve the bonding of mating surfaces. This study is focused on modifying the surface roughness of molds in order to improve the finish of cast PDMS devices, thereby enhancing their ability to bond to a glass substrate; standard practice to seal channels when manufacturing a microfluidic device.

The results of the laser parameters used in this study showed successful polishing of features with spatial frequencies above $\sim 75 \text{ mm}^{-1}$ (i.e., short wavelength features). Surface features with smaller spatial frequencies (longer wavelength) remained relatively unaffected. Adhesion tests only correlated with the surface roughness metrics that capture the low spatial frequency ($< 75 \text{ mm}^{-1}$) features. These results demonstrate that adhesion (bond strength) is a function of the wavelength (and amplitude) of surface features. In order to completely describe a bonding interface, roughness metrics need to be obtained across the range of feature sizes; i.e., data collected and analyzed at different magnifications/resolutions. Laser polishing parameters that target longer wavelength features must be applied to the molds in this study in order to enhance the adhesion of the cast PDMS surfaces.

INTRODUCTION

Micromachining allows for flexible production of precision parts with micrometer-sized features, such as molds to be used for micro-casting microfluidic devices. However, all manufacturing processes, including micro-machining, leave distinct surface finishes on the produced components. In addition, achieving sufficient precision in these parts is becoming more of a challenge because surface roughness levels are approaching part tolerances [1-4]. Additionally, the surface roughness of micro parts can present problems. For example, surface roughness can affect fluid flow in micro-fluidic applications [5]. In the case of micro-molds, surface roughness affects the quality of the replicated parts. The accumulation of plaque on metallic dental implants is

dependent on surface roughness [6]. For moving parts, the surface roughness can increase friction, wear [3] and heat generation at the interface. This study focuses on the effect that surface roughness has on the adhesion of a PDMS surface to a glass substrate.

Micro end milling inevitably leaves various patterns on the surface of a mold that will be replicated onto the final piece during the molding processes. These patterns are visible in Figure 1, which shows an optical micrograph of an aluminum surface micro-machined with a 500 μm diameter end mill at 10,000 rpm, 9 μm chip load, and 25 μm axial depth of cut.

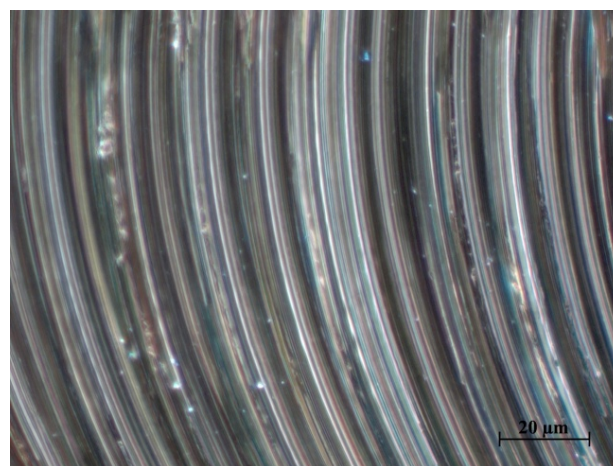


Figure 1: Pattern of surface features left by micromachining

Surface features are inevitable due to the tool-workpiece interactions, however, periodic nature and the surface roughness can be minimized by using a polishing operation. One process for selectively polishing areas on micro-scale parts is pulsed laser micro-polishing (PL μ P). The principle of polishing surfaces with pulsed laser radiation is based upon locally melting a shallow pool of material with negligible ablation [7-10]. Unlike polishing with continuous (CW) laser radiation where melt depths of tens to hundreds of micrometers are typical, the melt depth during pulsed laser polishing is in the order of one micrometer [11, 12]. While the pool is molten, surface tension forces attempt to “pull down” asperities with small radii of curvature. The

energy density (fluence, J/mm^2) deposited in the surface over a specified amount of time (pulse duration, ns) is critical because it controls the temperature history, *i.e.*, if phase change occurs, and how long the melt pool exists. Insufficient energy will not affect the features, while too much energy will ablate and remove material from the surface. Ablation of material usually is associated with considerable degradation of surface quality. For this reason the amount of energy deposited in the surface and the duration of this deposition process must be precisely tuned in order to reduce the magnitude of the desired surface features. Perry et al. [8-10] found that laser pulse duration had a significant impact on the surface features that were significantly reduced in amplitude. Longer laser pulse durations enable shorter spatial frequency (*i.e.*, longer wavelength) features to be polished. Therefore, the laser parameters chosen can also selectively polish certain surface features while leaving others unaffected.

