Studies on Forming Anti-Icing Railroad Traffic Signal Lens by Using Femtosecond Laser Surface Processing Directly (FLSP) or by Stamping Tungsten Carbide FLSP Surfaces into Railroad Lens Materials

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16. Abstract
During winter weather conditions snow and ice can accumulate on railroad traffic signal lenses especially when high efficiency light emitting diodes are used as the light source making them hazardous as signals for the engineers. Railroad signal lens polycarbonate (PC) was functionalized to be anti-wetting and anti-icing using femtosecond laser surface processing (FLSP) directly as well as by imprinting PC using a FLSP tungsten carbide (WC) die to produce micron and nanoscale roughness on the surface that is characteristic of anti-wetting surfaces. The micron and nanoscale surface structure on FLSP PC was controlled by adjusting the fluence per laser pulse and number of pulses impinging upon the sample. During stamping applied pressure, temperature, and stamp morphology influenced the imprinted surface structure. Through contact angle measurements, condensation freezing experiments, and placing samples in outdoor icing conditions we demonstrate that both directly processing PC using FLSP and imprinting an FLSP WC surface onto PC enhances the anti-wetting and anti-icing properties. Laboratory scales experiments have shown that the quality of the lens is not affected by the FLSP processing. The next step is to proceed to actual railroad test bed experiments.

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Abstract

During winter weather conditions snow and ice can accumulate on railroad traffic signal lenses especially when high efficiency light emitting diodes are used as the light source making them hazardous as signals for the engineers. Railroad signal lens polycarbonate (PC) was functionalized to be anti-wetting and anti-icing using femtosecond laser surface processing (FLSP) directly as well as by imprinting PC using a FLSP tungsten carbide (WC) die to produce micron and nanoscale roughness on the surface that is characteristic of anti-wetting surfaces. The micron and nanoscale surface structure on FLSP PC was controlled by adjusting the fluence per laser pulse and number of pulses impinging upon the sample. During stamping applied pressure, temperature, and stamp morphology influenced the imprinted surface structure. Through contact angle measurements, condensation freezing experiments, and placing samples in outdoor icing conditions we demonstrate that both directly processing PC using FLSP and imprinting an FLSP WC surface onto PC enhances the anti-wetting and anti-icing properties. Laboratory scales experiments have shown that the quality of the lens is not affected by the FLSP processing. The next step is to proceed to actual railroad test bed experiments.

Chapter 1 Objective

This University Transportation Center for Railway Safety project studied new techniques to functionalize railroad signal lens surfaces (shown in Figure 1) to prevent snow and ice formation on the lens during weather conditions that could lead to ice and snow covering the lens. This research stems from the railroad's use of higher energy efficient light sources inside the railroad signal, which produces less heat and therefore more ice buildup. The objective of this research project was to modify the wetting and icing properties of the polycarbonate (PC) used on railroad signal lenses. In this study we used three basic methods to alter the wetting properties of the lens: (1) direct writing using a femtosecond laser to produce self-organized micron and nanoscale anti-wetting surfaces, (2) imprinting PC using a tungsten carbide (WC) die (functionalized using femtosecond laser surface processing (FLSP)) as a function of specimen temperature and applied pressures during the stamping process, and (3) casting a transparent film using an FLSP surface as the mold. FLSP processing of PC was performed by tuning the laser processing parameters (fluence and pulse count) to produce micro and nanoscale surface roughness that is typical of anti-wetting/icing surfaces found in nature and from previous anti-icing work conducted for Boeing on Al 7075 O clad aircraft aluminum [1]. The imprinting work was divided into: (1) the morphology variation of the imprinted surface as a function of imprint conditions (temperature, pressure, etc.), (2) the wetting properties that result from different imprinted surface morphologies, and (3) the condensation and icing properties by the different imprint surface morphologies.

Figure 1.1 Typical red railway signal lens made of PC used as blank material in this research project (pieces were cut from this lens for specimens)

Chapter 2 Experimental Apparatus and Equipment Used

2.1 Microscopy

Microscopy facilities, including both a scanning electron microscopy (SEM) and laser scanning confocal microscopy (LSCM), were used to analyze the surfaces created using direct FLSP or by imprinting. The SEM used was a FEI Helios NanoLab 660 (pictured on the left in Figure 2). The LSCM used was a Keyence VK-X200K (pictured on the right in Figure 2).

2.2 Femtosecond Laser Surface Processing (FLSP)

FLSP is a one-step fabrication technique that can be used to induce morphological and chemical changes on a metal or dielectric surface [2, 3]. During FLSP, a high peak power femtosecond laser beam (~ 35 fs) is scanned in a raster pattern across a surface melting and ablating material, which results in micron and nanoscale self-organized surface features. A typical FLSP setup used in this research project is shown in Figure 3. By varying the laser average power, raster speed, raster pitch, and spot size, the self-organized features on the sample can be controlled. For this project, in the stamping part of the research, WC was selected because of its hardness, which makes it resistant to wear during stamping and is often used for machine tools [4]. The hardness of WC also makes it difficult to machine with general machine shop capabilities. However, during this project, we demonstrated that a femtosecond laser is able to create controlled micro/nano-scale surface roughness even on this very hard material. Using FLSP, we are able to create roughness on the scale that is characteristic of anti-wetting and anti-icing surfaces widely discussed in the literature [5-7].

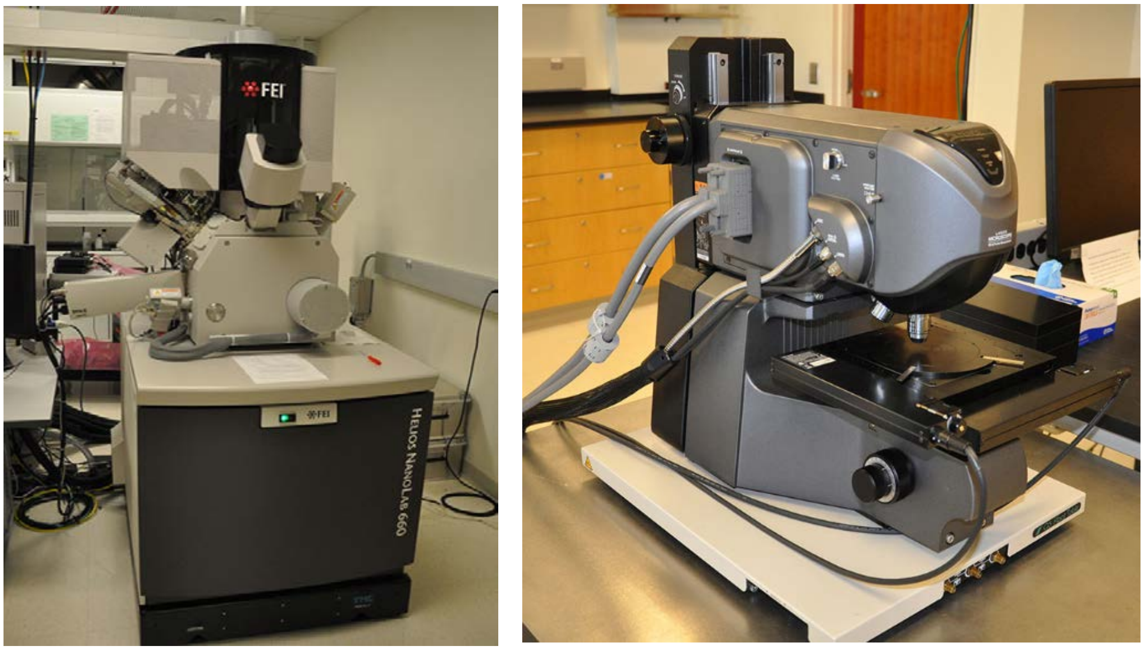

Figure 2.1 Microscope instruments used to observe surface structures. Left: FEI Helios NanoLab 660, right: Keyence laser scanning confocal microscope VK-X200K.

