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Femtosecond laser ablation of polytetrafluoroethylene (Teflon) in ambient air

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Teflon, polytetrafluoroethylene (PTFE), is an important material in bioscience and medical application due to its special characteristics (bio-compatible, nonflammable, antiadhesive, and heat resistant). The advantages of ultrashort laser processing of Teflon include a minimal thermal penetration region and low processing temperatures, precision removal of material, and good-quality feature definition. In this paper, laser processing of PTFE in ambient air by a Ti:sapphire femtosecond laser (780 nm, 110 fs) is investigated. It is found that the pulse number on each irradiated surface area must be large enough for a clear edge definition and the ablated depth increases with the pulse number. The air ionization effect at high laser fluences not only degrades the ablated structures quality but also reduces the ablation efficiency. High quality microstructures are demonstrated with controlling laser fluence below a critical fluence to exclude the air ionization effect. The ablated microstructures show strong adhesion property to liquids and clear edges that are suitable for bio-implantation applications. Theoretical calculation is used to analyze the evolution of the ablated width and depth at various laser fluences. © 2003 American Institute of Physics.

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I. INTRODUCTION

Polytetrafluoroethylene (PTFE) $(-CF_2-)_n$ has a wide range of unique physical and chemical properties such as biocompatible, low frictional resistance, low dielectric constant, low surface adhesion, thermal and chemical stability, which make it essential for numerous applications. Due to the compatibility with biological tissues, it has recently attracted special attention in bioscience and medical applications. The implantation of PTFE pieces with desired functional structures into human body is a routine job for surgery in nowadays. Micromachining of PTFE is, however, a difficult task. First, conventional micromachining technology^{1,2} cannot be applied to PTFE due to the low fluidity after it is melted. Second, PTFE is not sensitive to the attacks of agents and there is no solvent to dissolve it.² Moreover, as a dielectric polymer, PTFE is not a suitable material for electric discharge micromachining.³ Recently, some promising techniques, such as synchrotron radiation⁴ (SR) and pulsed laser ablation,^{5,6} have been successfully applied to make microparts of PTFE. In SR etching process, the sample is pre-processed by electron beam (EB) method and then exposed to SR through a mask with desired absorber pattern. The sample temperature is required to be a constant during irradiation. In contrast, pulsed laser ablation is more simple and versatile since it is a mask-free etching process.⁶ Under laser irradiation, PTFE exhibits almost no absorption from infra-

red (IR) to ultraviolet (UV) spectrum with its first linear absorption peak at 160 nm.⁷ Conventional nanosecond laser systems are thus not the ideal irradiation source for high quality micromachining of PTFE.⁵ Clean laser ablation of PTFE can only be achieved by vacuum-ultraviolet (VUV, 160 nm or less)^{8,9} lasers or ultrashort pulse lasers on a femtosecond (fs) time scale.^{5,10} In general, fs laser ablation offers advantages over long pulse laser because there is little or no collateral damage due to shock waves and heat conduction produced in the material being processed. Recent work,¹¹ however, shows that there are two completely different ablation phases for most dielectric materials by fs laser ablation: a gentle and smooth sputtering phase followed by a strongly violent explosive ablation phase. The latter phase is identified as the result of a thermal mechanism responsible for material removal. To exclude effects arising from the heating of the sample as well as the surrounding medium, many authors selected a low repetition rate of 1 Hz or a high vacuum with a pressure below 10^{-5} Torr in their investigations on fs laser ablation of PTFE.^{5,10} Fast structuring of PTFE in ambient air by ultrashort lasers remains a challenge due to the sensitivity of the temporal and spatial dependence of the laser intensity. Several studies have already shown the important role of ambient pressure¹² and ambient gas composition¹³ on the ablated structure quality for ultrashort laser irradiation. The screening effect of the plasma, on the other hand, exerts a significant influence on the ablation process at high laser fluences.¹⁴ Meanwhile, the plasma dynam-

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ics is strongly affected by the ambient pressure during its expansion, which in turn makes the laser ablation more complicated.