Pulsed laser micro-polishing is a non-contact finishing technique that enables the selective polishing of 3D geometries and does not produce any debris. This work aims to characterize the effect of pulsed laser micro-polishing on features created by micro end milling, and to quantify the various surface textures on the adhesion of cast PDMS to glass substrates. The interferometer image shown in Figure 2 demonstrates the effect of laser polishing: an area has been imaged containing both polished and unpolished zones. Laser polishing shows great promise as a technique for polishing otherwise inaccessible and select areas [11-16].

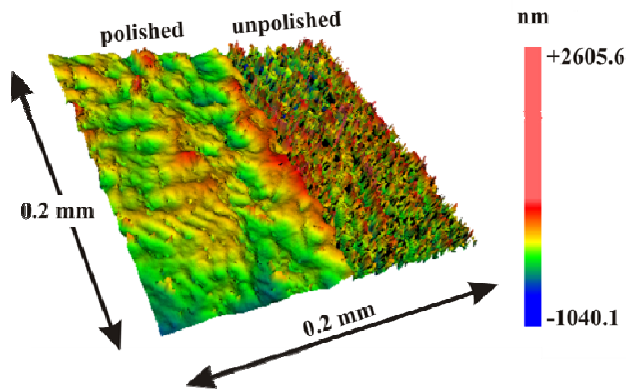


Figure 2: Effect of laser polishing

The effect of the reduced surface roughness on the adhesion of cast PDMS against glass is measured by adhesion testing cast PDMS samples and correlating adhesion bond strength to the cast samples surface roughness metrics (measured at different scales). Previous studies of the adhesion of PDMS to glass as a function of surface roughness showed as a general trend that the adhesion strength decreases with increasing roughness. These observations were also made in previous publications modeling adhesion strength and surface roughness interactions [17-19]. Due to the complex nature of surface features and the multitude of components that contribute to the catch all term “surface roughness”, investigation into which aspect or combination of aspects of surface roughness contribute to the adhesion

bond strength must be considered. For that reason multiple surface roughness metrics are taken and correlated to the change in adhesion. A dominant component of surface roughness focused upon in this study is the combination of different wavelength features upon a surface, as illustrated in Figure 3. The effective scale of these features plays an important role in the adhesion of elastic bodies.

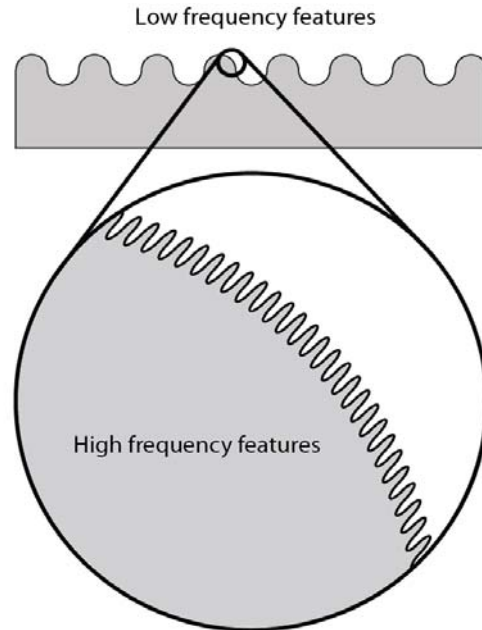


Figure 3: Simplified schematic of surface roughness features at various frequencies

EXPERIMENTAL METHODS

A. MICROMACHINING

The test molds were made out of SAE Grade 304 stainless steel. Machinability and material properties of this alloy make it a suitable candidate to be both micro machined and laser polished. The sample surfaces were machined onto the mold using an Atometric micro-end-milling system (Atometric Rockford, IL), industrial commercially-available machine specifically designed and devoted to micro-end milling. The tools used to manufacture the specimen were 2-flute 500 μm tools commonly used for micro endmilling tasks (PMT Micro tools Janesville, WI). Table 1 outlines the machining parameters used to create the molds. This computer numerically controlled micro end mill setup is reported to have capability of .6 μm position precision control. The different surfaces were created by adjusting the chip load used to machining each surface. Chip load is defined as the amount of material removed per tooth measured in the tool travel direction and is a function of feed rate and spindle speed. Changing the chip load for each surface created changes in the material removal dynamics, resulting in varying surface topographies. The specimens were all machined as a blind cut into a pre-planarized surface. The machining technique consisted of a number of constant depth parallel passes with prescribed step over to create the planar surface of interest. Each surface was machined ac-

ording to the feed rate and spindle speed shown in Table 1. The result was a set of surfaces with consistently varying topographies due to differences in feature spatial frequency content and amplitude trends.