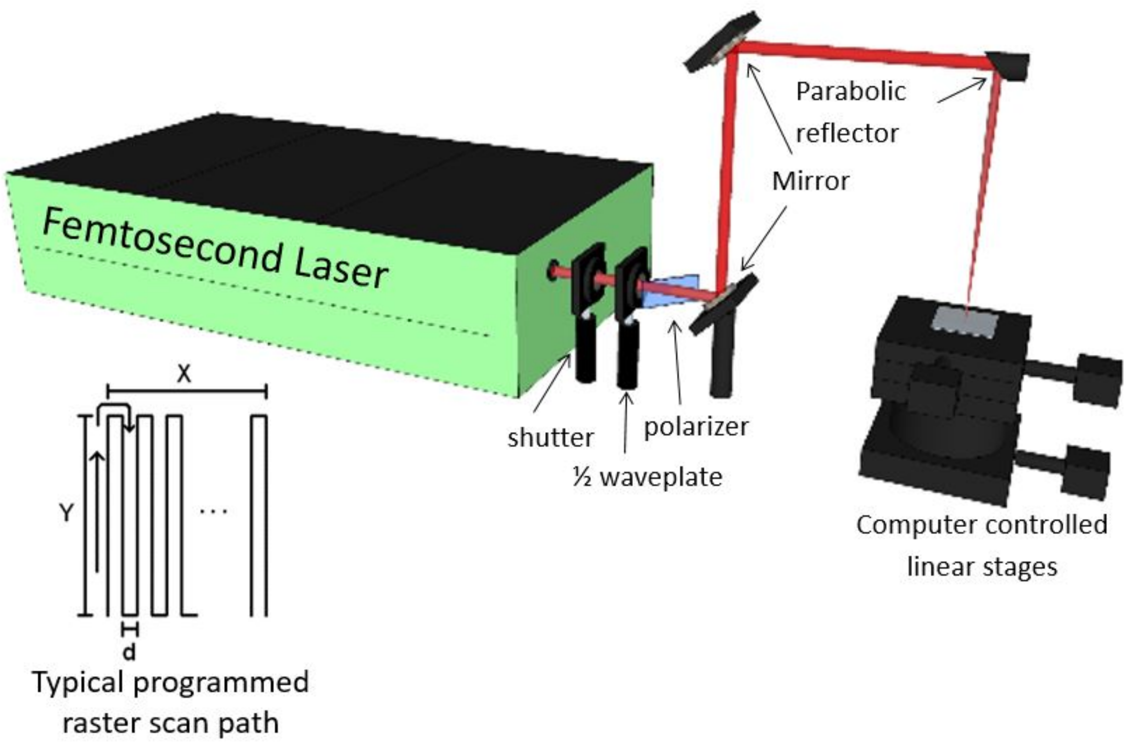

Figure 2.2 Diagram of a typical FLSP setup and raster pattern used to process large areas

WC imprinting dies were fabricated specifically to study what features on the die resulted in the best wetting properties on the polycarbonate (railroad lens specimen). The stamped surface was structured to have surface features that typically result in superhydrophobic surfaces on low surface energy materials (PC is a low surface energy material) [8].

2.3 Imprinting

Imprinting was considered as an alternative to direct FLSP as it might be more efficient both economically and temporally, especially for the large number of lenses that might be needed in larger scale applications. Imprinting is a simple process of applying pressure on a surface such that the structure on one die surface is transferred to the other softer material. WC was used as the die because of its extreme hardness (~9 on the Mohs hardness scale) while PC is a relatively soft material (~3 on the Mohs hardness scale). In this research project, we found that it was better to heat the PC to soften the surface while imprinting.

The device used in this stamping work is a CARVER® model 2699 with a load range of 0-15 ton (shown in Figure 4). During stamping, one custom-made steel holder was used to keep the force even across the die surface as the imprinting was carried out on the PC specimen.

Figure 2.3 CARVER® model 2699 compression machine for stamping, right bottom shows the stamp holder

2.4 Temperature and Pressure Effects on Stamping

In this research project, we studied two variables that affect stamping quality: temperature and pressure. To find the optimal temperature and pressure we performed the following experiments.

2.4.1 Temperature's Influence on the Imprint Morphology

Experiments were performed to understand the influence of temperature on the imprinting morphology. PC has a glass transition temperature of about 147 °C and a melting point of 200 ℃. For the work reported in this final report we used temperatures between 50~200 °C [8]. Imprinting was performed at 50, 100, 150, and 200 °C with four identical FLSP WC dies (shown in Figure 5) on four PC samples. The same experiment was repeated at increments of 5 °C between 100 °C-

150 °C and 120 °C-140 °C. Results of these studies indicate that optimal working temperature of 120 °C was the best to transfer the FLSP die surface features to the softer PC lens material.

2.4.2 Press Displacement's Influence on the Imprint Morphology

The other key variable to control during stamping is the pressure applied. Fixing the temperature at 120 °C, the compression force was then studied by changing the displacement set on the Carver machine. Four identical FLSP tungsten carbide stamps were compressed onto four polycarbonate samples ($1 \times 1\ cm^2$). The temperature was set to be 120 ℃ on the furnace while the compression distance was varied from 0.1 mm, 0.2 mm, 0.3 mm and 0.4 mm, separately.

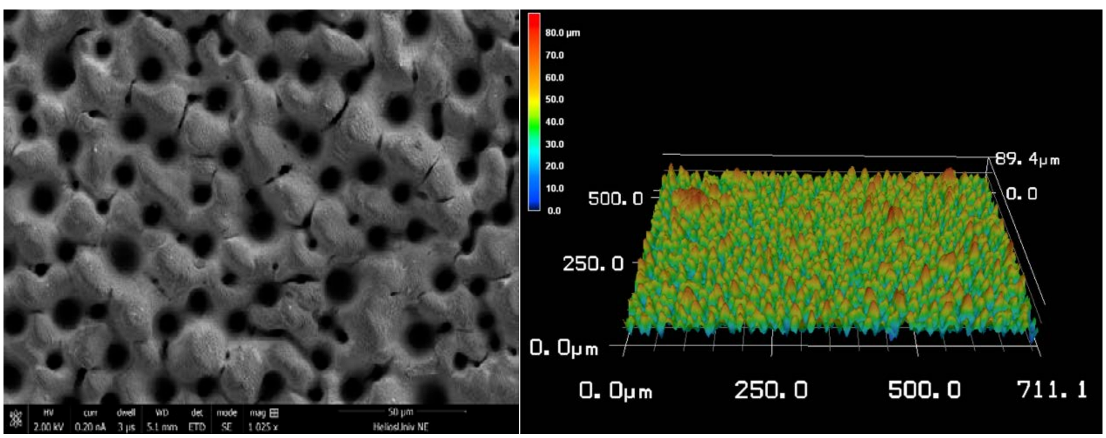

Figure 2.4 WC FLSP stamp used in temperature studies. Left: SEM image, right: LSCM image.

2.5 Effect of the Die Surface Morphology on the Imprinted Polycarbonate Surface

After determining the best conditions (temperature and pressure) to transfer the micro/nanostructure from the die onto a polycarbonate stamp, we investigated the influence that structure type had on the anti-wetting and anti-icing properties of PC. This allowed us to vary specific surface features and evaluate the properties of these surface features on wetting properties of the stamped surface. In other words, multiple dies were made with surface morphology being the “knob”.

