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Mechanical properties of ultrafast-laser cut poly(lactic acid) films

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Abstract

The use of ultrashort laser pulses to cut polymers for medical applications has important advantages. Heat transmission to the region surrounding the cuts is limited, so that the cuts are precise and the effects on the regions around the cuts are small; in this way, the need for post-processing is reduced and ultrashort-pulse laser cutting becomes interesting for industrial applications. In general, both cutting speed and heat effects increase with the energy of the pulses and the repetition rate of the laser; it is important to identify process parameters with which polymer samples can be cut quickly and without compromising the chemical and mechanical properties of the polymer. In this work we present measurements of mechanical properties (elastic modulus, ultimate tensile strength and elongation at break) of cut samples of films of poly(lactic acid) – a biodegradable polymer with many different medical applications – as a function of laser repetition rate and examine how mechanical properties are correlated with the width of the heat-affected zones that can be observed with an optical microscope.

Keywords: Micro-Cutting; Ultrafast laser sources; Processing of transparent materials; Polymers

1. Introduction

Ultrashort laser pulses are attractive for the machining of polymers for medical applications. Heat transmission to the region surrounding the cuts is limited, so that melting, burring and other heat effects on the regions around the cuts are small; the need for post-processing is reduced with respect to other cutting

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technologies and ultrashort-pulse laser cutting becomes interesting for industrial applications. In general, both cutting speed and heat effects increase with the energy of the pulses and the repetition rate of the laser; it is important to identify process parameters with which polymer samples can be cut quickly and without compromising the chemical and mechanical properties of the polymer (Matylitsky et al. 2013).

In this study we examine the quality of cuts machined on a 50-µm poly(lactic acid) (PLA) film, a bio-absorbable polymer with numerous medical applications (Ikada et al. 2010), using a Spirit femtosecond laser system (Spectra Physics Rankweil). We approach the problem in two steps. In the first step, we perform a series of cuts on the PLA film with varying pulse energy and laser repetition rate and we examine them with a light microscope to determine the presence of heat-affected zones. With this characterization we can identify an area of "good" parameters for PLA film processing in which the visual appearance of the polymer does not change. In the second step, we select a few sets of representative machining parameters and we measure the mechanical properties of the polymer next to the cut.

2. Material and experiments

For our experiments we use 50-µm thick sheets of PLA purchased from Goodfellow (reference ME331050). Characterization of the films has been performed at CNR in Catania, Italy (courtesy of Paola Rizzarelli); GPC chromatography gave a value of 40 KDa for the number-averaged molecular weight M_n and 172 kDa for the weight-averaged molecular weight M_w and proton NMR showed the presence of a small quantity of polycaprolactone (2.8 %).

Machining was performed with the Spirit femtosecond laser system integrated in a MicroStruct Vario machining center (Micromac 3d) operating at the second-harmonic wavelength of 520 nm. The length of the laser pulses is about 350 fs and the maximum energy is about 7.8 µJ; the repetition rate of the laser is set at 200 kHz and pulses at adjustable repetition rates can be obtained with the use of an acusto-optical pulse picker. The laser pulses are conveyed to a cutting head (PRECITEC) with which a gas flow can be directed towards the cutting area; the optics of the cutting head focuses the beam to a width of 5 µm.

The pulse energy for all experiments is fixed at the maximum energy of 7.8 µJ.

In the first set of experiments we performed parallel cuts at 1.5 mm of distance between each other at varying laser repetition rates (from 5 to 50 kHz) and adjusting the beam movement speed to obtain pulse-to-pulse distances varying from 0.01 to 0.5 µm; the PLA film is supported during cutting so that the cut area is free-standing. For this first experiment set, we applied a flow of air through the cutting head; air-assistance allows us to increase the maximum speed at which cutting can be achieved with minimal heat effects.

We then observed all of the cuts with an optical microscope and classified them according to the presence of heat-affected zones (HAZs).