Table 1: Machining and polishing parameters

Machining Parameters		Laser Processing Parameters	
Chip load	1, 3, 5, 7, 9 μm	Wavelength	1064 nm
Axial Depth of Cut	25 μm	Pulse Duration	650 ns
Spindle speed	20,000 RPM	Pulse Energy	0.62 - 0.94 mJ
Milling mode	Climb	Pulse Rate	4000 Hz
Tool Dia.	500 μm	Laser Spot Size	60 \pm 10 μm
Tool Type	2 flute-Stub	Scan Speed	45 mm/s
Stepover	70 μm	Dist. from Focus	1 mm
coolant	Mist applicated fluid	Scan mode	Zig-zag

B. PULSED LASER MICRO POLISHING

In order to experimentally prove the benefits of pulsed laser micro polishing on adhesion of cast components, one set of micro machined surfaces was polished using a 1064 nm wavelength laser (Nd:YAG Lee Laser Model 8250 MQ Lee Laser Orlando, FL). The laser was operated in Q-switch mode to deliver pulsed radiation. The energy in the pulse was varied for each sample due to observation that different wavelength samples require different polishing parameters to achieve polishing and to avoid ablation. For samples machined with chip loads of 1, 3, 5, 7 and 9 μm , the pulse energy was set to 0.625, 0.8125, 0.875, 0.9375 and 0.9375 mJ, respectively. Increased chip load of each sample meant increasing dominance of lower spatial frequency contents requiring more energy in order to be affected by the laser pulse. The beam was traversed along a zigzag pattern using a Scanlab HurrySCAN II (Scanlab Puchheim/Germany) scanhead. Refer to Table 1 for a list of laser processing parameters.

Laser pulse durations of 650 ns and laser spot diameters of \sim 60 μm are used in this study. Depending on material properties such as heat capacity, thermal conductivity and absorbance as well as process parameters such as pulse duration, there is an energy window for effective polishing unique for each of the surfaces. Too little energy input will only heat the material, whereas too much energy input will roughen the sample through ablation. In addition, the longer the material stays molten the more smoothing occurs therefore affecting features of lower spatial frequency content [1].

C. CASTING

Production of the actual PDMS samples attempted to mimic as closely as possible the actual manufacturing techniques and procedures that would be used in real device fabrication. Surface preparation of the mold consisted of cleaning using an ultrasonic bath, followed by consecutive rinsing in acetone, ethanol, methanol, and then isopropyl alcohol to remove any contaminants or residues. The PDMS mixture was prepared by mixing a 10:1 by weight ratio of Sylgard 184 silicone elastomer base to Sylgard 184 curing agent (Dow Corning Corporation Midland, MI), which was thoroughly stirred and degassed under vacuum to remove trapped air bubbles. The PDMS was then carefully poured

into the mold and any subsequent bubbles were mechanically removed using a sharp needle point. The PDMS was then cured at 80 $^{\circ}\text{C}$ for 2 hours on a hotplate. Once cured, the PDMS samples were peeled from the aluminum mold, the backside of the cast samples were oxygen plasma treated, and the sample was bonded to a microscope slide to ensure rigidity during testing.