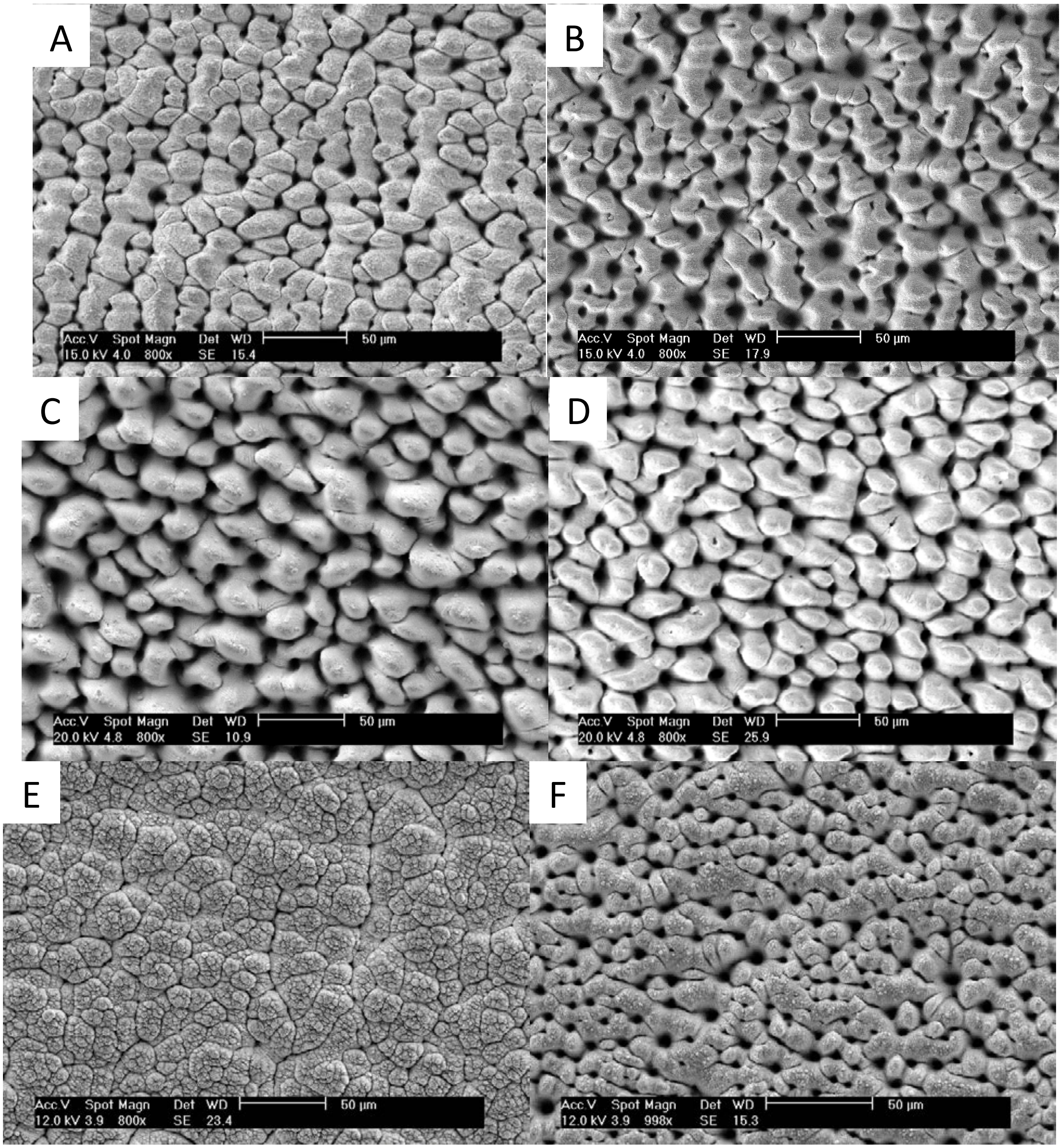

Figure 2.5 SEM images of samples used to examine the influence of die morphology on stamp properties.

Table 2.1 FLSP Parameters Used to Produce the Samples Discussed in this Report

SAMPLE	PEAK FLUENCE (J/CM^2)	PULSE COUNT
A	70.7	196
B	1.9	1211
C	53.1	295
D	53.1	196
E	0.4	844
F	2.8	1021

2.5.1 Roughness

A review of the literature clearly provides a guide that roughness has a strong effect on wetting properties. With this in mind, stamps were produced with different roughness while maintaining very similar structures. SEM images of the samples created to test roughness variability are shown in Figure 6. Samples A and B have a RMS surface roughness of 4.03 and 4.82 microns, respectively, as determined using the LSCM.

2.5.2 Mound Concentration Influence on the Imprint Morphology

Samples C and D have almost identical structure heights but a 2:1 peak-to-peak density ratio. These samples allow us to test how peak-to-peak density effects wetting properties of a stamped PC surface. The only difference between the processing parameters used to make these two samples was a change in shot number. SEM images of the samples created to test the influence of peak-to-peak distance are shown in Figure 6. Peak density was determined using the LSCM.

2.5.3 Different Mound Structure Morphology

Samples E and F, shown in Figure 6, have different structure types. Sample E has high roughness and low structure height. Sample F has lower roughness and higher structure height. Both influence and pulse count were varied between these two samples.

2.6 Casting Silicone Elastomer on FLSP Surface Molds

In this project, a third type of anti-icing surface was investigated, referred to as casting a liquid to form a thick transparent film (see Figure 7). The idea behind this was that the thick film could be placed over existing railroad lenses to make them anti-icing. Casting was explored here to duplicate the surface structures produce on a FLSP tungsten carbide surface by producing a film that can be peeled off the die surface. Dow Corning Sylgard 184 silicone elastomer was used as the liquid for casting experiments. This silicone elastomer is a polydimethylsiloxane (PDMS) made by mixing two components together to form a liquid that is poured onto the mold surface.

Figure 2.6 Part A and B of Dow Corning Sylgard 184 silicone elastomer (left) and the FLSP WC mold (right)

2.7 Anti-Icing Studies

Samples produced by direct FLSP and by stamping were studied for condensation and anti-icing properties. Condensation experiments were conducted by using the cooling system shown in Figure 8 (the temperature was set just below the freezing point of water). The sample was placed on a holder that can be moved under the optical microscope for observation of the actual freezing processes in real time.

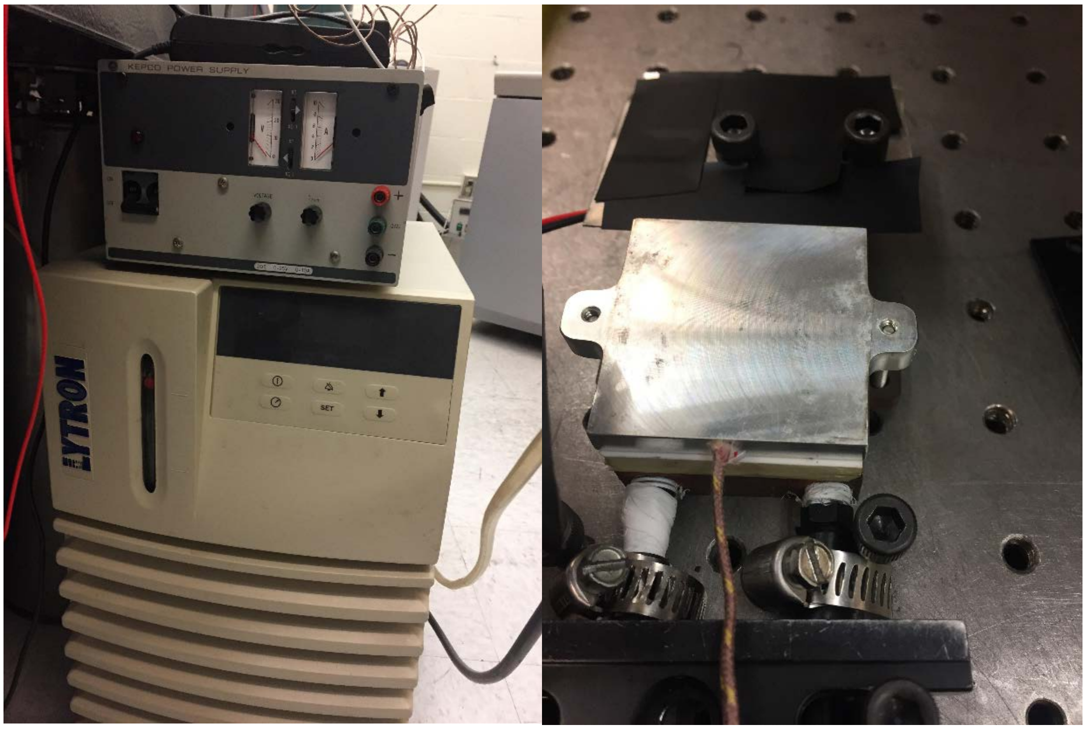

Figure 2.7 Litron thermal system for condensation freezing. Left: control system, right: sample holder

Chapter 3 Results and Discussion

3.1 Femtosecond Laser Processed Polycarbonate (PC)

FLSP was applied directly to a PC surface to produce superhydrophobic properties. A superhydrophobic surface with a contact angle of ~160° was made with a fluence of 0.6 J/cm^2 and pulse count of 930. An image and LSCM scan of the processed FLSP PC sample is shown in Figure 9. The PC samples became superhydrophobic immediately after processing. Applying FLSP directly to PC is a promising option to functionalize a railroad signal lens to be superhydrophobic because it is a one-step process and processing PC using FLSP is an order of magnitude faster than processing metals. As observed in Figure 9, the processed PC sample became a diffuse scatterer (bottom left), but appeared red when a LED cell-phone light was used to illuminate the lens sample (bottom right).

