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Beam waist position study for surface modification of polymethyl-methacrylate with femtosecond laser pulses

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ABSTRACT

Femtosecond lasers are versatile tools to process transparent materials. This optical property poses an issue for surface modification. In this case, laser radiation would not be absorbed at the surface unless the beam is just focused there. Otherwise, absorption would take place in the bulk leaving the surface unperturbed. Therefore, strategies to position the material surface at the laser beam waist with high accuracy are essential.

We investigated and compared two options to achieve this aim: the use of reflectance data and transmittance measurements across the sample, both obtained during *z*-scans with pulses from a 1027 nm wavelength laser and 450 fs pulse duration. As the material enters the beam waist region, a reflectance peak is detected while a transmittance drop is observed. With these observations, it is possible to control the position of the sample surface with respect to the beam waist with high resolution and attain pure surface modification. In the case of polymethyl-methacrylate (PMMA), this resolution is 0.6 μ m. The results prove that these methods are feasible for submicrometric processing of the surface.

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1. Introduction

In the last years, the use of femtosecond lasers in the fabrication of microdevices has been increasing. The applications of these devices include different areas such as microfluidics, optical waveguides and materials surface functionalization [1–3]. This short pulse duration produces really high power peaks that trigger non-linear absorption processes like multi-photon absorption, tunneling ionization [4,5] and avalanche ionization [6,7]. These processes make possible the absorption of the laser radiation and therefore modification of even transparent materials [8,9].

Among these materials, polymers are chosen for the production of microdevices because of their ease and low cost of manufacture, biocompatibility and optical transparency. One of the polymers used as substrate for microfluidic devices, polymethylmethacrylate (PMMA), is especially appropriate because of its adequate properties such as moderate hydrophilicity, good transmission at visible wavelengths (about 90%), and biocompatibility. Thus, PMMA substrates were also chosen for several applications in the field of biology that range from analysis of biomolecules to a wide diversity of observations in cellular biology [10].

http://dx.doi.org/10.1016/j.apsusc.2015.12.135 0169-4332/© 2015 Elsevier B.V. All rights reserved. Surface modification of the materials is crucial in this kind of studies since changes in the properties like the roughness of the material surface affects the flow resistance of liquids or the protein and cell adsorption efficiency on the surface [11–14]. Therefore, a controlled and precise surface modification of the material surface is essential for these studies.

Several studies with lasers of various wavelengths and pulse durations have shown that laser ablation can be a good microfabrication process on PMMA [15–17]. Nevertheless, thermal effects during the energy transfer to the material limit the resolution of the ablation. This problem can be minimized if the duration of the pulse is shorter than the time of the energy transfer to the lattice. In this regime, electron–electron impact ionization leads to the rapid formation of plasma that expands and ends producing ablation of the material, all confined in the tight region where the laser is focused [18]. The timescale of this exposure (some femtoseconds) is shorter than the energy transfer to the lattice (in the range of the picoseconds) and ablation occurs through direct plasma formation or non-thermal melting of the material [19]. Thus, thermal damage is markedly reduced in comparison with ablation produced by longer pulse duration lasers [20,21].

Since non-linear absorption processes are involved, tightly focused femtosecond lasers can produce modifications with lower size than the laser wavelength, overcoming the diffraction limit. This can be achieved by using laser pulses with incident fluence values slightly higher than the ablation threshold. In this way,

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transparent polymers have been modified with ultrashort pulsed lasers achieving submicrometric resolution [22].

High energy densities are necessary for these processes to take place, which are reached by tight focusing the laser beam at the material. Consequently, a good control of the relative position between the sample surface and the beam waist is necessary for precise surface modification. In case that the main objective is the surface functionalization, only surface changes should be produced. However, the modification of the surface of a transparent sample is not a trivial task, since finding out where the surface is depends largely on the implemented experimental system. Currently, some methods allow the adjustment of the position of the surface of transparent samples in the laser beam waist with few micrometers accuracy [23–25].

One of them is the *z*-scan method [26]. In this work we take advantage from this method to get a precise control on the sample surface position in regard to the beam waist to be able to optimize the fabrication of spots produced at the material surface and achieve high resolution for the dimensions of the spots. A sample of PMMA was irradiated at different incident energies and values for the relative position between the sample surface and the laser beam waist. Simultaneous reflectance and transmittance measurements were performed during sample irradiation. Finally, in order to characterize the results of the surface modification, scanning electron microscopy (SEM) images of the surface after irradiation were taken.