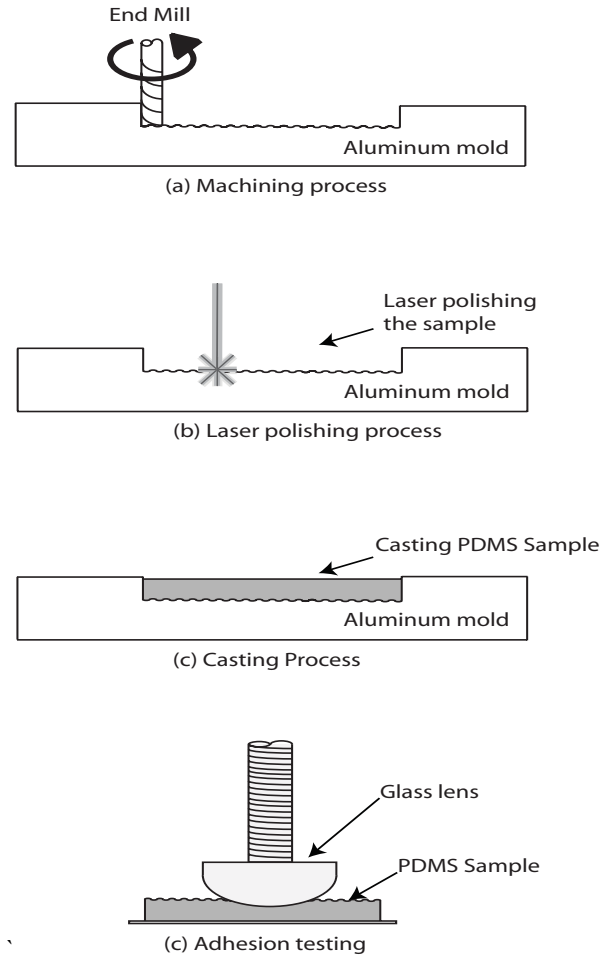


Figure 4: Experimentation process

D. SURFACE CHARACTERIZATION

The analysis of the specimens was done using a New View white light interferometer (Zygo Fremont, CA) in order to capture the surface topography at the micro-scale. White light interferometry is a non-contact measurement technique capable of submicron measurements. Before taking measurements the surfaces were cleaned of all debris and oils in several steps in an ultrasonic bath, followed by consecutive rinsing in acetone, ethanol, methanol, isopropyl alcohol and de-ionized water to remove any contaminants or residues. Data was recorded for both metal mold surfaces before PDMS was cast into them, and for the cast PDMS surfaces. All measurement were taken at 10x and 40x magnifications. While the reflectivity of machined steel was conducive for imaging, the cast PDMS surfaces needed to be gold-coated to increase reflectivity. A 20 nm thick gold

coating was applied, which did not have a significant effect on the surface topography measurements. Interferometer data for all surfaces was exported as data files and imported into Scanning Probe Image Processor, (Image Metrology, Hørsholm Denmark), a microscopy analysis software. In SPIP, discretized height data of the specimens was evaluated by calculating various commonly used metrics for roughness [20-23]. A number of these roughness parameters were found to directly relate to the effects of laser polishing and adhesion. The most promising of the surface metrics are average height, and RMS height. S_a (Average height) is the averaged absolute height of the data points within the sampled surface. S_q (RMS height) is the root means squared height of the data points within the sampled surface. The sampled height data contains contents of a range of spatial frequencies. Content at very low spatial frequencies (long wavelengths) is considered waviness as opposed to content at higher spatial frequencies (short wavelengths) which is considered roughness. Interferometer images taken at different magnifications (10x and 40x) resulted in different resolutions (1.1 μm and 0.275 μm respectively). This means different ranges of roughness and waviness were captured at each magnification. Higher magnification images tend to capture higher frequency features while lower frequency images tend to capture lower frequency features both because of the pixel size and image size. Due to small spot size and short melt time it was found that pulsed laser irradiation does not affect the low frequency content of the samples as much as higher frequencies.

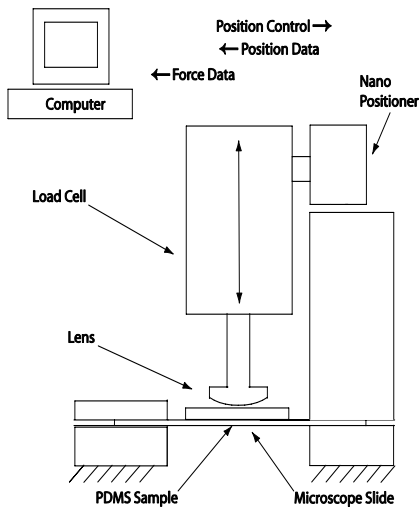


Figure 5: Adhesion testing apparatus

E. ADHESION TESTING

The work of adhesion was characterized using a Johnson-Kendall-Roberts (JKR) type test using the apparatus in Figure 5. The setup consists of a convex, 15.5 mm radius-of-curvature optical lens (Thorlabs, Newton, NJ) mounted on tension/compression load cell with a range 0.25 lb (LFS 242 Cooper Instruments & Systems Warrenton, VA). The load cell/lens assembly was positioned using a 3 axis linear stage (461-XYZ-M, Newport Corporation, Ir-

vine, CA). A NanoPZ actuator and controller (PZA12 Newport Corporation, Irvine, CA) is used to displace the contactor during the tests. The entire setup is secured to a sample fixture, which stabilizes and fixes all equipment relative to the sample. A MATLAB (The MathWorks) interface is utilized to control and record; speed, timing, position, and loads throughout the test.