In the second set of experiments, we cut oar-shaped samples for mechanical testing according to a design found in the scientific literature (Stepak et al., 2014). The sample dimensions, which do not conform to standards, allow us to measure the effect of the HAZs on the mechanical properties of the polymer as suggested by Stepak et al. 2014; the measurement basis were 12.0 mm and we chose nominal widths of 1.0, 0.5 and 0.3 mm (Fig. 1); we present in this proceedings contribution mostly the results we obtained with the 1-mm thick samples and we postpone the discussion of 0.5 and 0.3-mm thick samples to a next publication. For this second experiment set we did not use an air flow, to make it easier to machine cuts at small distances from each other without distortions.

We performed tensile tests with a zwicki-Line 500 N (Zwick-Roell) testing machine equipped with a XForce HP load cell with a nominal maximum load of 500 N; the test speed was 1 mm/s for small strains (in the elastic response region) and 8 mm/s for larger strains (outside the elastic response region) . We measured Young's modulus, elongation at yield and at break, maximum tensile stress and stress at break; as an extensometer was not available, we measured the extension of the sample using the crosshead movement – the validity of our measurements is therefore limited to comparisons between samples of the same nominal dimensions.

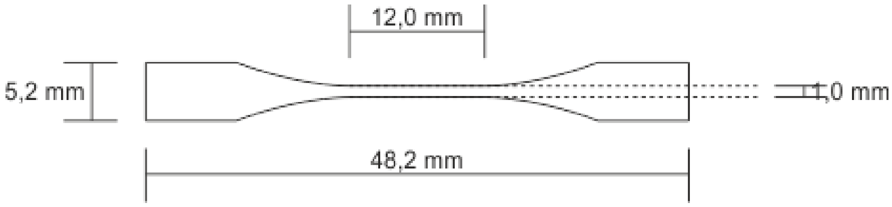


Fig. 1. 1-mm thick mechanical test sample

3. Results and discussion

3.1. Visual examination of cuts

Typical cuts with very small and with relatively larger heat effects can be seen in Fig. 2, left (sample cut at 5 kHz and 0.02 µm pulse separation) and Fig.2, right (cut at 25 kHz with 0.5 µm pulse separation), respectively. Note that for our range of parameters melting – when it appears - can only be seen on the back side of the cut polymer film (represented in Fig. 2, left) and most of the heat effects consist in burring. In Fig. 2, left the measurements represent the typical size of the few burr grains generated with this parameter set.

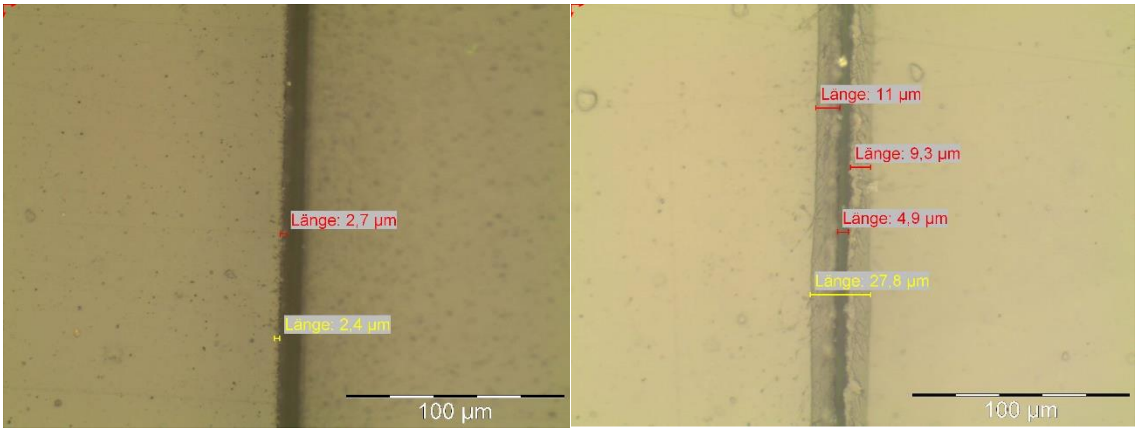


Fig. 2. Left, Typical cuts with minimal heat effects (no heat effects are observed either on the top or on the bottom side of the sample, top is depicted); right, with a melting zone (heat effects are observed on the bottom side of the sample only, bottom side depicted)

According to the visual examination we classified the machining parameters in two categories: parameters which cause and which do not cause melting; we represent them respectively with red and green squares in the graph of Fig. 3. The amount of burr decreases with decreasing pulse-to-pulse distance until just a few isolated grains can be found. The presence of a melted zone at high pulse-to-pulse separation does not have an immediate explanation and more investigation is needed on this point.

