High-Quality Femtosecond Laser Surface Micro/Nano-Structuring Assisted by A Thin Frost Layer

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Femtosecond laser ablation has been demonstrated to be a versatile tool to produce micro/nanoscale features with high precision and accuracy. However, the use of high laser fluence to increase the ablation efficiency usually results in unwanted effects, such as redeposition of debris, formation of recast layer, and heat-affected zone in or around the ablation craters. Here this limitation is circumvented by exploiting a thin frost layer with a thickness of tens of microns, which can be directly formed by the condensation of water vapor from the air onto the exposed surface whose temperature is below the freezing point. When the femtosecond laser beam is focused onto the target surface covered with a thin frost layer, only the local frost layer around the laser-irradiated spot melts into water, helping to boost ablation efficiency, suppress the recast layer, and reduce the heat-affected zone, while the remaining frost layer can prevent ablation debris from adhering to the target surface. By this frost-assisted strategy, high-quality surface micro/nano-structures are successfully achieved on both plane and curved surfaces at high laser fluences, and the mechanism behind the formation of high-spatial-frequency (HSF) laser-induced periodic surface structures (LIPSSs) on silicon is discussed.

1. Introduction

Ultrafast laser surface micro/nano-structuring has emerged as an enabling technology with a wide variety of applications involving self-cleaning, anti-reflection, anti-microbe, and anti-friction, due to its high flexibility in tuning optical, mechanical, or chemical properties at surfaces and interfaces of materials.[1] In the meantime, recent rapid advances in ultrafast laser technology have made high-average-power ultrafast lasers increasingly reliable and cost-effective for high-throughput material processing.[2,3] Although ultrafast laser ablation, under the proper control of parameters, has proved its uniqueness to produce micro/nanoscale features with high precision and accuracy,[4] there still remain several inevitable issues as the laser fluence increased, such as redeposition of debris, formation of recast layer and heat-affected zone in or around the ablation regions. The debris and recast layer, which was formed by rapid cooling and re-solidification of high-temperature plasma or molten material generated by laser ablation, usually has strong adhesiveness and is difficult to be removed.[5,6] Meanwhile, located on the surface close to the irradiated zone or in the ablated craters and grooves, the debris and recast layer may block the propagating path of subsequent laser pulses. In this regard, the ablated debris and recast layer not only affect the surface quality and deteriorates the functionality, but also reduce the ablation efficiency.

Many efforts have been made to remove the debris and recast layer in the laser ablation process.[7–12] Conventionally, water-assisted laser ablation has been extensively exploited to remove the debris, in which the debris can be carried away by thermal convection of liquid and bubble-induced liquid motion. Nevertheless, water-assisted laser processing suffers from scattering or deviation of the incident laser by the water surface, cavitation bubbles and suspended ablated material.[10] An ice shield layer recently was introduced to prevent debris redeposition while reducing surface disturbance,[11–15] in which the ice layer was preliminarily formed by freezing a water layer on the sample surface, and locally melted or vaporized in the process of laser ablation. However, the ice layer obtained by freezing water usually has a thickness of more than several hundred microns due

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to the large surface tension of water. For femtosecond laser precise ablation, it is difficult to melt or remove the thick ice layer by tightly focused femtosecond pulses with low pulse energy.

Here, we present a novel approach toward high-quality surface micro/nano-structuring by femtosecond laser ablation assisted by a thin layer of condensed water frost on cold surfaces. By controlling the sample surface temperature and relative humidity of the surrounding atmosphere, a thin frost layer with a thickness of tens of microns could be homogeneously deposited on all surfaces of the sample. When a femtosecond laser was focused on the sample surface covered with the thin frost layer, the frost located in the focusing area would rapidly melt to a thin layer of water, helping to disperse debris and reduce the recast layer in subsequent laser ablation, while the remaining frost layer could prevent ablation debris from adhering to the target surface. The ablation efficiency and surface micromorphology were comparatively studied by femtosecond laser ablation on different materials with and without the assistance of the frost layer. High-quality micro/nanostructures on both plane and curved surfaces of different materials were demonstrated by the frost-assisted strategy, and the mechanism behind the high-quality and high-precision nanostructures was discussed.