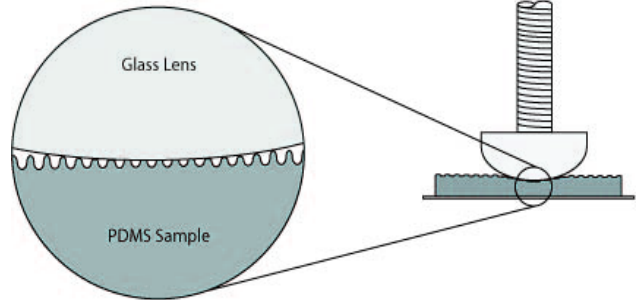


Figure 6: Adhesion testing lens to sample contact

In a typical test, a convex lens is brought into contact with the test PDMS surface using the micro-positioner (Fig. 6). The compressive force increases while the indenter moves into the surface at 7.6 μm per second until the set pre-load is reached. After a specified dwell of 5 seconds, the indenter is retracted from the PDMS surface at 12 μm per second. The pull-off force is defined as the load at which the tensile force reaches a maximum during retraction. This value is used to calculate the work of adhesion using the theory presented by Johnson, Kendall and Roberts [18, 24]. Adhesion tests were performed on the cast against polished and cast against machined surfaces for a series of trials each surface. The apparent work of adhesion was calculated knowing the pull-off force and indenter radius of curvature using the Equation 3, where P is the max load experienced during pull-off, γ is the work of adhesion and R is the radius of curvature of the indenter.

$$\gamma = \frac{2P}{9\pi R} \quad (3)$$

The term ‘‘apparent work of adhesion’’ is used because the measured value includes the effect of roughness as well as the surface energy.

RESULTS

It was found that laser polishing can significantly improve the surface roughness of machined surface. Although at the specific laser parameters chosen to polish our samples, polishing only affected very high frequency features. This occurred because laser parameters were chosen to enhance surface roughness at the 0.275 μm length scale (the resolution of the images used to verify results when choosing laser parameters which were taken at 40x magnification). Imaging showed surface roughness were improved significantly at small length scales as shown in Figure 6 which highlights the effectiveness of laser polishing of surfaces are measured at 40x. Each of these data points represents the mean of four measurements (each at a

different location) of average surface roughness for each sample.

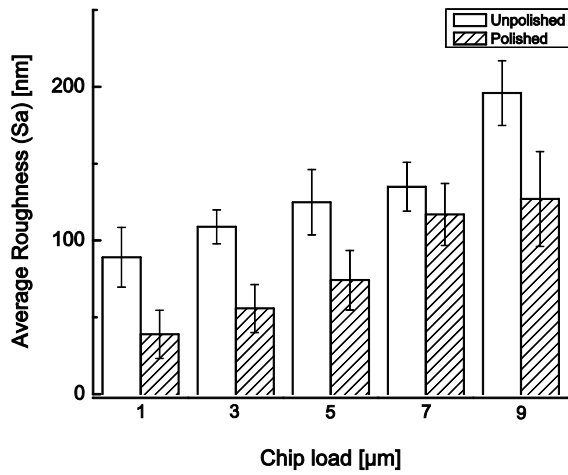


Figure 6: Apparent work of adhesion as a function of RMS gradient at high magnification (S_{dq})

The adhesion measurements revealed that the apparent work of adhesion for the polished surfaces did not follow the trend of improvement as maybe expected from the 40x magnification surface metric analysis in Figure 6. Figure 7 shows correlations of the surface roughness on the $0.275 \mu\text{m}$ length scale (taken from 40x magnification images) to the respective adhesion values for each sample. It can be seen that there is no significant trend between the adhesion and roughness at this small length scale.