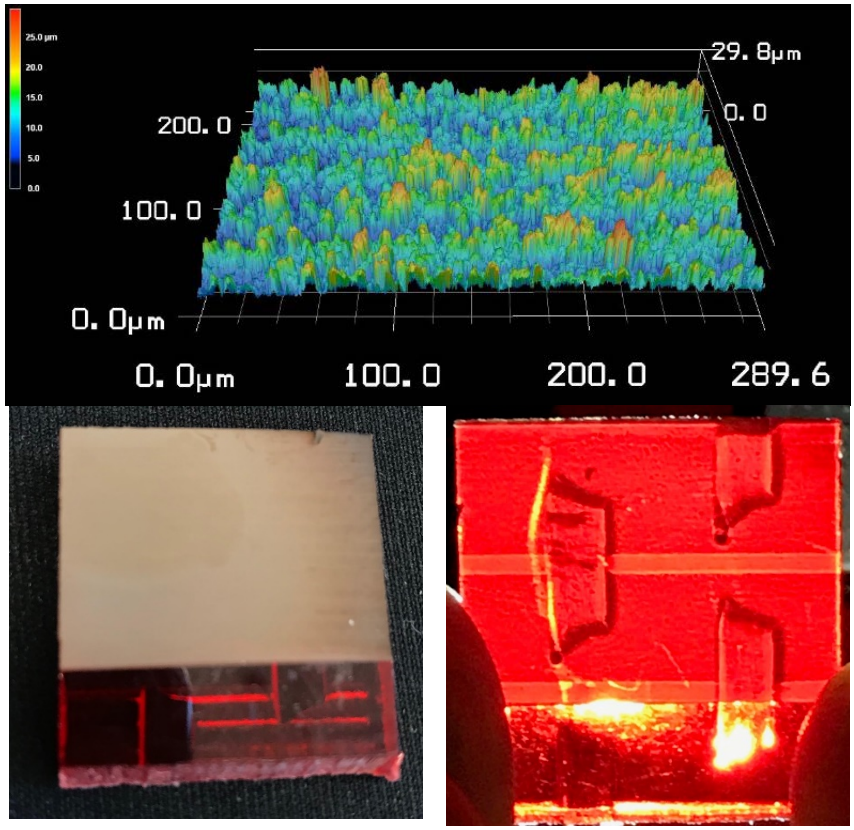

Figure 3.1 LSCM scan of the processed PC (top), an image of a partially processed PC sample to show the contrast between the two regions (bottom left), and the FLSP PC sample with a LED light illuminating the back side (bottom right)

3.2 Imprinted Surface Morphology

The temperature of the sample is one of the three key factors that affect the quality of the resulting stamped material surface (the others being pressure and die morphology). SEM images of four typical stamped surfaces are shown in Figure 10. The surface morphology changes greatly for temperatures in the range of 100~150℃, varying from pit-like structures to mound-like structures. Using sample temperatures below 50 ℃, only the taller mounds penetrate into the PC surface so the morphology includes pit-like structures. As surface temperature during stamping is increased, the PC becomes softer and more of the FLSP mounds can be imprinted into the PC. An additional detail not shown in Figure 10 is that 150℃ is right at the glass transition temperature for

PC, resulting in PC becoming rubber-like and the springing back into its original shape. Around 200°C the PC softens and therefore takes the shape of the WC die. After stamping, the PC cools and solidifies with the morphology shown in the bottom right image of Figure 10.

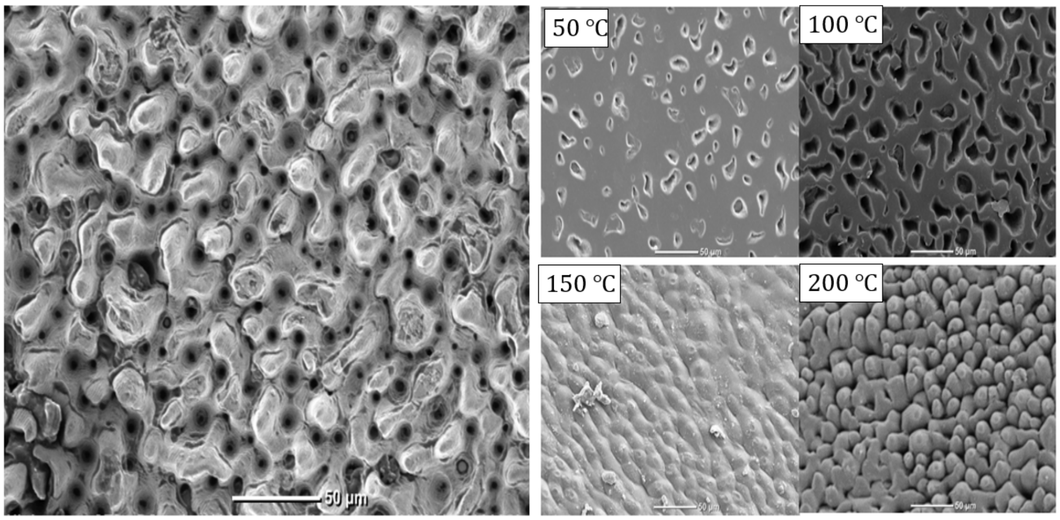

Figure 3.2 SEM images of a WC die (left) and various PC stamps (right) created using different PC sample temperatures during stamping

Experiments were performed with smaller increments in temperature. At 120 °C structures were imprinted onto PC with a peak to valley height of 44 μm and a contact angle of 115°. SEM images of the WC die and LSCM images of the PC stamps are shown in Figure 11.