2. Experimental Section

The schematic of our experimental setup is illustrated in Figure 1a, which mainly consists of a femtosecond laser source, an objective lens mounted on a Z-axis translation stage, a charge-coupled device camera system for optical microscopy and a thermoelectric cooling system fixed on an XY translation stage. To perform precision surface structuring, an industrial femtosecond fiber laser was used to produce linearly polarized laser pulses with a center wavelength at 1030 nm, a pulse duration of ~500 fs and a repetition rate of 100 kHz. Standard single-crystalline silicon wafers (100)-oriented, single side polished, 500-µm thick), fused silica glass pieces, and borosilicate glass rods (Schott Borofloat 33, 10-mm diameter) were used as the workpiece materials. According to the thickness of the frost layer, several objective lenses with different numerical apertures (N.A.) were employed for focusing the laser beam onto the workpieces. To form the frost layer, a thermoelectric cooler sandwiched between the workpiece and a heat sink was used to keep the surface temperature of the workpiece below the freezing point. The surface temperature was automatically controlled by a closed-loop temperature control system including a temperature detector and a proportional integral differential controller.

The thickness of the frost layer on the workpiece was measured by a three-axis measuring microscope with a 50× objective lens (N.A. = 0.85) and an axial resolution of ~1 µm. The surface morphology of ablation patterns was obtained by an optical microscope (Olympus BX43) and a field emission scanning electron microscope (SEM, Tescan Mira3). The 2D depth profiles of the ablated craters were captured by the use of a white-light interferometric microscope (Bruker Contour GT). Before microscope observations, all the samples were rinsed with distilled water for 5 min and dried at room temperature. The laser power and pulse energy were measured on the target surfaces with a pyroelectric detector (Thorlabs, S442C).

3. Results and Discussion

3.1. Formation of a Thin Frost Layer

Frosting is a ubiquitous natural phenomenon by condensing water vapor in a humid environment onto the exposed surface

![Figure 1.](image-url)
whose temperature is below the freezing point. During the frost formation process, the water vapor in the air can continuously freeze on and between the existing frost crystals, which contributes to the increase in thickness and density of the frost layer.[16] Figure 1b presents a microscope image of the thin frost layer on the silicon wafer, in which the cooled silicon wafer was exposed to ambient air and its surface temperature was maintained at −5 °C. The ambient temperature and relative humidity were kept constant at 25 °C and 35%, respectively. It can be observed that, after a frosting time of ≈1 min, the frost layer was relatively uniform and dense over the whole surface, and the average size of the frost crystals was ≈4 μm. The dense frost crystals can be attributed to its relatively high frost surface temperature close to the melting temperature,[17] which is helpful for achieving a good consistency of frost-assisted laser processing.

Equation S1 (Supporting Information), suggests that the slow rate of frost growth depends on high frost surface temperature, low ambient humidity, and low convective mass transfer coefficient. In our experiments, to obtain a thin frost layer with stable thickness, the surface of the silicon wafer was maintained at a temperature slightly below the freezing point, and the relative humidity of ambient air was set at 35%. Figure 1c presents the evolution of frost thickness over condensation time at different cold surface temperatures, showing that the thickness of frost increases slowly with time. It can be seen that a frost layer with a thickness in the range of 5–15 μm can be obtained in a condensation process of 10 min. The stability of frost layer thickness could be further improved by using an on-line thickness measurement system in the future.

3.2. Surface Morphology and Ablation Rate

The surface morphology and ablation rate between traditional ablation in air and frost-assisted ablation were compared under the same machining parameters, including laser parameters, focusing lens, exposure time, scanning velocities, etc. For simplicity, the focusing planes in frost-assisted ablation were set at the surface of the thin frost layer, while the focusing planes of laser ablation in the air were exactly set at the silicon surface. As a result, the focus spot sizes on the target surface are not strictly identical for the two comparative cases. Fortunately, the slight difference in spot size does not significantly change the surface morphologies for high-fluence ablation far above the threshold value (See Section 2, Supporting Information).

