Lasers in Manufacturing Conference 2015

Laser Induced Micro-Dot Generation Inside Transparent Materials: A) Formation Dynamics, Refractive Character and Internal Stress

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Abstract

The formation dynamics of ultra-short laser-induced micro-dots inside the bulk of transparent materials was studied using time-resolved phase contrast microscopy. A random laser is used as a stroboscopic illumination source, enabling the acquisition of speckle-free, time-resolved phase-contrast images with a temporal resolution in the nanosecond range.
The results demonstrate the onset and propagation of a heat front following the laser energy deposition. The heat-affected zone develops in a region that exceeds largely the footprint of the microdot. Based on the analysis of the thermal transients, it is possible to provide an estimate of the heat diffusion coefficient of the host substrate.
Complementary results obtained with polarization microscopy reveal the appearance of a permanent stress field around the microdot. Interestingly, the amount of laser-induced permanent stress depends strongly on the number of pulses and on the laser polarization. We demonstrate that by using appropriate laser parameters, ultra-short laser-induced microdots are a suitable method for embedded direct part marking in stress-sensitive materials.

Keywords: ultra-short laser processing, micro-dots, time-resolved microscopy

1. Introduction

Fs-laser pulses stand out as unique tools to perform embedded direct-part marking of transparent devices. The combination of ultrashort pulses and high numerical aperture focusing results in a highly

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confined interaction region, opening the door to volume part marking over a few cubic micrometers only. In the following, we refer to this region of permanent modification as a microdot. In the context of laser marking, a microdot constitutes a bit of information.

When the substrate is very thin and/or extremely brittle, the remaining laser-induced stress is a severe limiting factor to damage-free marking. The laser-induced stress results from the thermomechanical events which take place as an aftermath of heat dissipation.

In this proceeding, we study the formation dynamics and the characteristics of fs-laser induced microdots in the volume of a fused silica sample. The extent of the heat-affected zone during the microdot formation will be estimated. Furthermore, a quantitative value of the refractive index inside the microdot will be provided. Finally, the distribution of the laser-induced stress around the microdot will be studied.

2. Experiments

2.1. Irradiation conditions

The microdots studied in this proceeding have been generated with 80 fs duration laser pulses (FWHM duration) delivered by a Ti:sapphire system (Spitfire, Coherent Inc.). The central wavelength was 790 nm. The laser pulse energy was kept constant at a level of 2.5 µJ. The laser pulses were focused with the help of a refractive microscope objective (NA=0.5) about 200 µm deep in the bulk of a polished fused silica sample.

2.2. Formation dynamics

The formation dynamics of microdot formation was monitored with the help of a time-resolved microscopy setup (Mermillod-Blondin et al., 2013). The originality of this apparatus resides in using a pulsed random laser as an illumination source. By synchronizing the illumination with the laser pulses responsible for the microdot generation, it is possible to obtain snapshots of the material response at arbitrary time delays after the excitation. The time resolution offered by the random laser is < 10 ns, which is sufficient to study thermomechanical phenomena.

2.3. Refractive index measurement

The study of laser-induced microdots is motivated by the potential applications in laser direct part marking in the volume. In this perspective, characterizing the readability of the microdots is of prime importance. Keeping in mind that microdots are essentially phase-objects (i.e. they do not absorb light), quantitative phase-contrast microscopy techniques must be employed to reveal the internal refractive structure. Among the plethora of quantitative light imaging microscopy methods that have been recently developed, spatial light interferometry (SLIM) is particularly well suited to the study of microdots. Thanks to its designed based on common path, white-light interferometry, SLIM offers excellent guarantees in terms of stability (spatial and temporal) and resolution (< 1 nm in the direction of the optical axis of the microscope), see Wang and Popescu, 2010.

2.4. Stress visualization

A permanent stress distribution remains into the material as an aftermath of the thermomechanical events following energy deposition. In order to visualize which regions are affected, we record the stress-induced birefringence in the vicinity of the microdot by placing the sample between two crossed-polarizers with a high extinction ratio ($< 5 \times 10^{-6}$).