In this paper, fast structuring of PTFE in ambient air with ultrashort pulses at a high repetition rate is investigated. Morphology of the irradiated surface by single pulse and multiple pulse irradiation is compared. Ablated structure quality is analyzed to check the reproducibility of cross section profile and groove edge quality. Ablated width and depth are measured and compared with theoretical calculations. This technique is finally applied to fabricate high quality microstructures on PTFE in ambient air and two biological microstructures are demonstrated. The ablated structures are tested with sessile liquid drops to characterize its hydrophilic property.

II. EXPERIMENT

The laser system consisting of a Ti:sapphire oscillator (Spectra-Physics Tsunami) and a regenerative amplifier (Spectra-Physics Spitfire) provides high-intensity fs laser pulses for the study. A self-mode-locked Ti:sapphire laser oscillator produces ~ 80 fs pulses at a wavelength of 780 nm and a repetition rate of 80 MHz. The oscillator provides seed pulses into the regenerative amplifier, which is based on chirped pulse amplification (CPA) technique. The pulse duration of the output beam from amplifier is 110 fs with nominal wavelength at 780 nm. The repetition rate can be set to either 1 or 1000 Hz and the beam profile emitted from the regenerative amplifier is approximately Gaussian shape. Beam diameter at the laser exiting window is elliptical with an average diameter of ~ 5 mm. The laser beam is redirected with a 45° high reflectivity dielectric mirror through an adjustable aperture placed in front of a BK7 Plano-convex lens with a focal length of 40.6 mm, as shown in Fig. 1. The focused spot is around $100 \mu\text{m}$ on the substrate surface. The aperture diameter can be varied from 1 to 10 mm and is used to minimize the effects of spherical aberrations. It provides a round beam at the workpiece and improves the final feature quality. The whole system is turned on and kept continuous working for more than 30 minutes to have a stable laser energy output before the fs laser processing of PTFE.

The 1 mm thick commercial Teflon PTFE (DuPont) with a density of approximately 2.15 g/cm^3 is used as the sample. The surface is polished and cleaned with methanol before laser ablation. A three-axis precision translation stage is used to change the laser irradiation spot on sample surface. Resolution of the stage is $1 \mu\text{m}$ with a total travel range of 25 mm. Translation speed of the stage can be adjusted from $1 \mu\text{m/s}$ to 3.5 mm/s . A series of experiments are carried out to ensure that the system is well focused. Since high fs laser fluences can induce the ionization of ambient air that in turn presents a bright plasma dot at the focus position, the sample is first placed slightly below but very near to the white dot position from a side-view inspection. Then a 10 mm long groove is engraved onto the sample surface at a speed of 1 mm/s and a laser fluence of 4.0 J/cm^2 . After that, the sample is moved towards the focal lens by $10 \mu\text{m}$ by the z stage. Another 10 mm long groove is engraved at the same pulse

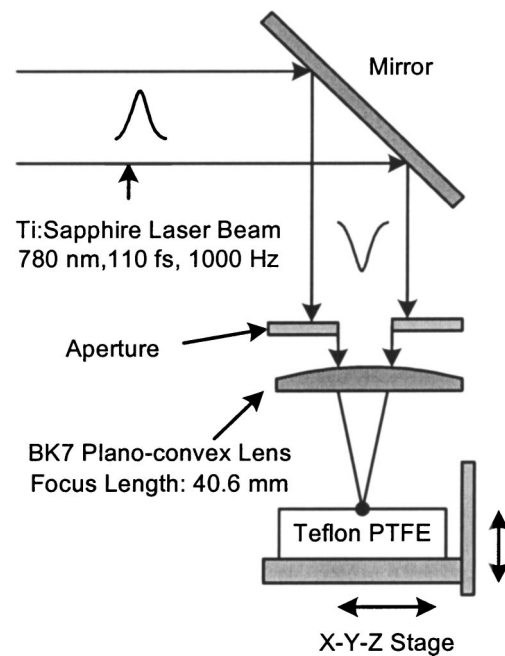


FIG. 1. Femtosecond laser ablation experimental setup.