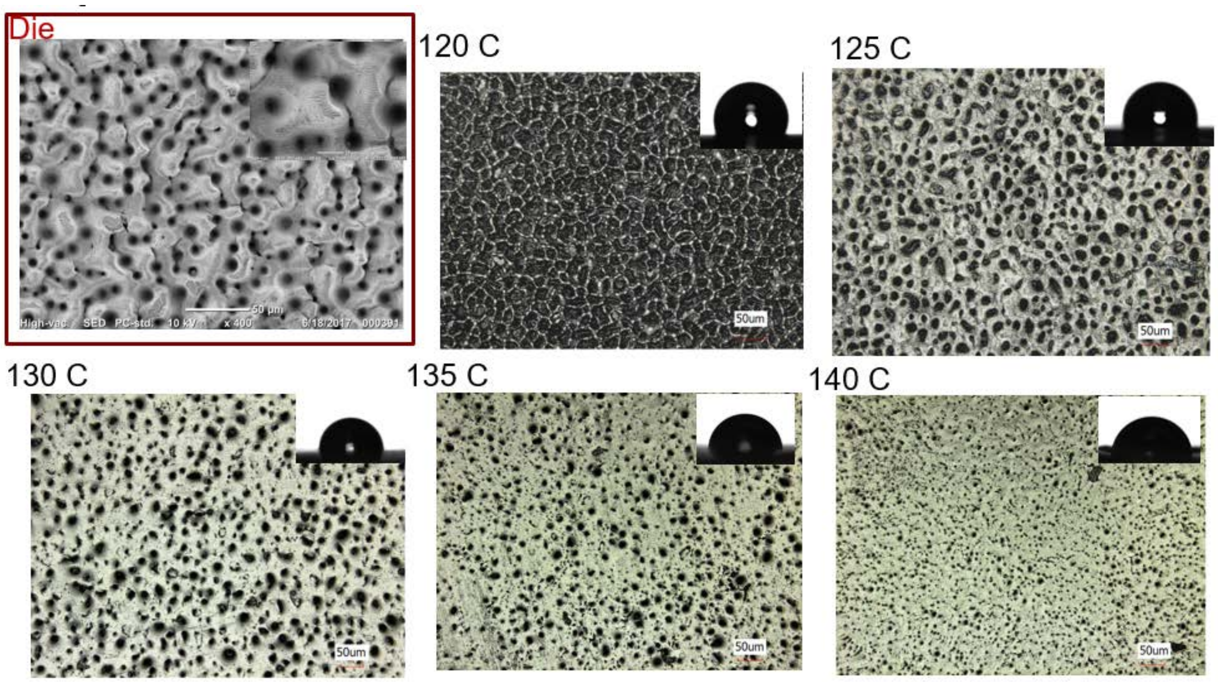

Figure 3.3 SEM images of a WC die (left) and LSCM images of PC stamps (right) stamped at various temperatures to optimize the resulting wetting properties shown by the droplet contact angle in the upper right corner of each image

Peak-to-valley distance (“R_z“) is used to quantify the imprint quality. Shown in Figure 12 is a graph of how R_z changes. R_z reaches its peak value of 6 ± 10 μm at 120°C. Therefore, based on information presented in Figure 12, 120°C is the optimal stamping temperature for creating structures on PC. Contact angle data for the various PC stamps created at various temperatures is presented in Figure 13. As expected, the most hydrophobic PC stamp corresponds with the highest peak-to-valley distance created at 120°C. It should be noted that the best PC stamp (36±10 microns) is still less than half as rough as the WC die (83±6 microns).

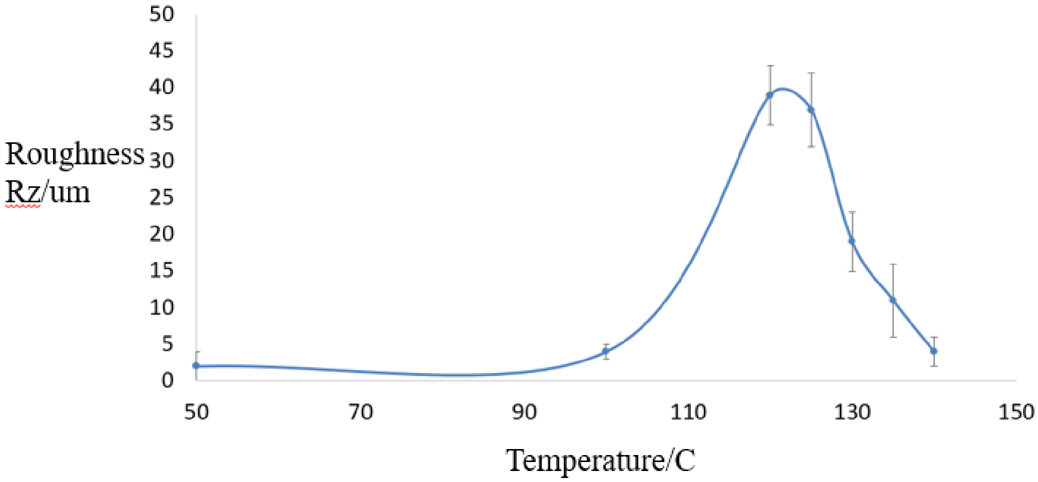

Figure 3.4 Graph of the peak to valley roughness of stamps created using various temperatures

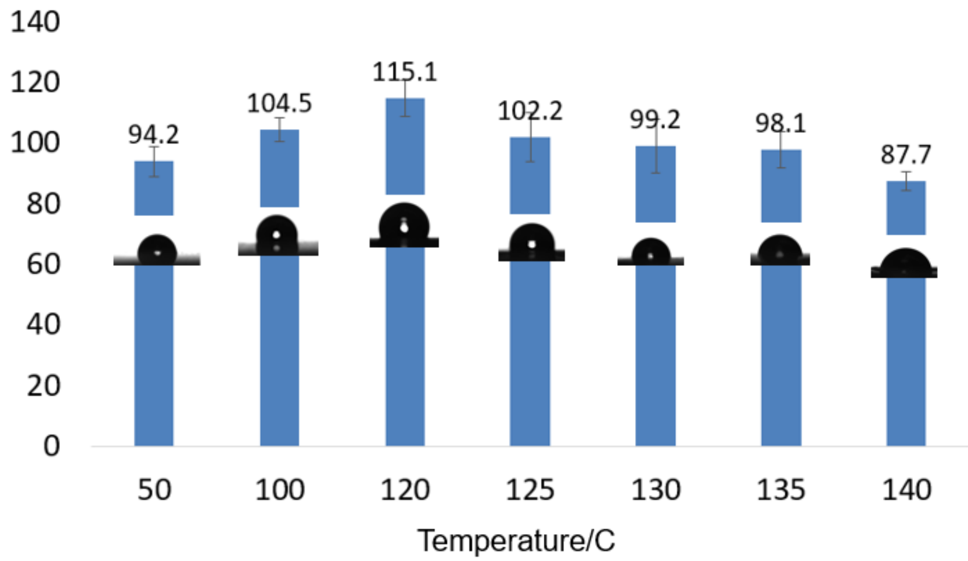

Figure 3.5 Graph of the contact angle measured on PC stamps created using various temperatures

3.3 Influence of Compression Displacement on Stamp Quality

The next variable optimized was the displacement of the stamping machine (e.g. the force).

The peak-to-valley distance and morphology of the PC surface is affected by the displacement of the press. In these experiments, displacements of 0.1, 0.2, 0.3, and 0.4 mm were used. A displacement of 0.1 mm resulted in the best transfer of the FLSP structures to the PC stamp. For the 0.2, 0.3, and 0.4 mm samples the regions between the valleys are relatively flat (full FLSP structure was not transferred) compared to the 0.1 mm sample surface. The surfaces produced for all of the displacements used are shown in Figure 14 along with the corresponding water droplet interacting with the surface. The mound-like structures play an important role in improving the surface wetting properties, with a maximum contact angle of 130° resulting by applying a displacement of 0.1 mm.

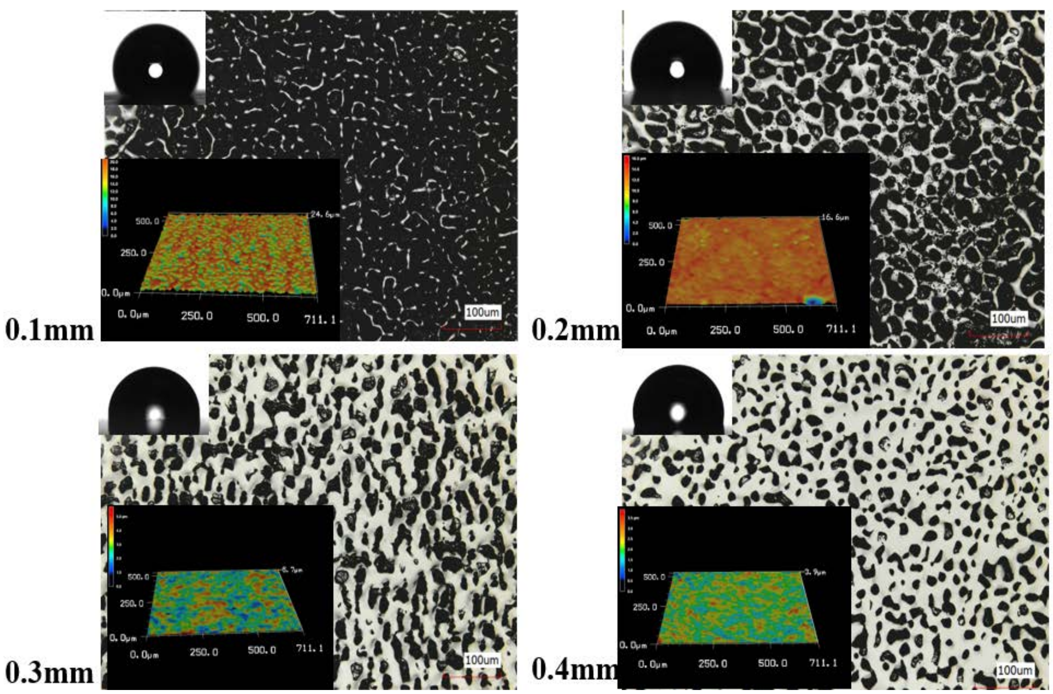

Figure 3.6 LSCM images of PC samples created using 0.1, 0.2, 0.3, and 0.4 mm displacements

