Micromachining of Materials using Femtosecond Laser Pulses A Parametric Study

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1 RESEARCH PROBLEM

In the presented research, I have undertaken the problem of femtosecond laser interactions with matter, under various laser pulse parameters and considering a wide group of materials, i.e. metals, plastics, graphene, silicon. There is a considerable interest in the precise material micro and nanomachining with ultrashort laser pulses. It is well established, that the quality of ablation process with femtosecond laser is much better than when using long pulse lasers. The use of femtosecond laser pulses creates an attractive opportunity for high quality micromachining of many groups of materials and opens an interesting field in fundamental research. The objective of the fundamental research proposed within this research is a complex study of the physical phenomena occurring during shortpulsed (femtosecond) laser interactions with matter. Although the research topic itself is very popular in the scientific community, it is relatively little elucidated. A better examination of the effects of electromagnetic irradiation on the materials in the femtosecond regime will provide a better understanding of the subject. The experimental results obtained from this project and theoretical considerations will provide a verification of the existing theories on laser-matter interactions. Additionally, the experimental investigations will be carried out on novel materials, i.e. graphene.

I have specifically taken under investigation the laser ablation phenomena, which occurs during a highly energetic laser irradiation of the material and results in detachment of microparticles from the material. The physics of the ablation process in an ultrashort laser pulses regime differs from the ablation caused by the long (nanosecond and picoseconds) laser pulses. The nature of the process is more complex with the short pulses and the complete, research based understanding of the phenomena is not available yet.

2 STATE OF THE ART

Since the development of mode-locked lasers, ultrashort pulse durations became available, allowing measurements in the femtosecond range. The next significant advance in laser technology was the development of chirped pulse amplification (CPA) technique (Strickland and Mourou, 1985; Mourou, 1997). Pulse energies in the mJ range are easily obtained via CPA. The CPA technique also allows the pulse duration to be varied from a few femtoseconds to even nanoseconds, while keeping all other beam parameters constant. Through this technique, (Liu et al., 1997), studied the ablation dynamics with pulse width varying from 10 ns to 100 fs and observed a decrease in the ablation threshold with decreasing pulse duration. However, the details of the physical mechanisms behind femtosecond laser ablation were still far from complete understanding.

A numerous groups studied the ablation processes analytically and numerically. Many models have been proposed to explain various aspects of the femtosecond ablation process including: ultrafast laser pulse absorption by solid targets, femtosecond heating, expansion, stress generation, defect capture and formation of periodic surface structures on surfaces (Anisimov et al., 1999; Peterlongo et al. 1994, Emelyanov and Babak, 2002).

The experimental and theoretical investigations lead to some improvement in the physical understanding of the ultrafast laser ablation process. The dynamics of the ablation process can be roughly divided into several stages: energy absorption, energy transfer to the lattice and subsequent material removal. The first step of the ablation process is deposition of energy into the material. The primary absorption mechanism involves excitation of electrons from the valance to the conduction band and free carrier absorption. The interband excitation can occur through nonlinear processes, such as multiphoton and avalanche ionization, with high enough laser intensity. Nonlinear absorption is very important in femtosecond interaction due to the high intensity of the incoming radiation (Sokolowski-Tinten and Von der Linde 2000). During the lasermater interaction all of the processes occur simultaneously and it is difficult to estimate the contribution of each one. Due to the complexity of the process, it is also difficult to calculate or measure the effective penetration depth of the radiation.