3. Results and discussion

3.1. Heat-affected region

Laser-induced material modification involves an energy transfer between the laser pulse and the material. The energy deposited in the sample relaxes into localized or delocalized channels. Localized channels involve for instance the creation of point defects and the appearance of new molecular structures whereas delocalized channels involve heat generation accompanied with thermo-induced stress. Point defects and structural changes are restricted to the focal volume and its immediate vicinity while thermomechanical events result in a material alteration that can extend well beyond the energy deposition region. The kinetics of thermo-induced phenomena are governed by the magnitude of the heat diffusivity coefficient *Dth*. On-axis, the temperature evolution *ΔT(t)* follows (see Sakakura et al., 2007):

$$\Delta T(t) = \frac{(w_{th}/2)^2 T_0}{(w_{th}/2)^2 + 4 D_{th} t} , \qquad (1)$$

where (w_{th} / 2) is the radius of the laser-heated volume (about 0.6 - 1 µm in our case).

In fused silica, D_{th} ~ 0.75 $\mu m^2 \mu s^{-1}$, involving cooling dynamics on the microsecond time scale. Figure 1 shows a snapshot of the laser-induced microdot formation 3.8 µs after the laser excitation acquired in positive phase-contrast microscopy (PCM). Phase-contrast microscopy translates refractive index variations Δ*n* into intensity variations. In positive PCM, the intensity on the detector is high/low for negative/positive-refractive index variations. For instance, the dark region visible around the optical axis (labelled 2 in Fig. 1) indicates the presence of a zone of high refractive index. We associate this feature to heat dissipation inside the sample. This is consistent with conventional models which predict a positive thermooptical coefficient (i.e. *dn/dT > 0)* for fused silica (see for instance Ghosh, 1995). Additionally, on each side of this dark regions, a region characterized by a refractive index weaker than the refractive index of the pristine sample appears (region 1 in Fig. 1). The origin of this transient feature is not clear. However, Fig. 1 shows clearly that the laser-affected zone exceeds by far the volume of energy deposition. Here, the energy is deposited in the vicinity of the optical axis but the energy dissipation affects the material over a cylindrical volume extending up to 10 µm from the optical axis.

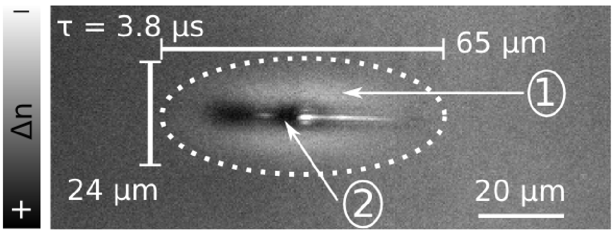

Fig. 1: Time-resolved phase contrast microscopy picture of a microdot 3.8 µs after laser excitation. The laser pulse comes from the left side of the picture. The laser pulse energy was 2.5 µJ.

3.2. Refractive index estimate

Conventional phase-contrast microscopy maps a refractive index variation *Δn* to an intensity variation on the detector. Unfortunately the mapping is not linear, making it impossible to retrieve Δn quantitatively with conventional phase contrast microscopy alone. Therefore, we characterized the refractive index profile of the microdot with a quantitative imaging method called SLIM, and found that the magnitude of Δn is as high as -3% in the region where $\Delta n < 0$ (see Fig. 2). The filamentary part of the microdot (where $\Delta n > 0$) exhibits a refractive index variation of 0.6 %. The original SLIM pictures can be found in Mermillod-Blondin et al., 2014.

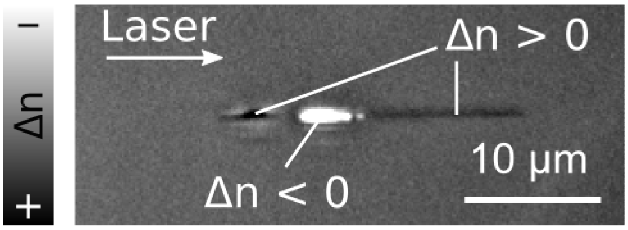

Fig. 2: Phase contrast microscopy picture of a microdot generated by focusing a single laser pulse (N=1) with a 2.5 μJ energy. The laser was incident from the left side of the picture. SLIM measurements revealed that the magnitude of Δn<0 was about 3% and 0.6 % where Δn>0.

3.3. Remaining stress

Polarization-microscopy pictures showing the distribution of the stress-induced birefringence as the number of laser pulses increases are presented in Fig. 3.