Figure 2a,b shows the micromorphology of 2D barcodes inscribed on a silicon plate without and with frost assistance, respectively. The barcodes consist of an array of ablated craters ablated by a tightly focused femtosecond laser (N.A. = 0.45). The thickness of the frost layer was controlled at ≈15 μm. The single ablated crater was produced by 1000-pulse irradiation at a pulse energy of 2.5 μJ. One can see that there is a large amount of debris around the ablated craters, and a thick recast layer is formed in the craters for traditional processing. In contrast, for the frost-assisted counterpart, the silicon surface is very clean and no resolidified material can be seen in or around the craters. This phenomenon may be attributed to the rapid melting of the thin frost layer under irradiation of femtosecond pulses. In the process of frost-assisted laser ablation, one can clearly see that the melted water was frozen when the laser is off (See Movie Clip 1, Supporting Information). The ice-water phase transition after a fast temperature jump could occur on a time scale of 100 ps.[18] During this period, heat conduction to the silicon substrate and the lateral frost layer is not obvious.[19,20] Therefore, a local and thin water film could be instantaneously produced under the irradiation of first laser pulses, resulting in clean and debris-free ablated craters by successive laser pulses. These results are consistent with previously reported femtosecond ablation under sprayed thin water film.[21] Nevertheless, the thickness of the frost layer is more uniform and controllable over the sample surfaces in comparison with a water film.

The efficiency of frost-assisted laser ablation was evaluated by comparing profiles of craters. Figure 3a,b presents 2D depth profiles of the ablation craters after multiple-pulse irradiation on silicon covered without and with the frost layer. The thickness of the frost layer was controlled at ≈20 μm and a 10× objective lens (N.A. = 0.3) was used to produce ablation craters with a diameter of ≈12 μm. One can see that the crater depths by frost-assisted ablation are significantly larger than those without a frost layer, while the crater diameters remain roughly unchanged. For different pulse energies from 5 μJ to 9 μJ, an ≈40–60% improvement in ablation rate was found, as shown in Figure 3c. Here, the ablation rate is defined as the average crater depth divided by the number of applied laser pulses. This improvement could be based on the same mechanism for water-assisted laser ablation:[22] Water from melting frost disperses the ablation debris and reduces the recast layer, resulting in less blockage for successive laser pulses into the ablation craters. Note that the 40–60% improvement of ablation rate by frost-assisted ablation is lower than the previously reported results achieved by the use of a thin layer of water film with a thickness of few micrometers,[23] which could be due to the fact that the extra laser pulses are required to melt the opaque frost layer.

3.3. High-Spatial-Frequency (HSF) LIPSSs on Silicon

To demonstrate the high-precision processing capability of frost-assisted laser ablation, a square grid with a size of 20 × 20 μm was inscribed on a silicon plate with a tightly focused femtosecond laser at a pulse energy of 1 μJ. The ablated grooves with widths as small as a few microns were produced with a 50× objective lens (N.A. = 0.85) and a single scan with a translation speed of 1 mm s⁻¹. At the same time, in order to rapidly make the frost layer melt under low-energy laser irradiation, the thickness of the frost layer was controlled to be as tiny as ≈9 μm. Figure 4a,b shows laser-ablated microgrooves without frost assistance, and there are a large amount of debris and considerable recast layers around these microgrooves, which cannot be removed even after a 5-min ultrasonic cleaning in water. In contrast, when the silicon wafer was covered with a thin frost layer, both debris deposition and recast layer are fully eliminated, and smooth and uniform microgrooves can be obtained, as shown in Figure 4c. It can be seen from Figure 4d that HSF laser-induced periodic surface structures (LIPSSs) with a ripple spacing of 110 ± 10 nm
Figure 2. Optical micrographs of 2D barcodes inscribed on silicon covered without (a) and with (b) frost layer; SEM micrographs of a single ablated crater inscribed on silicon covered without (c) and with (d) frost layer.

Figure 3. Crater depth profiles in the air and under a thin frost layer after 1000-pulse irradiation at different pulse energies: a) 5 µJ and b) 9 µJ; c) Ablation rates of silicon as a function of laser pulse energy in the air and under thin frost layer.
(≈λ/9) are uniformly distributed in the whole grooves. The orientation of the frost-assisted HSF LIPSSs is perpendicular to the laser polarization, coinciding with the orientation of the HSF LIPSSs formed in water, \[23,24\] which suggests that both structures could have a similar formation mechanism. To the best of our knowledge, the formation of HSF Si-LiPSSs is previously confined in water \[23,24\] or at very low laser fluence close to the ablation threshold, \[25\] and it is the first demonstration that large-area uniform HSF Si-LiPSSs at high laser fluence (≈25 J cm\(^{-2}\), Supporting Information) could be achieved with the assistance of thin frost layer, which offers a potential to achieve high-efficiency and large-area uniform hierarchical nanotexturing.\[26\]