energy. This process is repeated until a side-view inspection shows that the white-dot position is below the sample surface. The final grooves are then evaluated under an optical microscope to measure their grooved width. The z axis position relating to the smallest groove is corresponding to the best focus position. Treated PTFE sample is examined with an optical microscope and a scanning electron microscopy (SEM: Hitachi S-4100). The ablated profile is analyzed by an Alpha-Step 500 profilometer (Tencor Instruments). Since fs laser ablation of dielectrics materials at high laser fluences or large pulse numbers could produce deep channels,¹⁵ it should be noted that these narrow channels would prevent the probing tip of a profilometer dipping into the groove bottom and therefore calibration of an accurate measurement of ablated depth is necessary. In the experiment, the ablated depth are first measured by an Alpha-Step 500 profilometer and then compared with SEM cross-section observation results. In hydrophilic tests, Bromonaphthalene, doubly distilled water, and aqueous solutions of different PHs (prepared by adding NaOH or HCl in water according to Ref. 16) are used. The contact angles are measured using a Rama-Hart contact angle measuring system (Model: 100-00) equipped with a camera and an image processing system. This allows us to determine the adhesion properties of ablated surface with the liquid and to investigate the acidic and basic properties of the surfaces.¹⁶

III. RESULTS AND DISCUSSION

A. Influence of pulse number on fs ablation

In our experiment, there are two controllable laser parameters: pulse number N and laser fluence F . The quantity N is determined by the setting of laser repetition rate and sample scanning speed as

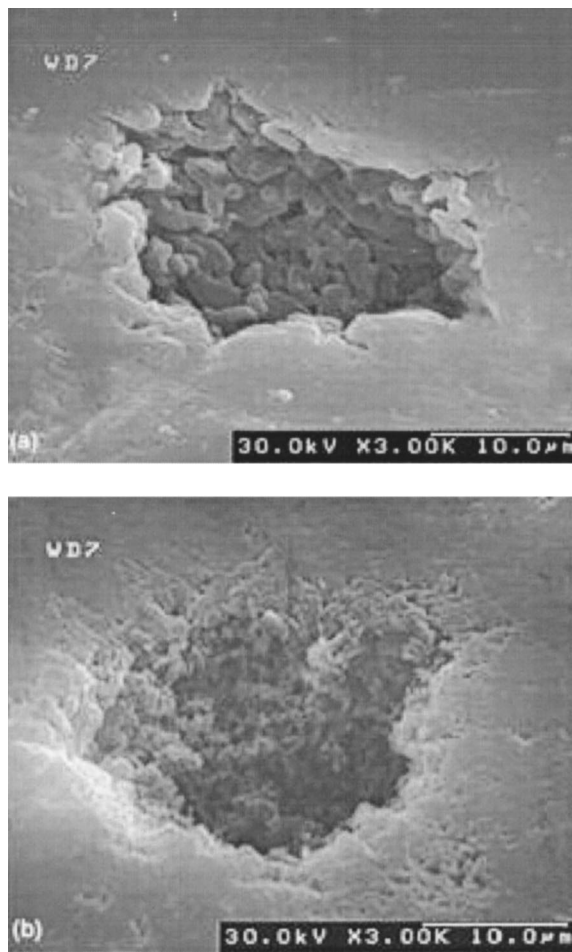


FIG. 2. (a) and (b) SEM images of craters formed on PTFE by fs laser ablation at a laser fluence of 1.0 J/cm^2 and different pulse numbers of (a) 1 and (b) 5, respectively.