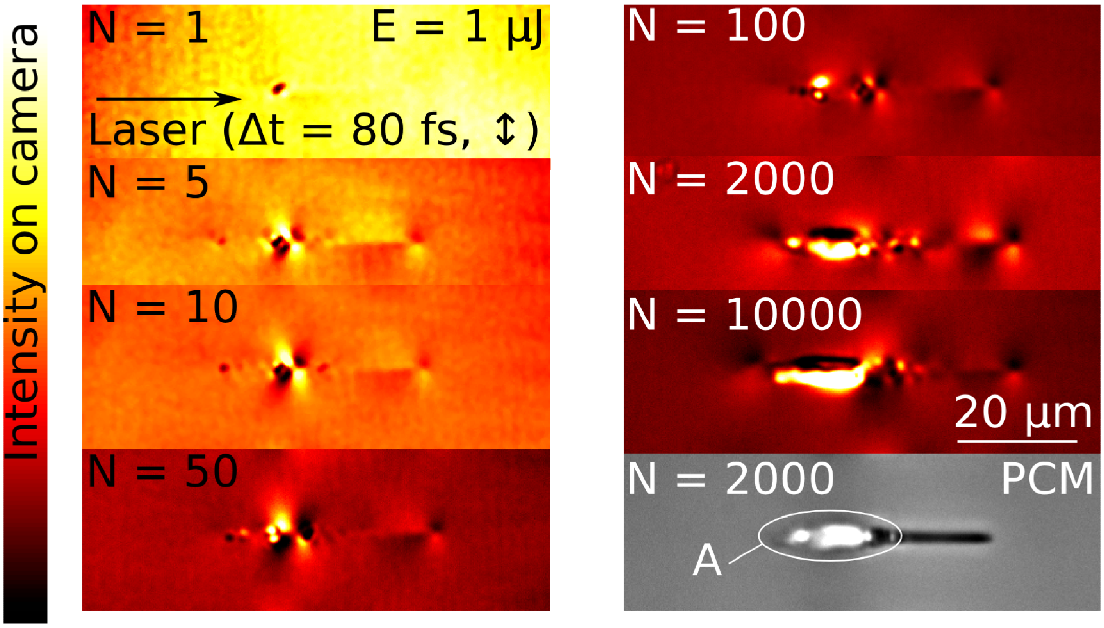

Fig. 3: Distribution of the laser-induced stress around the microdot region for number of pulses N ranging from 1 to 10000. A phase-contrast picture(PCM) is also provided.

The birefringence observed in region A is due to the form birefringence of self-
ordered nanoplanes which appear upon laser pulse accumulation (Taylor et al., 2008). Therefore, it does not indicate the presence of stress. Stress-induced birefringence is clearly visible in the vicinity of the optical axis and all around the region of energy deposition. It grows with the number of pulses N. Remarkably, we could not detect any stress-related signal for N=1. Additionally, Fig. 3 shows that the laser-induced stress distribution extends preferentially in a direction transverse to the laser propagation. For N > 1000, the stress-induced birefringence spans over distances of several microns on each side of the optical axis.

The current setup does not enable a quantitative measurement. In order to do so, a quarter waveplate must be inserted before the second polarizer according to the experimental scheme developed by De Senarmont and Friedel.

4. Conclusion

Fs-laser induced microdot formation involves heat dissipation and subsequent thermomechanical effects. As a result, a permanent stress distribution remains around the region of energy deposition.

The heat affected zone extends up to 10 µm away from the optical axis. The resulting microdot is characterized by a refractive index difference with respect to the pristine material that can reach -0.03. This high value provides a good contrast which is important regarding readability issues. The amount of stress left around the microdot depends strongly of N, the number of laser-pulses accumulated. The remaining stress seems to be very modest in the single pulse photoinscription regime (N=1) even at a laser-pulse energy of 2.5 µJ.

Acknowledgements

This study was funded through the project “16891 BG – microdots”, financed by the Forschungsvereinigung Feinmechanik Optik und Medizintechnik e.V. (FOM) through the Arbeitsgemeinschaft industrieller Forschung (AiF) in the frame of the program “Förderung der industriellen Gemeinschaftsforschung (IGF)” from the ministry for economy and technology (BMWi), in application of a decision from the German parliament.

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