Figure 5(a) shows a schematic scenario of frost-assisted femtosecond laser ablation at high fluence. Due to the transient ice-water phase transition induced by femtosecond laser,\[18\] the ablated grooves could be covered by a local water film resulting from the melting of the peripheral frost layer. Moreover, the ablated silicon surface could be oxidized under high-fluence irradiation, resulting in a thin silicon oxide layer sandwiched

\[\text{Figure 4. SEM micrographs of micro-grids inscribed on silicon covered without (a) and with (c) frost layer. b,d): Zoomed-in images of the area marked by the white rectangle in (a) and (c), respectively. The laser beam polarization is indicated by a white arrow in (d).} \]

\[\text{Figure 5. a) Schematic scenario for frost-assisted grooving at high laser fluence, the inset: scheme of the thin-film SPP model accounting for the HSF LIPSSs. b) The calculated SPP period as a function of electron density } N_e \text{ of water film for three different thicknesses of the silicon oxide layer. The green region indicates the experimentally observed LIPSS periods ranging from 100 nm to 120 nm. The purple dashed line corresponds to the critical carrier density threshold of water } (N_{ce} = 1.1 \times 10^{21} \text{ cm}^{-3}). \]
between the silicon substrate and the water film, as illustrated in the inset of Figure 5(a). Based on the above scenarios, a thin-film surface plasmon polaritons (SPP) model\cite{27} is used to explain the HSF LIPSS obtained in our experiment. Under high-fluence irradiation of successive laser pulses, the optically excited carrier densities in both silicon substrate and water film could transiently reach their critical values for the SPP excitation, while keeping the thin silicon oxide layer transparent. Therefore, it is reasonable to assume the SPPs were excited at both interfaces of the sandwich structure.

An implicit equation of SPP dispersion relation can be derived from the continuity conditions of the electromagnetic component as follows\cite{28}:

\[
e^{-2ik_t} = \frac{k_2 + k_1}{\varepsilon_1} \frac{k_2 - k_3}{\varepsilon_2} = \frac{k_3 - k_1}{\varepsilon_3} \frac{k_3 + k_2}{\varepsilon_4}
\]

where \(k_i\) and \(\varepsilon_i\) (\(i = 1,2,3\)) are the complex wave vector component of allowed SPP modes and the complex dielectric permittivity, respectively, corresponding to three different mediums as shown in Figure 5(a), and \(t\) is the thickness of the sandwiched silicon oxide layer. The propagation constant \(\beta\) of SPP modes is determined by \(k_2 = \beta^2 - k_1^2\varepsilon_1\), and the SPP period \(\Lambda_{SPP}\) can be calculated by \(\Lambda_{SPP} = 2\pi / \text{Re}(\beta)\). Using a Drude model, the complex dielectric permittivity of the excited silicon and water as a function of carrier density \(N_e\) can be derived as follows\cite{29}:

\[
e_{SPP} = \varepsilon_{\infty} - \frac{N_e\varepsilon_0^2}{\varepsilon_0 m^* (\omega^2 + i\omega/\tau)}
\]

in which \(\omega\) is the incident light frequency in vacuum, \(\tau\) is the Drude damping time of free electrons, and \(\varepsilon_{\infty}, m, m^*\) and \(\varepsilon\) are the dielectric permittivity of the vacuum, the electron mass, the optical effective mass of carriers, and the electron charge, respectively. \(\varepsilon_{\infty}\) is the dielectric constant of the non-excited material at the irradiation wavelength written as \(\varepsilon_{\infty} = (n_f + i\kappa)^2\), where \(n_f\) and \(\kappa_f\) are the refractive index and the extinction coefficient, respectively.