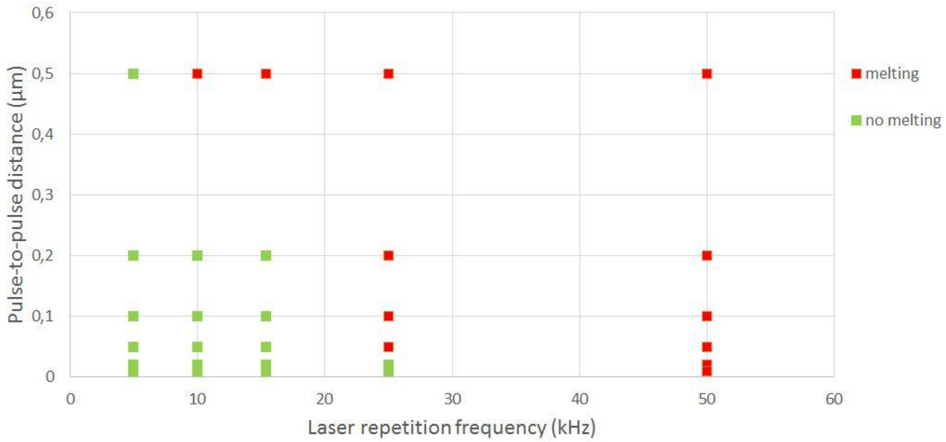
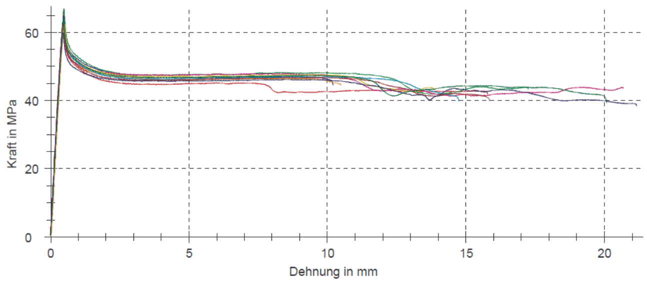


Fig. 3. Classification of cuts according to visual heat effects

3.2. Mechanical testing

We cut the samples for mechanical testing without air assistance, as in this way it is easier to obtain undistorted cuts at small relative separations. We prepared samples with small/negligible heat effects cutting at 2 kHz repetition rate with a pulse-to-pulse separation of 0.1 µm, and samples with large heat effects with 50 kHz and 0.02 µm.



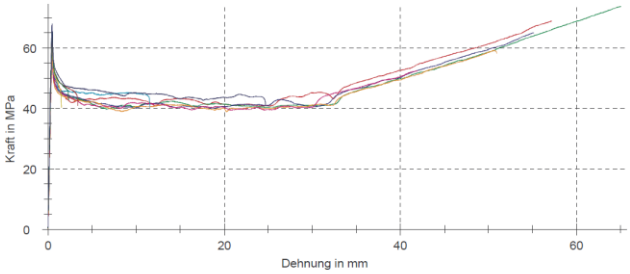
Statistik:

Serie n = 10	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	4900	63,4	63,4	1,4	41,8	48,5	0,05	0,995	0,05
s	424	2,28	2,28	0,0	2,27	11,8	0,000	0,000	0,00
ν [%]	8,66	3,59	3,59	3,27	5,42	24,35	0,00	0,00	0,00

Fig 4. Results of tensile tests on 2 kHz-cut samples

All of the samples cut at 2 kHz (without heat effects) show ductile failure (Fig. 4).