$$N = \frac{R \times S}{\nu}, \quad (1)$$

where R represents repetition rate (either 1 or 1000 Hz), S spot size ($\sim 100 \mu\text{m}$), and ν scanning speed (from $1 \mu\text{m/s}$ to $3500 \mu\text{m/s}$). It is found that the ablated surface shows different morphologies for different pulse numbers. Figure 2 shows the SEM images of craters formed on PTFE with fs laser ablation at a laser fluence of 1.0 J/cm^2 and a pulse number of (a) 1 and (b) 5, respectively. It can be observed from Fig. 2(a) that laser ablation by one pulse induces the growth of microcones with $3\text{--}4 \mu\text{m}$ diameter inside the crater. This phenomenon has been reported for many other polymers and the fundamental physics is still under investigation.^{17–19} However, it is clear that one pulse laser ablation cannot produce clear ablation edge and its shape is highly inhomogeneous. Compared with original PTFE surface, the ablated crater surface presents different absorption properties to the consecutive laser pulses and in turn changes its morphology.²⁰ In our experiment, as pulse number increases to 5, the crater becomes a little bit wide and microcones are disappeared. The accompanying phenomenon is the formation of many micrograins with submicron diameter inside the crater. The improvement in crater quality is ben-

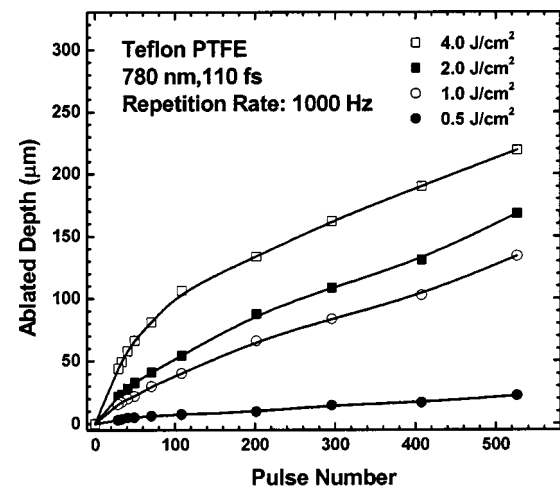


FIG. 3. Ablated depth versus pulse number for laser fluences of 0.5, 1.0, 2.0, and 4.0 J/cm^2 .

eficial to high-quality microstructuring process and therefore a high repetition rate is recommended for the fast structuring of PTFE.

Figure 3 presents the ablated depth as a function of pulse number at a repetition rate of 1000 Hz and a laser fluence of 0.5, 1.0, 2.0, and 4.0 J/cm^2 , respectively. The ablated depth increases linearly with the pulse number when the applied pulse number is smaller than 100. At high laser fluences, the slope of the ablated depth versus pulse number decreases due to the beam divergence at the depth over $100 \mu\text{m}$. The linear dependence makes it possible to calculate the ablated depth by multiplying the ablation rate and the irradiated pulse number. In order to meet a good linear dependence condition, the following experiments are carried out at a scanning speed of 1 mm/s , which corresponds to a number of 100 shots on each irradiated surface area.

B. Ablated groove quality

In most cases, the pulse number on each irradiated surface area can be fixed as a constant. In the following study the sample scanning speed is set as 1 mm/s (corresponding to 100 pulses/spot) and 10 mm long grooves are engraved on PTFE with different laser fluences in the range from 0.4 J/cm^2 to 5.0 J/cm^2 . Optical microscope examination reveals that the debris depositions become observable for those grooves ablated at laser fluences above 1.2 J/cm^2 . Figures 4(a) and 4(b) give the top view of the grooves formed on PTFE by fs laser ablation at a laser fluence of 1.0 J/cm^2 and 2.0 J/cm^2 , respectively. There is no clear debris deposited along the groove edges in Fig. 4(a) while the edges quality slightly degrade and deposited debris can be observed along the groove edges in Fig. 4(b). It may be due to the plasma generation from multiphoton ionization of the ambient air when laser intensity goes higher than 10^{13} W/cm^2 , which corresponds to 1.1 J/cm^2 for $\tau_p = 110 \text{ fs}$. We found that the debris is stick very stubborn around the groove edges and it cannot be easily removed by methanol cleaning. It is therefore suggested to use a low laser fluence below 1.2 J/cm^2 for fast microstructuring of PTFE in ambient air.