2. Experimental

The used laser produces pulses of linearly polarized nearinfrared radiation with a wavelength of 1027 nm and duration of 450 fs in an active medium consisting of a crystal of Yb:KYW. These pulses arrive at the sample after being reflected and passing through different optical elements. The beam is focused by a $50 \times$, 0.55 numerical aperture objective at about 13 mm of its outer lens. A glass coverslip between the objective and the sample surface was also included to prevent ablation debris to deposit on the objective lens. The energy distribution profile after the objective was Gaussian.

The experimental setup was equipped with three energy detectors that allowed the measurement of reflectance and transmittance through the sample for each laser pulse. One detector was used to control the energy of the incident light. The reflected light was collected by the same objective after being reflected at the sample surface facing the objective. After leaving the objective, the reflected beam followed the same path as the light coming from the laser until it encounters the coated side of a beam-splitter, which reflects it to a lens that collects this energy and focuses it into the energy detector for the reflected light. For the transmitted light, it was measured by a detector placed just after the sample.

The experiments were performed on samples of PMMA with rectangular shape ($20 \text{ mm} \times 10 \text{ mm}$, 1 mm thickness) cut with a diamond blade saw from a commercial 100% pure PMMA sheet available by Goodfellow. No special treatment was needed for the sample preparation before or after laser irradiation.

Previous to the fabrication of the spots, the sample was scanned horizontally in a total range of 2 mm at steps of 10 μ m while transmittance and reflectance were tracked in order to correct the tilt of the sample surface. When the sample was not perpendicular to the laser beam, a non flat tendency for the measured values was obtained. The horizontality of the sample surface plane with reference to the beam propagation axis was assumed when a flat tendency of the transmittance with a $\pm 5\%$ variation along the sample surface was observed.

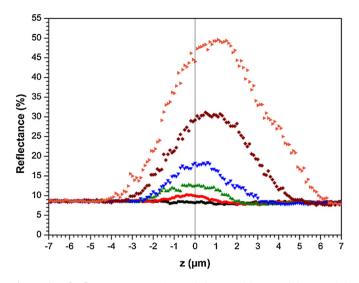


Fig. 1. Plot of reflectance vs. *z*-position at (**I**) 100 nJ, (•) 125 nJ, (**A**) 150 nJ, (**V**) 200 nJ, (**(**) 300 nJ, and (**)** 500 nJ of incident energy. The experimental error for the reflectance data is $\pm 0.3\%$.

During spot production, series at fixed incident energy and with 5 μ m separation between the impinging laser pulses on the surface were produced. The used incident energies took values in the range of 50–500 nJ. While keeping the incident energy of the pulses fixed, spots at different *z*-positions (relative position between the sample surface and the beam waist) were produced by scanning the sample surface horizontally along a total distance of 2 mm. In this way, laser pulses always impinged in pristine areas of the material. Reflectance and transmittance measurements were performed simultaneously to the spot production.

The range of *z*-position values at which lines were produced was 20 μ m with the center of this range in the position *z* = 0 μ m where the sample surface lays at the beam waist. Additional details and a further description about the setup and the *z*-scan method can be found in [25,26].

After laser irradiation, the modified surface of the sample was inspected through scanning electron microscopy (SEM).

3. Results and discussion

Reflectance changes for each single pulse on pristine material for different *z*-positions are shown in Fig. 1. Negative values for *z* indicate that the beam waist is outside the sample, while positive values correspond to positions where the beam waist is inside the sample.

These observed tendencies for reflectance measurements can be contrasted with transmittance measures that were simultaneously taken. These values are represented in Fig. 2 in the same way as reflectance measurements were plotted in Fig. 1. As it has been observed and explained in previous studies [25-27], the transmittance decrease observed as the sample enters the beam waist can be used to monitor the relative position between the sample surface and the beam waist and makes possible to ensure a good position control during the experiments. It also allows determining the beam waist position $z = 0 \,\mu m$ and therefore correct the z-position values for the experimental data [28]. The correction is based on the fact that at $z=0 \mu m$, the corresponding transmittance value is found approximately at an 83% decrease of the total transmittance difference between the transmittance value where no absorption is detected (91.4%) and the bulk transmittance value for the corresponding incident energy.

This correction makes that the *z*-position of the reflectance peak is found close to the value corresponding to the sample surface

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