The energy transfer from electrons to the lattice occurs via carrier-phonon scattering on a timescale estimated from several hundred femtoseconds to a few picoseconds, depending on the material. Since the electrons and lattice are not in equilibrium, this situation is often described by a two temperature model, where a distinction is made between the electron and the lattice temperature (Chichkov et al, 1996). The energy transferred to the lattice leads to rapid thermal or nonthermal melting (Tom et al., 1988). Since the timescale for mass transport is significantly longer than for non-thermal or even thermal melting, the melted material is left at near solid state densities and a high initial temperature. The subsequent processes of material removal have been described in terms of transient thermal processes. Following melting, the hydrodynamic expansion of the ablated material begins a few 100 ps after the initial excitation (Sokolowski-Tinten et al., 1998). In spite of numerous investigations the fundamental mechanisms leading to the material removal are still rather poorly understood. Several different ablation mechanisms were identified in theoretical investigations including: spallation, explosive boiling and vaporization (Zhigilei and Garrison, 2000; Perez and Lewis, 2002; Shafer et al., 2002, Ivanov and Zhigilei, 2003).

Spallation occurs at a fluence slightly exceeding the ablation threshold, and refers to ejection of a complete layer of material induced by material fracture due to internal stress buildup brought on by constant volume heating. At a higher fluence, or in materials where spallation might not apply, the expansion of material can occur through phase explosion. In phase explosion, the melted material enters a liquid-gas metastable state during expansion and homogenous nucleation of gas bubbles sets in, leading to formation of a heterogeneous phase of gas and liquid droplets. Phase explosion is believed to be the primary mechanism in femtosecond ablation below the threshold for plasma formation (Perez and Lewis, 2003). At a high enough excitation fluence the surface layer of the material can be completely

atomized and material removal proceeds by process vaporization.

The entire ablation process occurs on time scales of several tens ns. Ablation experiments are usually performed with laser beams that have a near Gaussian spatial profile, therefore energy deposition varies across sample surface.

The ablation process mechanisms briefly described above depends, on the one hand, on the laser radiation parameters, such as pulse duration, wavelength, pulse energy, repetition rate or irradiation time, and on the other hand – material properties, i.e. absorption coefficient or thermal conductivity (Chichkov et al, 1996).

In laser – irradiated material sample, a various features, such as crater profiles, ablated volume, local changes in crystallography and chemistry, surface modifications can be related to various dynamical mechanisms and the ablation threshold and ablation rates can be readily obtained from the analysis of the final state of material. Together with a detailed observation of the laser generated plasma plume during ablation, the results of the proposed research project will provide a complex understanding of the laser-matter interaction mechanisms in the ultrashort pulse region.

3 OUTLINE OF OBJECTIVES

As previously mentioned, the physics of laser ablation is strongly dependant on the material type and is still an object of interest of many research groups worldwide. It has been established, that the character of the physical mechanisms occurring during the laser ablation is different for the typical, often investigated materials, such as plastics or thin metal foils, and different in case of specific or novel materials, such as graphene or silicon. With these non-typical materials, the character of laser-matter interactions doesn't the theoretical match explanations found in the literature.

Exploring the physical mechanisms during laser irradiation is crucial for the full understanding of the laser ablation phenomena. In the ultrashort pulse region, the most significant mechanisms are: liquid phase explosion due to the heterogenic and homogenic heating, due to the subsurface heating and the ablation plasma interactions with the material surface in so called Knudsen layer. Apart from the liquid phase ablation, the phenomena can also occur through direct sublimation. In that case, the most important ablation mechanisms are: spallation, fragmentation, charge separation due to avalanche and multiphoton ionization and coulomb explosion. The contribution of these particular mechanisms depends on the laser radiation parameters and material properties. Because of the complexity of the ablation process, a complete theory describing the laser-matter interactions in ultrashort pulse region has not been developed yet.

The results of this research project will provide a deeper knowledge about the contribution of the different ablation mechanisms in the ablation process and their dependence on laser radiation parameters, which will contribute in a coherent theory of ultrashort pulse laser-matter interactions. We will also expand the existing knowledge base with novel materials. The conditions of the ablation process, such as pulse energy, wavelength or repetition rate need to be determined separetly for each of the investigated materials. The main difference from the nanosecond ablation sums up in a fact, that the radiation energy absorbed by the material remains stored in a very shallow layer, predefined by the optical absorptive properties (Chichkov et al, 1996; Dowden 2009).