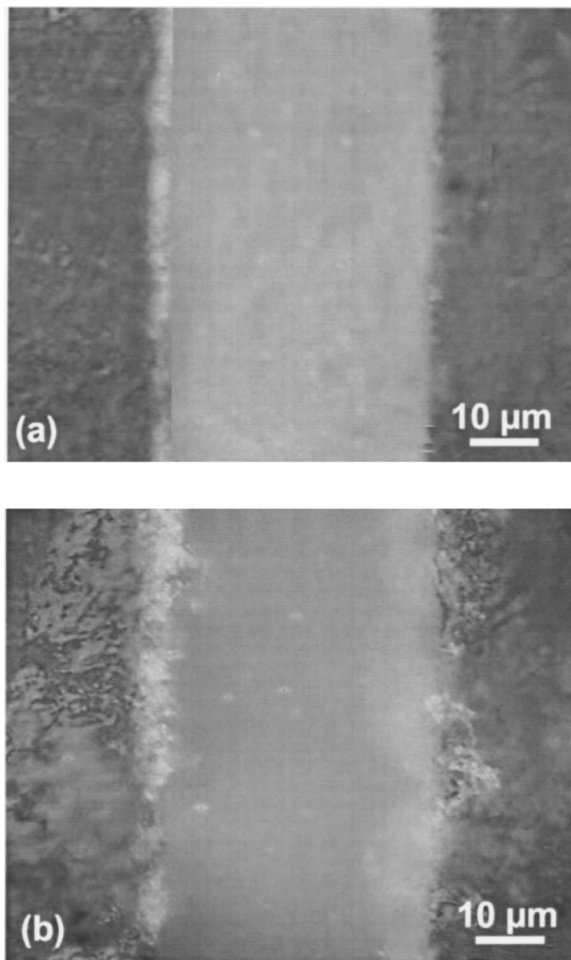


FIG. 4. (a) and (b) Top view of the grooves ablated by fs laser at laser fluences of (a) 1.0 J/cm^2 and (b) 2.0 J/cm^2 .

Furthermore examination of the ablated grooves in terms of the cross section quality is carried out. Cross section analysis is repeated five times for each groove at different positions by the profilometer and the results are compared with SEM cross-section observation results. Figure 5 shows the typical cross sections of the grooves by fs laser ablation

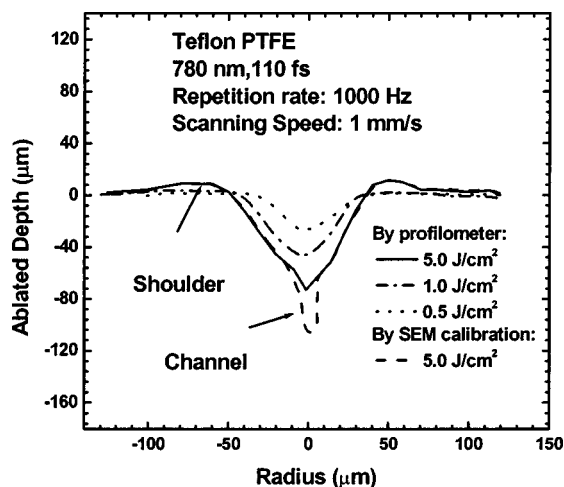


FIG. 5. Cross-section profiles of the grooves ablated by fs laser at laser fluences of 0.5, 1.0, and 5.0 J/cm^2 .