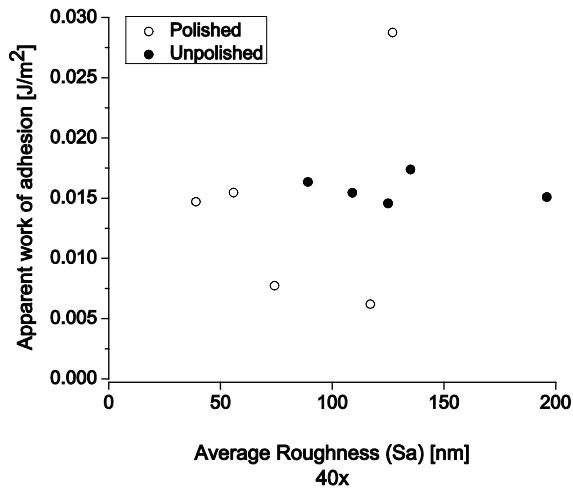


Figure 7: Apparent work of adhesion as a function of average roughness at high magnification (40x)

Further investigation revealed that the roughness metrics measured in $1.1 \mu\text{m}$ resolution images had better correlation with adhesion results. Correlations of surface roughness on the $1.1 \mu\text{m}$ scale (taken from images at 10x) to associated adhesion values showed very good relation and extended

results gathered in previous experiments where aluminum molds were machined but not polished. Figures 8 and 9 show apparent work of adhesion for the samples cast from polished steel, unpolished steel and unpolished aluminum molds. The difference in agreement of data at different scales suggests that the features affecting adhesion are larger than those which were altered via a laser polishing in this experiment.

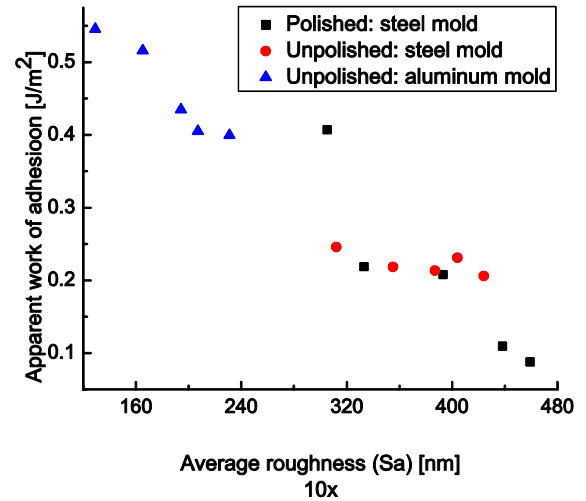


Figure 8: Apparent work of adhesion as a function of average roughness at low magnification (10x)

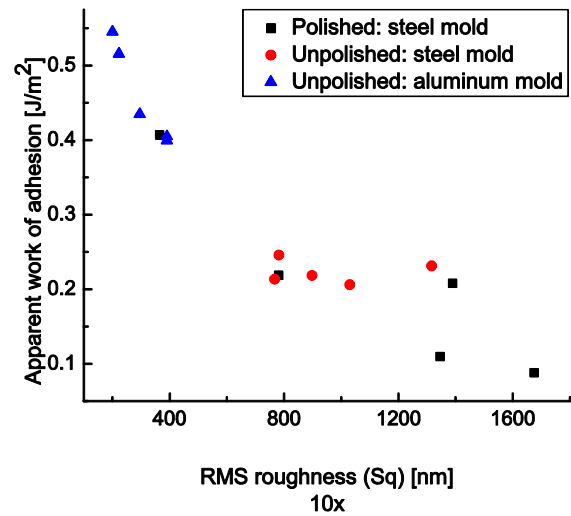


Figure 9: Apparent work of adhesion as a function of RMS roughness at low magnification (10x)

The change in roughness at the lower magnification (10x) due to laser polishing is shown in Figure 10. It is apparent that the laser polishing at the chosen parameters was not as effective at smoothing features at the larger scale. Each of these data points represents the mean of four measurements of average surface roughness (taken at different locations) for each sample.

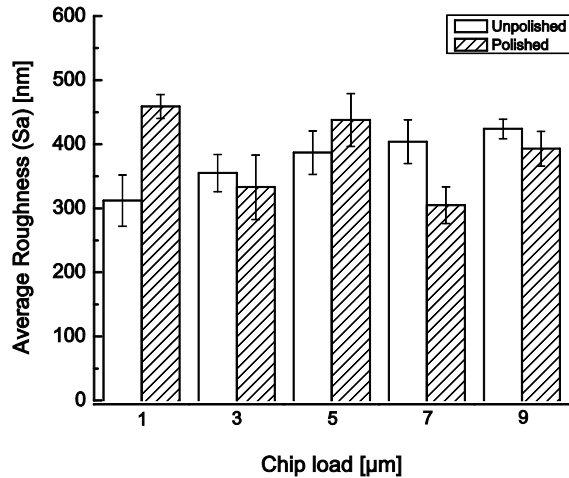


Figure 10: Apparent work of adhesion as a function of RMS gradient at low magnification (S_{dq})