3.4 Die Morphology Influence

FLSP WC die morphology is the strongest factor in determining the wetting properties of the imprinted surface. The imprinted surface always contains the negative surface morphology of the FLSP dies. This section shows how different surface morphology characteristics influence the surface morphology and the wetting properties with the stamping environment fixed.

3.4.1 Roughness $\boldsymbol{R_z}$

The two FLSP stamps used in this part of the work have similar types of surface structures but differ by 10 µm in their average peak-to-valley height. The morphology of two imprinted PC surfaces stamped from FLSP dies is shown in Figure 15. The left image in Figure 15 has a peak to valley height of 12 ±5 µm, and the image on the right has an average peak to valley height of 20±7 µm. The difference in average peak-to-valley height of the stamped PC samples is consistent with the dies used to produce them.

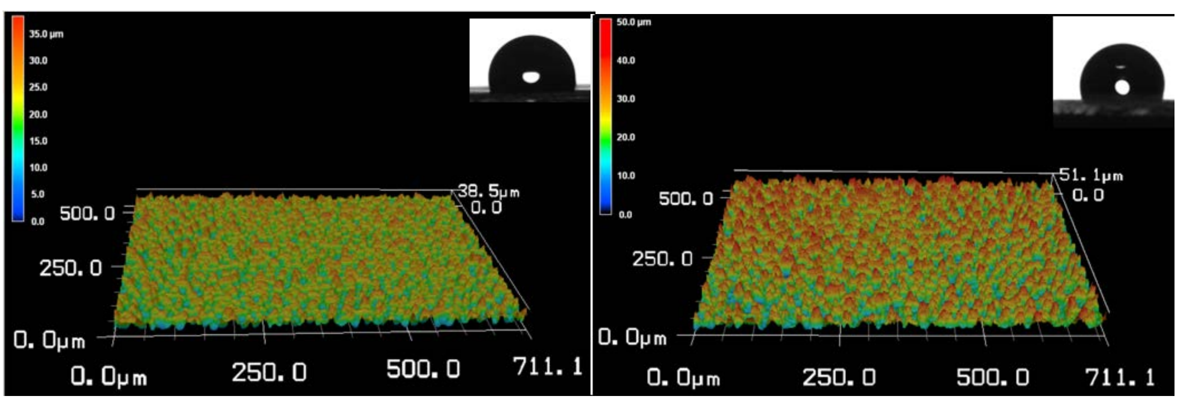

Figure 3.7 LSCM scans and droplets on the PC stamps with different peak-to-valley heights

3.4.2 Peak-to-Peak Distance

Using the WC dies made with identical average peak-to-valley heights, but a 2:1 ratio in the peak-to-peak distance, PC stamps were made. The two FLSP stamps used in this stamp process have identical peak-to-valley heights of $R_z = 20\mu m$, but the peak-to-peak distance ratio between the surfaces is 2:1 (Figure 16 bottom row vs. top row). Images of droplets on the surface and LSCM are included in Figure 16. The sample on the top of Figure 16 has the higher contact angle and peak density.

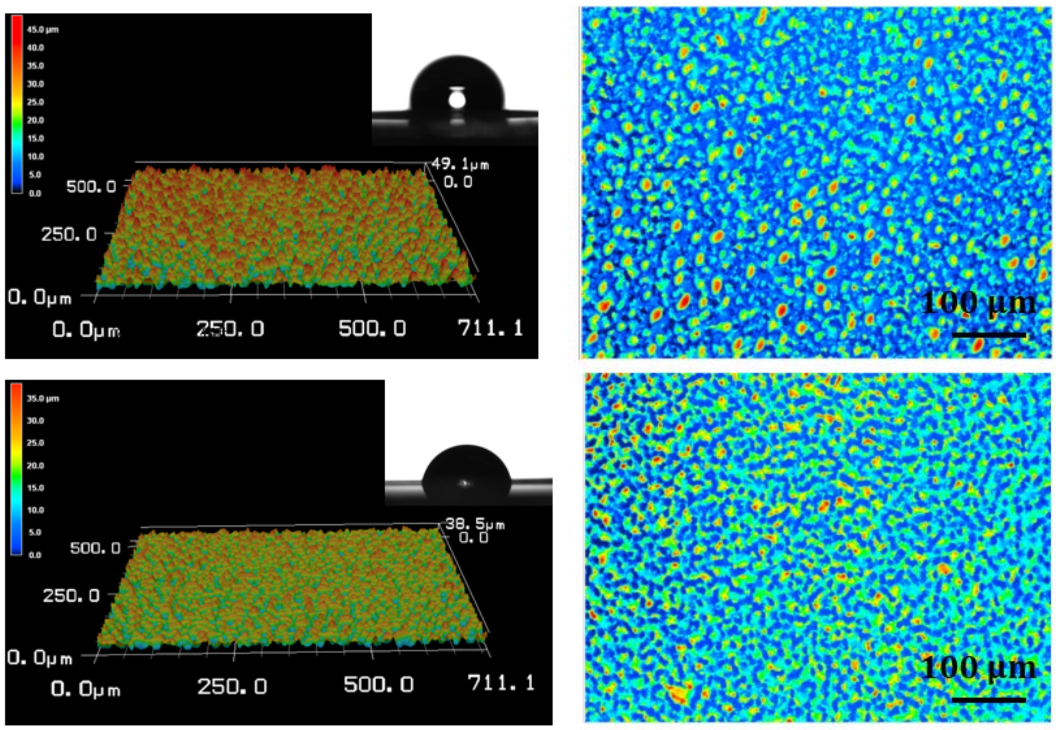

Figure 3.8 LSCM and droplet images of PC stamps created with varied peak density (peaks/area)

3.4.3 Different Surface Structure Types

Two different types of structures were tested: mounds with and mounds without pits. As can be seen in the contact angle image in the corners of the LSCM images in Figure 17, the surfaces have similarly poor anti-wetting properties.

3.5 Duplicating a Dies' Surface Pattern by Using Casting

Patterned PDMS by casting from the FLSP tungsten carbide surface had similar morphology with the die (shown in Figure 18). The cast PDMS surface had a roughness of $R_z = 61 \pm 5\ \mu m$, which is only a $20\ \mu m$ difference compared to the FLSP stamp ($R_z = 83 \pm 6\ \mu m$). Therefore, casting provides a good way of duplicating FLSP surface features. Furthermore, this patterned PDMS surface had a contact angle $> 150\,°$ as compared to flat PDMS (~135°). The duplicated surface features improved the PDMS from hydrophobic to superhydrophobic without any post-casting modifications.