at laser fluences of 0.5, 1.0, and 5.0 J/cm^2 . It is found that deep channels would be formed when high laser fluences are applied. It is also found that, for grooves ablated with laser fluences below 1.2 J/cm^2 , profiles at five different positions are almost the same and there is no thermal-induced shoulder along the groove edges. As laser fluence increases, the thermal-induced shoulder emerges due to the enhancement of heating process. At a laser fluence of 5.0 J/cm^2 , the shoulder height can be up to $8 \mu\text{m}$. Meanwhile, when the laser fluence is higher than 1.2 J/cm^2 , profiles at different positions show poor reproducibility. It can be attributed to fact that the debris deposition inside the grooves ablated with high laser fluences. Another possible reason is that, at high laser fluence, multiphoton ionization becomes stronger that in turn causes the fs pulses undergoing strong spatial and temporal reshaping. In the experiment, “bright-plasma-dot (white-light continuum)” at the focus position due to the air ionization is observed when laser fluence is higher than 1.2 J/cm^2 (the sample is temporally taken away from the focus position for the observation).

To get high quality microstructures by fs laser ablation in ambient air, it is now clear that we need to control the laser fluence below a critical value of 1.2 J/cm^2 . Two high quality biological microstructures, as shown in Figs. 6(a) and 6(b), are fabricated by fs laser ablation at a laser fluence of 1.0 J/cm^2 and a scanning speed of 1 mm/s . These devices are in a size range of several hundred micrometers and feature their clear edge definitions and contamination-free surfaces. The hydrophilic tests of these devices are based on the contact angle measurements by using prepared neutral, acidic and base solutions and the reversible work of adhesion which indicated the variation of the polar component of the work of adhesion are automatically given by the measurement system (no significant modification of the dispersive component is ever observed after the treatment). The total work of adhesion of the untreated PTFE surface with neutral water is around 90 mJ/m^2 . The ablated surfaces show big increase in the work of adhesion with all test solutions. The measured values are 243, 243, and 255 mJ/m^2 for neutral, acidic and base solutions respectively; this indicates a weak acidic character of the ablated surface that makes these devices suitable for bio-implantation applications.²¹ The improvements in the surface adhesion properties could be due to the surface morphology modification as well as the photochemical modification of the ablated surface. After laser irradiation of PTFE in ambient air, it may result in defluorination of the irradiated surface with surface oxidation²² and substitution of F atoms by different functional groups.^{23,24}

C. Ablated depth and width versus laser fluence

For laser fluences above the threshold fluence, the multiphoton absorption dominates and absorbs laser energy efficiently into the material. Figure 7 shows the ablated groove depth and width as a function of laser fluence. The width φ can be calculated by assuming that Gaussian-shape laser intensity at the ablated groove edge should be higher than the threshold intensity for material removal, which is given by²⁵