Femtosecond lasers of high repetition rates have not yet been fully examined for interactions with materials. One of the experiments were carried out by [17], taking into consideration a few significant parameters at once, i.e. pulse repetition rate, limited to megahertz range, at an average laser power level and fixed wavelength. During these studies, numerous problems have appeared, such as instability of the laser pulse energy and power fluctuations. Although the fiber solid state laser was used, so far the researchers have failed to take full advantage of CPA amplified lasers laser-matter interactions investigations, mostly because of the low repetition rates used (Dowden 2009; Tan and Dalili, 2009).

A femtosecond laser in our laboratory, not only provides the opportunity to achieve a wide range of repetition rates, but also can offer a stable output power at different levels. Very strong focusing of the laser beam by an advanced optical system will provide a high pulse energy and will eliminate the problem of power fluctuations. Furthermore, the possibility of generating three different wavelengths of UV, VIS, IR range will allow to perform the research using a single laser, while the other radiation parameters will remind constant. Whereas most of the short pulse lasers applied in the research offers a pulse duration in the range of several tens of femtoseconds, our laser generates 500 fs pulses, what places it in the subpicosecond range, a range which we find a far more interesting from the scientific point of view. Working with a several hundred femtosecond pulses creates an unique opportunity to observes the ablation mechanisms in the border area between long and ultrashort laser pulses.

4 METHODOLOGY

The laser micromachining of materials will be carried out using a prototype diode-pumped Yb:KYW fiber laser, developed within a research project by prof. Radzewicz's Laser Centre group from Institute of Physical Chemistry PAS (Fig. 1). The use of fiber laser technology among other femtosecond laser technologies is very beneficial. No thermal problems within the active medium occur, high average power and temporal stability is obtained.



Figure 1: Yb:KYW laser construction.

The source of a high power femtosecond pulses in our laser is the Yb:KYW crystal-based oscillator with a z-type resonator. A passive mode-locking technique is used for pulse synchronization, which is implemented with a semiconductor saturable absorber mirror (SESAM). In a SESAM-type construction the absorber mirror is a periodic structure (diffraction grating), reflecting light due to Bragg diffraction phenomenon. The Yb:KYW crystal is pumped with a single-mode laser diode with a 980 nm wavelength. The fundamental wavelength of the output beam generated by the oscillator is 1030 nm (3 nm FWHM), but also second (515 nm) and third (343 nm) harmonic can be generated. From the oscillator output, femtosecond pulses go directly to the amplifier. Due to the high peak power values in a single pulse, significant damages of the optical elements can be caused during the amplification process. Therefore, chirped pulse amplification (CPA) technique needs to be applied. Stretched pulses pass through the electro-optic modulator (Pockels cell) that reduces the repetition frequency. This allows us to modulate pulse repetition rate from kilo to megahertz range. The typical operating range of the laser is 100 - 900

kHz. At the Pockels cell output, the beam is amplified in two stages, which provides a better efficiency of the pumping process. The photonic fiber with a large diameter ytterbium doped core is used in both stages. Laser diode pumping to the core is continuous at a wavelength of 976 nm. At maximum power of the laser diode we obtain the amplified beam power at the level of 50 W in the fundamental beam. M² factor, an important measure of beam quality is at maximum power, 1.27. These parameters are proved to be very promising in the laser material processing. The output pulse length of the laser is 500 fs, which is close to the so-called subpicosecond region, which opens a new, interesting field of research for the short-pulsed laser-matter interactions.

From the laser output, the beam is passing through the optical collimator and focusing system, containing of mirrors and lenses adequate for the applied laser wavelength. Microscopic lenses can be applied to obtain a higher laser fluence. The concept of the micromachining implies, that the output femtosecond laser beam, after passing through the optical collimator, will be directed into the positioning system. To move and focus the UV laser beam on the surface of the workpiece, an optical scanner with focusing lens is used. The scanner is equipped with two galvanometric mirrors that deflect the laser beam, making it possible to move according to a given pattern in the XY plane. A dedicated control software, supporting HPGL files is provided to create patterns. A telecentric lens is used to focus the beam as it provides a uniform beam interaction with material in the whole scanning area. The samples are put on the z-axis adjustable table, directly beneath the scanning area, on the exact level of the laser beam focus (Fig.2).