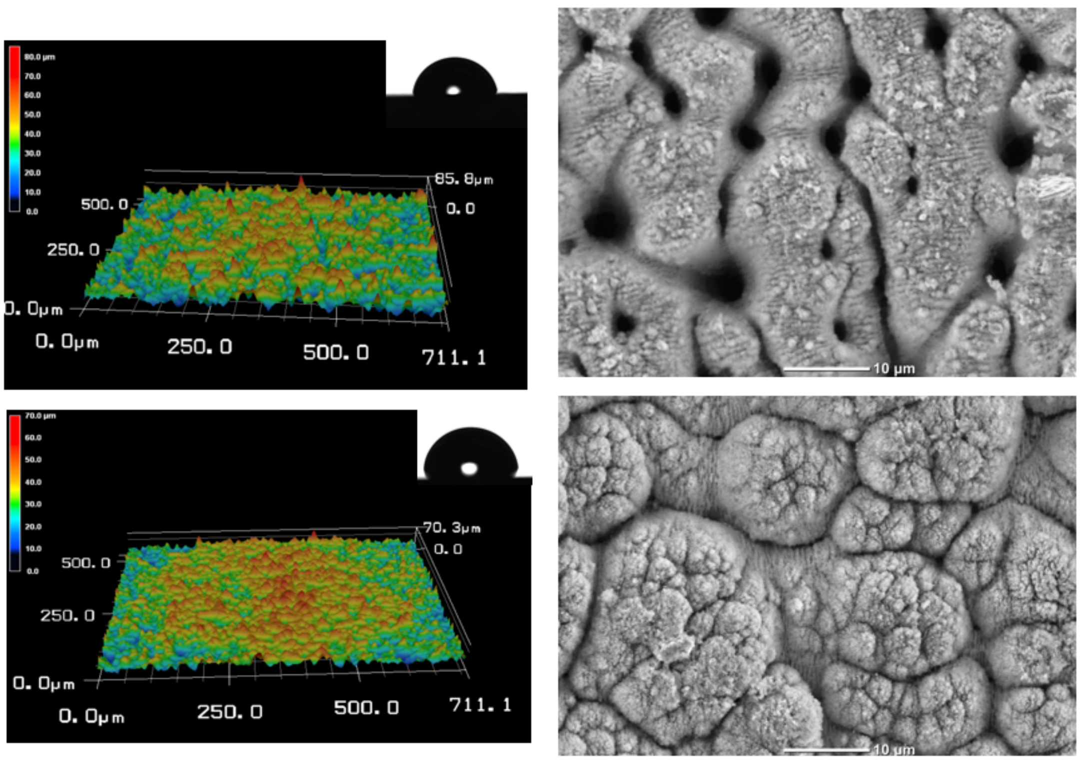

Figure 3.9 LSCM, contact angle, and SEM images of two PC stamps with different types of surface structuring

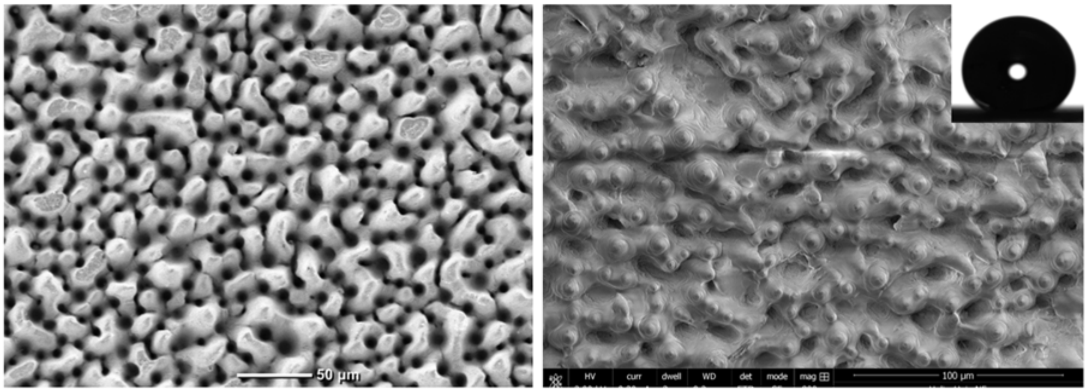

Figure 3.10 SEM images of FLSP tungsten carbide surface and the casting PDMS surface; left: FLSP tungsten carbide surface; right: PDMS surface

3.6 Die Degradation During Stamping

During the stamping process, the die endures large forces, so the mechanical integrity of the micro and nanostructure can be damaged despite the hardness of WC die surface features. Surface scans were taken and compared before and after stamping (shown Figure 19). The average peak-to-valley height decreased 3 μm after stamping but the general morphology of the die remained the same.

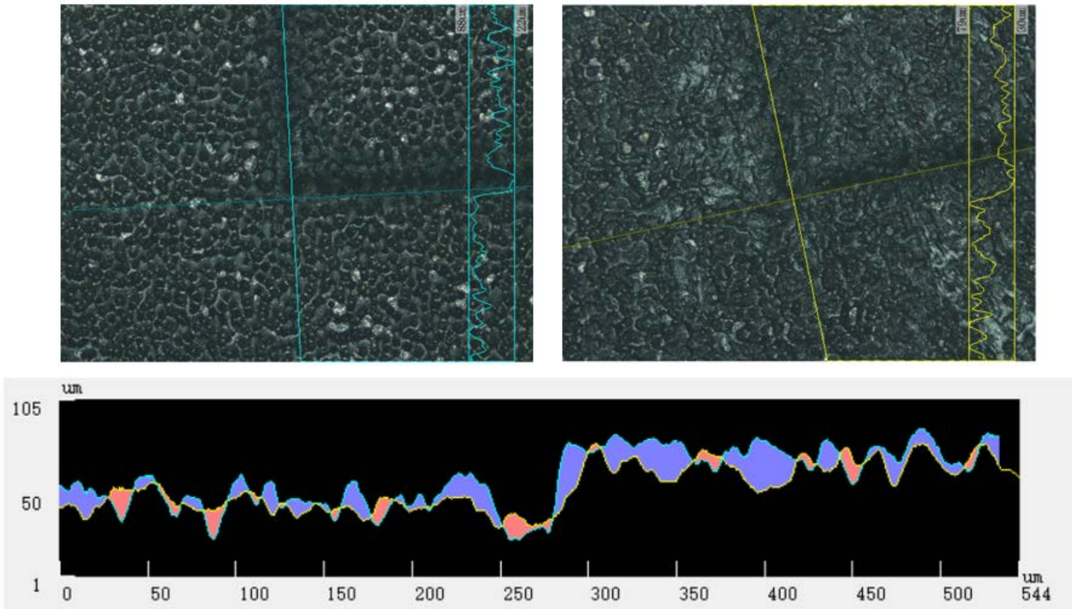

Figure 3.11 Two line scans through the same portion of a WC die before (left) and after (right) stamping

3.7 Condensation Studies

Condensation is the first step before ice forms on the surface. A comparison between unprocessed and imprinted PC surface is shown in Figure 20. The condensation is formed because of the temperature difference between sample surface and surrounding air. It can be clearly seen that round water drops formed on the unprocessed PC surface, while this condensation phenomenon is not observed on the imprinted PC surface (the structure observed is the surface roughness of the imprinted PC surface).