The difference in the surface metrics at each magnification is due to the change in physical area represented by the pixels for the respective magnifications. In the higher magnification each pixel represents a smaller physical area therefore more detail can be seen although there is less physical area captured in the image. The lower magnification allows the capture of more physical area, therefore more features are sampled, the downside of this being smaller features are not captured due to the sampling frequency being too low. The images taken at 10x were able to capture more of the low frequency features that play a stronger role in adhesion although the lower spatial sampling frequency was not able to capture the variation as well as seen in the larger error bars on the 10x data points. This resulted in the analysis of surface roughness that described the change in adhesion not capturing the enhancement due to polishing.

Analysis of spatial frequency content of the samples was performed at both the 10x magnification and the 40x magnification. Overlaid plots of polished and unpolished samples are shown in Figures 11 and 12; where the representative case of the 1 μm sample is examined. It can be seen that higher frequency content within the samples was largely diminished in both cases, although lower frequency content was not significantly affected.

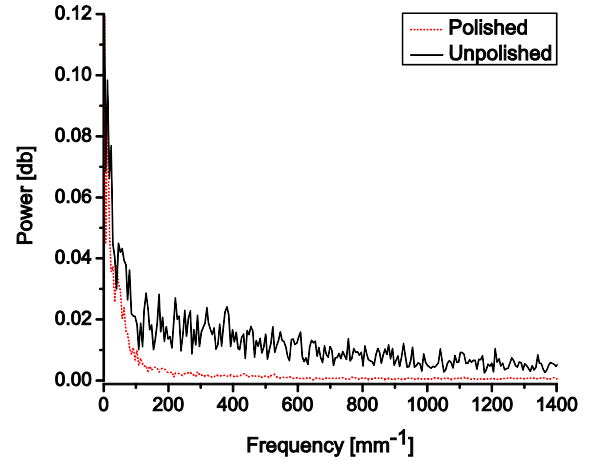


Figure 11: Spatial frequency content for 1 μm chip load sample taken at 40x magnification

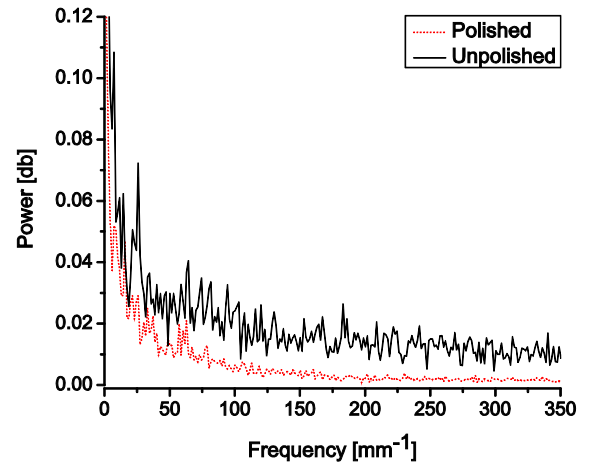


Figure 12: Spatial frequency content for 1 μm chip load sample taken at 10x magnification

CONCLUSION

Laser polishing compliments microscale manufacturing by providing the ability to polish complex geometries and selectively polish spatial frequency content of a surface. In the present work, it has been shown that laser polishing is capable of making significant improvements to the roughness of machined surfaces, however, the parameters must be chosen to reduce the amplitude of the features relevant to the function of the part. As a result of the magnification (40x) selected in choosing laser polishing parameters, a meaningful improvement in adhesion was not realized due to laser polishing. Subsequent tests suggest that improvement in adhesion could be achieved if laser polishing parameters were chosen to reduce the roughness metrics calculated from data collected at a smaller magnification (10x).

ACKNOWLEDGMENTS

This research was partially supported by, a 3M gift, NSF

grant CMMI-0900044, NSF supported shared characterization facilities, and the College of Engineering at the University of Wisconsin-Madison.

NOMENCLATURE

γ - Work of Adhesion (J/m²)

R- Radius of Curvature (m)

P- Imparted load (N)

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