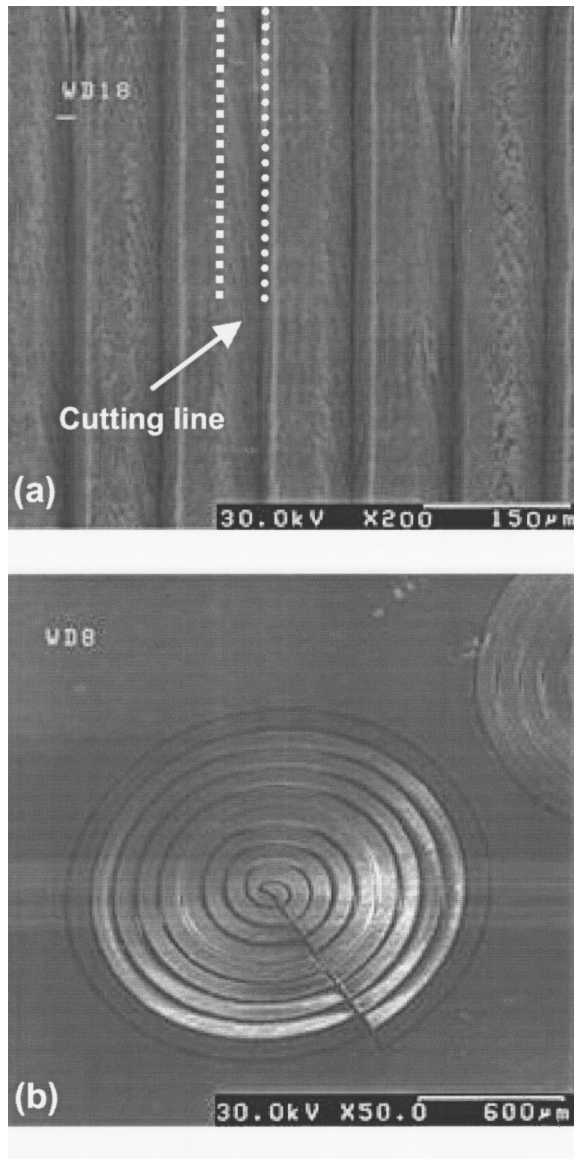


FIG. 6. (a) and (b) SEM images of high quality (a) channel-like structure and (b) concentric structure ablated by fs laser at a laser fluence of 1.0 J/cm² and a scanning speed of 1 mm/s.

$$\varphi(\mu\text{m}) = \varphi_0(\mu\text{m}) \times \sqrt{\ln \frac{I(\text{W}/\text{cm}^2)}{I_0(\text{W}/\text{cm}^2)}}, \quad (2)$$

where φ_0 and I_0 are ablated groove width and laser intensity at ablation threshold. The theoretical fitting curve is represented in Fig. 7 by the dashed line and it is in good agreement with the experimental results.

In the case of fs laser ablation of PTFE in vacuum, Kumagai *et al.* have shown both experimentally and theoretically that the ablation rate of fs laser ablation of polymer materials follows^{10,26,27}

$$d(\mu\text{m}/\text{pulse}) = \frac{1}{n\rho_0} \int_{S_{\text{th}}}^{S_0} \frac{dS}{1 - \exp(-\sigma_n K_n S^n)}, \quad (3)$$

where n represents n -photon simultaneous absorption, ρ_0 the density of ground-state chromophores, S_{th} the threshold photon density for n -photon ablation, S_0 the incident photon density, $K_n = A_n / \tau^{n-1}$ the pulse-shape-dependent constant, τ

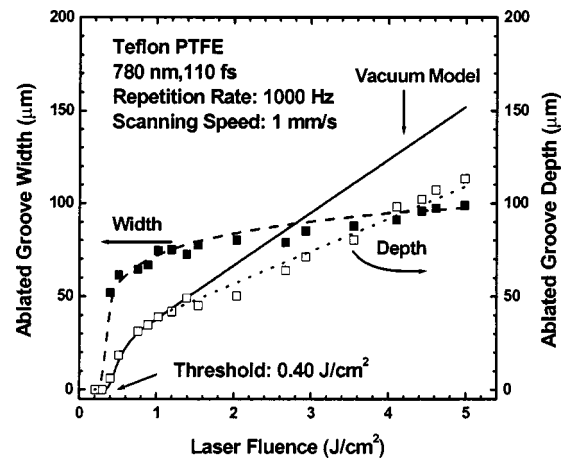


FIG. 7. Dependence of ablated groove width and depth on laser fluence for fs laser ablation of PTFE in ambient air at a repetition rate of 1000 Hz and a scanning speed of 1 mm/s. Dashed line for width fitting and solid line for depth fitting (vacuum-ablation model).