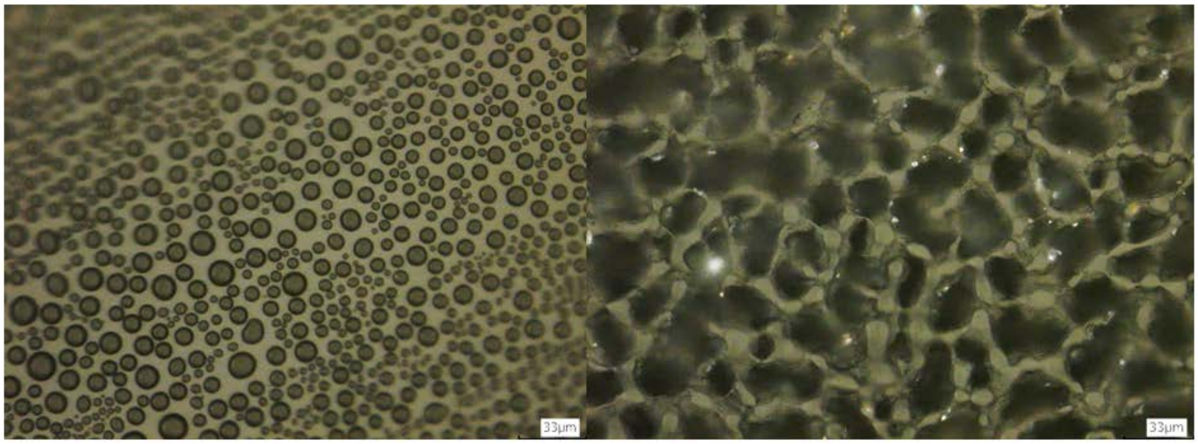

Figure 3.12 Condensation comparison between unprocessed PC on the left and imprinted PC surface on the right

3.8 Icing Studies During an Actual Ice Storm

When outdoor icing conditions arose on the University of Nebraska-Lincoln campus, we decided to take advantage of the opportunity. A rain-ice-snow storm was forecast for January 11th, 2018. A simple apparatus, pictured in Figure 21, was made to hold the PC samples off the ground at 45 degrees facing in the direction of the wind. The samples were exposed to icing conditions for three hours on the north side of the Walter Scott Engineering Center. The results of the outdoor icing experiment (pictured in Figure 22) showed the FLSP PC had the least accumulation of ice on the surfaces being studied. Again, on January 22, 2018, a storm was forecast to produce freezing rain and snow. Three PC samples were placed outside to be exposed to the weather. The before and after images of the samples placed outside are shown in Figure 23. Droplets and ice accumulation on the FLSP surface were much smaller and less dense than on the unprocessed sample. Once snow accumulated on the FLSP surfaces, it was easy to blow the snow away by lightly blowing on the surface.

Figure 3.13 Image of apparatus used to place PC samples in outdoor icing conditions

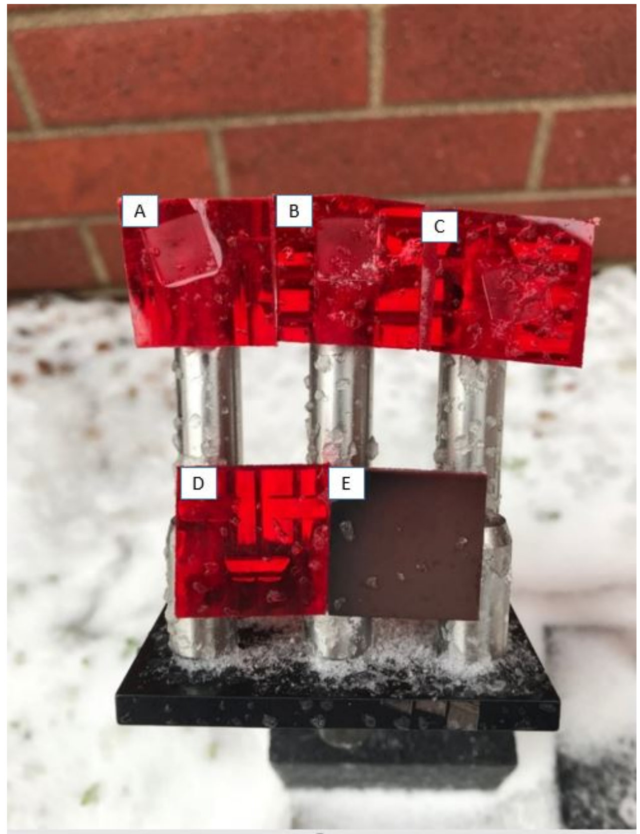

Figure 3.14 Outdoor icing test during the January 11, 2018 ice storm. Top row (A-C) are specimens with stamped regions as marked. Specimen D is unprocessed. Specimen E is FLSP.

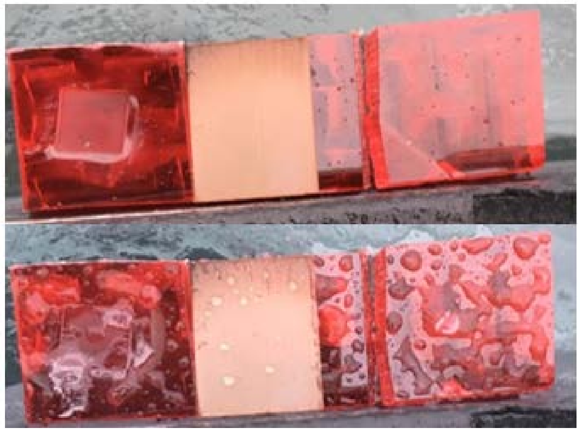

Figure 3.15 Outdoor icing test during the January 22, 2018 ice storm; from left to right: Imprinted PC, FLSP PC, unprocessed PC. Top row is before exposure to icing conditions, and the bottom row is after exposure to icing condition.

Chapter 4 Conclusion

Based on the research performed for the University Transportation Center for Railway Safety, the following conclusions can be made.

1) Femtosecond laser processing of railroad signal lens covers is a feasible way to make the railroad lens cover superhydrophobic. The direct writing process slightly alters the color of the lens, but does not alter the color when illuminated. The FLSP surfaces performed very well in an outdoor real life icing environment.
2) Stamping of FLSP surfaces into a heated railroad lens cover also was demonstrated to be able to affect the anti-icing properties of railroad lenses. Furthermore, the stamping conditions influence the imprint quality and ultimately the wetting properties and icing properties.

    ⅰ. Temperature: 120 ℃ is an ideal temperature for stamping a railroad PC signal lens. Both lower and higher temperatures result in poor imprint quality. For lower temperatures, only the pit-like morphology developed; for higher temperatures, it is easy to damage the PC samples since they stick to the stamps and can be deformed when separated.

    ⅱ. Load: With the stamping temperature set to 120 ℃ (optimum), there is an optimum force to apply for duplicating the surface morphology (due to large elasticity).
3) The structure morphology on the dies also has a large influence on the imprint quality.

    ⅰ. Roughness R_z: higher roughness results in higher imprinted surface roughness, and it can improve the surface hydrophobicity.

ii. Peak-to-peak distance: the imprinted peak-to-peak distance is inverse with the stamp. For the PC materials, the higher peak-to-peak distance, the more hydrophilic it is.

iii. Different surface structure types: different surface structure types result in different surface morphology on the imprinted PC surface but have limited influence on the wetting property.

4) Casting PDMS improved its hydrophobicity from hydrophobic to superhydrophobic without any post-casing modification.
5) No water drops formed on the FLSP imprinted surface during a condensation test.
6) Imprinted, FLSP, and casting have promise for anti-icing railroad lens applications.

In summary, railroad signal lens polycarbonate (PC) became anti-icing for directly writing of FLSP on PC surfaces. Imprinting studies were inconclusive but showed promise for anti-icing applications with additional research. Casting of PDM films showed superhydrophobic properties, but no icing work was carried out on these surfaces. Further research will be needed to determine the most economical method to actually use in a commercial application of the technology developed in this study. What is encouraging is that directly processing PC using FLSP can be applied at high processing rates.

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Appendix A Publications Resulting from this Research

1. Y. Song, A. Tsubaki, C. Zuhlke, et al. Effect of topology and material properties on the imprint quality of the femtosecond-laser-induced surface structures. *J Mater Sci*. vol.53, no. 5, pp. 3836-3845, 2018.

2. Y. Song, A. Tsubaki, C Zuhlke, et al. (2018), Variation of Metal Surface's Wetting Property by Imprinting Femtosecond Laser Surface Processed Micro/nano-structure. Paper presented at *Asscoicaiton for Iron and Steel Tech Coference,* Retrieved May 7, 2018.

3. Y. Song, A. Ediger, R. Bell, C. Zuhlke, A. Tsubaki, J, Shield, D. Alexander. Studies on forming anti-icing railroad traffic signal lens by femtosecond laser surface processing directly (FLSP) or by stamping tungsten carbide FLSP surfaces into railroad lens materials. Poster presentation prepared for UNL visit to Pueblo, CO, August 2018.