Tensile testing on 1-mm wide samples cut at 50 kHz, in which the total extent of the HAZs is approximately 0.3 mm, (Fig. 5) shows that their failure mode is also ductile. The “cold drawing” part of the strain-stress diagram (present in only some of the samples) might not depend on the presence of the HAZs as it could be reproduced on samples without HAZs cut on the same day in which the 50 kHz-cut samples were tested. The ductile failure mode of 50 kHz-cut samples is found again in the tests of .3 mm-wide samples, in which the HAZs cover the entire or almost the entire width of the sample. Images of a 50 kHz-cut sample after testing (Fig. 6, chosen among the samples which are not cold-drawn) shows that less crazes develop in the HAZs as in the visually pristine material.



Statistik:

Serie n = 10	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	5000	63,9	66,3	69,9	52,3	95,0	0,05	0,9228	0,05
s	205	3,45	3,99	89,0	13,2	76,9	0,000	0,007955	0,00
ν [%]	4,10	5,40	6,02	127,30	25,26	80,99	0,00	0,86	0,86

Fig 5. Results of tensile tests on 50 kHz-cut samples

Both Young modulus and stress at yield are equal for samples with HAZ and without HAZ within experimental error (note that the absolute values of the Young modulus cannot be trusted due to the measurement of the elongation through the crosshead movement; the comparison between different measurements are on the opposite valid).

For PLA ductile failure is associated with the amorphous state, and brittle failure is associated with crystallinity (Perego et al., 1996, Renouf-Glauser et al., 2005). Calorimetry (measured at the Interstaatliche Hoschschule für Technik Buchs, courtesy of Ramona Bernet and Dietmar Bertsch) show that our polymer films are amorphous (fusion enthalpy is about 3 J/g, very small compared to the fusion enthalpy of theoretically 100% crystalline material which is equal to 93 J/g, as referenced by Gámez-Pérez et al., 2011) both before and after machining (the calorimetry data after machining are measured on a sample where a set of cuts machined at 200 kHz was arranged in such a way that the HAZs covered the entire sample surface).

4. Summary

We investigated ultrashort-pulsed laser cutting of poly(lactic acid) films using visual examinations at the optical microscope, mechanical testing, and calorimetry data. Our tests show that there exist a large region of machining parameters that do not generate visual heat effects during laser cutting. The pristine polymer is amorphous and ductile under tensile testing, the ductility is preserved after laser cutting. The first tests on heat-affected zones do not show large effects on the mechanical properties of the material.

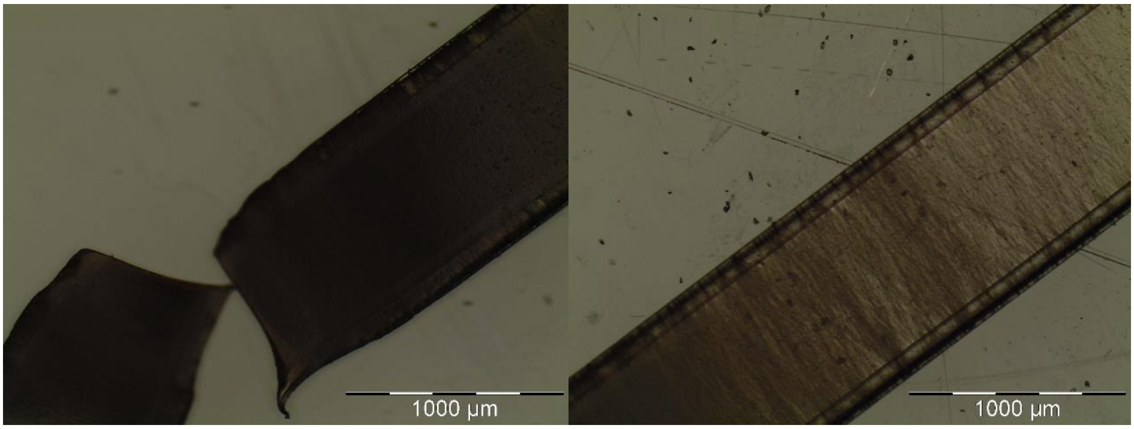


Fig 6. 50 kHz-cut sample. Left: breaking site; right: at some distance from the breaking site

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