Figure 2: The experimental setup.

A Yb:KYW femtosecond laser offers high repetition rates and stable power level, as long as the possibility of generating three different wavelengths of UV, VIS, IR range, which allows the micromachining processes at full spectrum of laser radiation. For the IR and VIS experiments, the galvo-scanner cannot be applied. Instead, two high-precision linear motors are provided to move the sample in the XY plane and a separate focusing optics, dedicated for the given wavelength range, is used.

The presented micromachining setup provides a 2 μ m micromachining accuracy and allows to perform a wide variety of the machining processes: cutting, scribing, engraving, structuring, drilling and dicing.

The laser, together with the optical focusing system and high precision positioning setup are the most important tools to achieve our research goals and have already proven a very high-quality of the results.

Evaluation of the experiment results is based mostly on the microscopic images. The stereoscopic metallographic microscope to 200x(up magnification) and Carl Zeiss Scanning Electron Microscope (magnification up to 1000x) are used for crater dimensions, line width, HAZ range and derbis measurement for most of the materials. These tools are adequate, but not sufficient for the detailed sample analysis, especially when it comes to transparent or nonconductive materials, the exact crater depth measurements or investigating changes in the inner structure of the material. Additionally, a confocal laser Olympus LEXT microscope or the Optical Coherence Tomography System (OCT) is used. The confocal laser microscope is a powerful tool for sample diagnostics with $a < 1 \mu m$ resolution, performing non-contact 3D observations and a fast image acquisition.

The OCT system I am using, was build and designed at the faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology. This diagnostics tool is available to us due to the collaboration with the GUT's Optoelectronic department. This is still a rare used and an innovative approach to research methodology, when it comes to laser-matter interaction diagnostics. The microscopic images



Figure 3: A PS-OCT image of the microcraters after Nd:YAG nanosecond laser micromachining of alundum ceramics.

allows the observation of the surface modifications, roughness or derbis, as well as crater size, line width or aspect ratio of the material. Using the OCT technique, it is also possible to cross-examine the material, thus resulting changes in its structure at the full penetration depth of laser radiation (Fig. 3).

5 STAGE OF THE RESEARCH

On an early stage of the research, laser beam characteristics were carefully measured, in order to ensure the best knowledge of the laser beam parameter range, accuracy and stability and to provide the maximum quality of the laser micromachining processes. During this experiments, spatial and temporal characteristics of both, focused and unfocused laser beam were examined, as long as the Rayleigh range and minimum spot diameter. These values are crucial for accuracy of the further experiments. The stability of the spatial position of the laser beam will was determined. In order to perform the parameter examination, a Spiricon SP620U beam analyzer was used, together with a set of adequate filters and lenses. The output power level and the temporal power stability of the laser beam was measured using Thorlabs PM200 power meter with a S350C probe (measurement range: 10mW - 40W). Additionally, a spectroscopic study of the laser beam was performed. The spectral characteristics of the femtosecond laser beam were measured using MAYA11734 spectrometer and Andor Mechelle spectrometer. To maintain the optimal laser parameters, measurements were taken on the laser output and inside the laser, on the oscillator output, with the fundamental beam and both of the harmonics.

The most important aim of the research is to investigate femtosecond laser interactions with a variety of materials under a wide range of laser irradiation parameters, and to determine laser ablation mechanisms. Thus, the next step in the work plan is irradiating the material samples with single and multiple laser pulses with different variables, such us: pulse energy (laser output power is up to 50 W in the fundamental beam), pulse repetition rate (100 kHz to 900 kHz), wavelength (1030 nm / 515 nm / 343 nm), irradiation time, etc. All the experiments are to be carried out with a single femtosecond laser (detailed description can be found in the methodology chapter), which ensures a complexity of the research and guarantees constancy of the invariable parameters in each step.