In the numerical calculation of the SPP period, for simplicity, the carrier density of the laser-excited silicon is fixed to \(6 \times 10^{21} \text{ cm}^{-3}\).\cite{27} The Drude model parameters and the optical constants at the irradiation wavelength of 1030 nm were given as follows: \(n_1 = 1.321, n_2 = 1.450, n_3 = 3.563, \kappa_1 = \kappa_2 = 0, \kappa_3 = 2.2 \times 10^{-4}, m_{\text{silicon}} = 0.18, m_{\text{water}} = 0.5, \tau_{\text{water}} = 1.1 \text{ fs}, \tau_{\text{silicon}} = 1.7 \text{ fs}\).\cite{27,10} Figure 6(b) shows the SPP period calculated as a function of the carrier density of the laser-excited water for different thicknesses of the silicon oxide layer. One can see that at a carrier density of \(\approx 2.2 \times 10^{21} \text{ cm}^{-3}\), the SPP periods for different thicknesses (silicon oxide layer) of 5 nm, 8 nm, and 12 nm simultaneously reach minimum values of \(\approx 44 \text{ nm}, 70 \text{ nm},\) and \(104 \text{ nm}\), respectively. The results shown in Figure 6(b) suggest
that the thickness of sandwiched silicon oxide has a significant influence on the SPP period. When the silicon oxide thickness is below 12 nm, the experimentally observed LIPSS periods of ≈110 nm can be achieved at both low and high carrier density, which are in good agreement with our experimental results on HSF LIPSSs at high fluence, also consistent with the previously reported HSF LIPSSs at low fluence.[27]

3.4. Microtexturing on Curved Surfaces

The homogeneous frost layer could be formed on the whole cold surfaces exposed to humid air if the cold surfaces are maintained at a uniform temperature below the freezing point. As a result, the frost-assisted strategy is also applicable to curved surfaces. To demonstrate this capability, a proof-of-principle experiment was performed for microtexturing on the cylindrical surface of a borosilicate glass rod by frost-assisted laser ablation. As illustrated in Figure 6a, a borosilicate glass rod was clamped in a rotating fixture and was close to a set of V-shape thermoelectric coolers, and a homogenous frost layer was produced on the surface of the glass rod by slowly rotating the cooled rod. The high-quality hexagonal micropatterns, as shown in Figure 6b, were inscribed on the cylindrical surface by the combined motion of a rotary motor and a galvo scanner. The femtosecond laser with a pulse energy of 25 µJ was focused by an f-theta lens with a focal length of 100 mm. The ablated grooves with a width of ≈12 µm were fabricated with a single scan at a speed of ≈100 mm s⁻¹. Figure 6c,d presents zoom-in micropatterns fabricated by traditional and frost-assisted microtexturing, respectively. It can be clearly observed that there are debris redeposition zones with a width of ≈30 µm along the grooves, while the textured surface with frost assistance is quite clean and debris-free. The high-quality and high-efficiency surface microtexturing on curved surfaces will find many practical applications in anti-friction bearings and wear-resistant tools.

With the assistance of a thin frost layer, HSF LIPSSs on silicon can be induced by a single fast scan with high laser fluence, which offers the potential to achieve high-efficiency and large-area uniform hierarchical nanotexturing. A thin-film SPP model is successfully used to explain the HSF LIPSSs obtained in our experiment, suggesting that the thickness of sandwiched silicon oxide layer has a significant influence on the SPP period. Noting that the intrinsic frost formation process makes its thickness controllable and homogeneous at large scales, we speculate that the frost-assisted femtosecond laser ablation could open a new way to large-area uniform and high-quality 2D and 3D surface micro/nano-structuring across a broad range of applications.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords
femtosecond laser, frost-assisted, high-spatial-frequency, laser induced periodic surface structures, surface micro/nanostructures

4. Conclusion

In this work, we present a novel frost-assistance strategy to tackle the common issues for femtosecond laser ablation at high laser fluence, such as redeposition of ablated debris and formation of recast layer. A thin frost layer was preliminarily formed by condensation of ambient water vapor onto the sample surfaces with a temperature below the freezing point. When femtosecond laser is focused onto the frost-covered surfaces, the local frost layer around the laser spot will melt into water film, helping to boost ablation efficiency, suppress recast layer, and reduce debris redeposition. The experiment results demonstrate that the frost-assistance strategy enables high-quality and high-efficiency surface texturing at high laser fluence, and also is applicable to curved surfaces and various different materials (Supporting Information). It is noteworthy that the redeposition of debris and the formation of a recast layer can also be improved by lowering the laser fluences to the ablation threshold or decreasing the repetition rate of laser pulses to suppress the thermal effect, but these methods will significantly reduce the efficiency of surface texturing.

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