the characteristic time for the laser pulse, and σ_n the effective n -photon absorption cross section. Since the ablated depth increases linearly with the pulse number in a range from 1 to 100 pulses, the ablated depth can therefore be calculated as the product of the ablation rate and the pulse number as $D_n = N_{\text{pulse}} d_n$. The laser fluence is related to photon density S by $F = h\nu S$, where $h\nu$ is the single photon energy. The following equation can be substituted for Eq. (3) as

$$D_n(\mu\text{m}) = \frac{\lambda N_{\text{pulse}}}{n\rho_0 h c} \int_{F_{\text{th}}}^{F_0} \frac{dF}{1 - \exp(-\sigma_n K_n (\lambda/hc)^n F^n)}, \quad (4)$$

where λ is the laser wavelength, h is the Planck's constant, and c is the speed of light in the medium.

Using Eq. (4), the dependence of the ablated depth of PTFE on laser fluence is calculated for $n = 5$, i.e., five-photon absorption that is the dominant multiphoton absorption channel for IR fs laser ablation of PTFE. The threshold of the laser fluence $F_{\text{th}} = 0.40 \text{ J}/\text{cm}^2$ is taken from the experiment and the density of chromophores $\rho_0 = 2.75 \times 10^{22} \text{ cm}^{-3}$ is assumed to be the same in Ref. 10. The unknown pulse-shape-dependent constant $\sigma_5 K_5$ for five-photon absorption is estimated as $1.1 \times 10^{-92} \text{ cm}^{10}$ by solving Eq. (4) for one selected experimental data point $F_0 = 0.8 \text{ J}/\text{cm}^2$ and $D_n = 32 \mu\text{m}$. The calculated ablated depth for the laser fluence in a range from 0.40 J/cm² to 5.0 J/cm² is shown in Fig. 7 by the solid line. The fitting work reveals two important points: (1) when the laser fluence is below the critical value of 1.2 J/cm², the ablated depth in ambient air is almost the same as that in vacuum. The air influence on the ablation process is negligible. (2) However, when the laser fluence goes beyond the critical fluence, the ablated depth in vacuum is higher than that in ambient air at a same laser fluence, and this deviation increases as the laser fluence increases. The difference comes from the employment of different processing environment (in ambient air or in vacuum). According to Aközbeke *et al.*,²⁸ when a strong fs laser pulse propagates through the air, self-focusing, and plasma generation process take place and complete with each other. Self-focusing is the

mostly dominant effect and plasma contribution may be neglected for laser intensity below 10^{13} W/cm². This laser intensity corresponds to a laser fluence of 1.1 J/cm² for a 110 fs pulse, which is close to the critical fluence of 1.2 J/cm² in the experiment. When laser intensity exceeds 10^{13} W/cm², multiphoton ionization of two main ingredients of the atmosphere, N₂ and O₂, dominates and shields the incident laser beam. Therefore, the ablation efficiency is reduced. To explain the curve of the ablated depth for laser fluence higher than 1.2 J/cm², a more complicated model that takes into account both the laser-matter interaction during the pulse period, and the different stages of relaxation of the solid, including plasma expansion, thermal diffusion, vaporization and cooling is under investigation.

IV. CONCLUSIONS

This work studies direct processing of Teflon PTFE in ambient air by an ultrashort Ti:sapphire laser. It is found that PTFE exhibits different surface morphologies when different pulse numbers are used and the ablated depth increases with the pulse number. To obtain a high quality microstructure, each irradiated surface area should receive a sufficient pulse number of laser irradiation and laser fluence should be controlled below a critical fluence of 1.2 J/cm². Above the critical fluence, the ablated structure quality is degraded and debris deposition is formed along the edges. Theoretical fitting based on a vacuum-ablation model reveals that, in the low fluence regime, the ablation efficiency in ambient air is almost the same as that in vacuum. However, in the high fluence regime above the critical value of 1.2 J/cm², the ablation efficiency in the air is smaller than that in the vacuum due to air ionization. Two high quality biological microstructures with strong adhesion to water, acid and base solutions (good hydrophilic properties) are demonstrated.

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