As a reference, I also plan to use a long pulsed (nanosecond) laser, available in our laboratory and a short pulsed femtosecond laser from the Institute of Solid State Physics, available through our research collaboration with the Bulgarian Academy of Sciences. Especially the latter can bring a significant contribution to the research, providing a 35 fs and 120 fs pulse width and low repetition rates (1 - 10 kHz), with a wavelength in the close IR range.

So far, the laser micromachining experiments were carried out in the UV and IR range. For the 343 nm beam, a various scanning velocities, number of repetitions, output power levels and different radiation time were tested, while performing cutting, scribing and drilling. For the 1030 nm, laser scribing and drilling was examined with variable power, repetition rate and radiation time. Not only the laser radiation parameters, but also material properties have strong effect on laser micromachining and ablation mechanisms, therefore all of the experiments were carried out on metals (stainless steel with different surface finish, aluminum of various thickness, nickel, copper, bronze, zinc, titanium) and plastics (PTFE, polypropylene, polycarbonate, acetal, nylon). A closer attention was given to silicon wafers, graphene on copper and ceramic pieces.



Figure 4a: Si wafers sample after laser scribing, images from the confocal Olympus LEXT microscope.



Figure 4b: Si wafers sample after laser cutting, images from the Zeiss SEM microscope.

Pre diagnostics of the of the micromachining results was performed using the metallographic microscope. The crater diameter, depth and aspect ratio, the heat affected zone range and debris were measured. For more advanced result analysis, the confocal laser microscope, SEM and OCT will be used. These techniques will provide the ability to measure such features, as: measurement of transparent layers, micro roughness profiles, sample thickness or measurements of micro areas and surface analysis with high resolution. The results for some of the measurement methods are represented by a silicon sample after laser cutting on Fig.4.

With the collected data, the laser fluence [J/cm²] is calculated for each experiment and the dependence of crater measurements, HAZ, line width, etc. on laser and material parameters is investigated.

The effects of the laser pulse parameters on ablation threshold, heat accumulation, ablation efficiency, cold and hot ablation mechanisms and the amount of liquid phase occurring during the laser irradiation will be studied for materials mentioned above. The specific material parameters, like absorption rate or heat conductivity will be included as a significant variables. The results will be discussed against theoretical models and other experimental results, in order to determine most probable physical phenomena during laser-matter interactions for short pulse irradiation.

For the complex understanding of the laser

ablation mechanism, the next work plan step is an investigation of the laser generated plasma plume. The plasma plume consists of particles, atoms, clusters, ions and electrons, detached from the material surface during laser irradiation. A timeresolved spectroscopic study of the ablation plasma and the dynamics of the expansion of ablation plasma will be performed. The spectroscopic study will be carried out with a Mechelle spectrometer, whereas the expansion dynamics observation will be done with an Intensified CCD (ICCD) camera with a very short time of exposure. The use of the ICCD camera will allow us to observe the formation and the evolution of the plasma with a very high temporal resolution (Fig.5).

The experimental results will provide a knowledge about the shape of the plasma plume on an early stage of development (plasma onset) and the velocity of the plasma front expansion. The spectroscopic study will allow to determine an excitation time of atoms and ions forming a plasma plume. Based on this results, an electron temperature of plasma and its ionization degree can be concluded.



Figure 5: The evolution of the laser generated plasma plume in ambient air at the early stage of its expansion. Images recorded using an ICCD camera.

6 EXPECTED OUTCOME

The research results will contribute to the deeper understanding of the laser-matter interaction theory. I believe that the overall results obtained in this research will enhance the knowledge of fundamental phenomena in scientific field of interaction of laser pulses with matter in ultrashort timescales, in particular of the ablation mechanisms that take place in this regime. Based on the experimental results it will be possible to find the answers to various problems in this field that are still opened for academic discussion. I expect that those result will allow to verify the existing models of ultrafast lasermatter interactions and extend its applicability for

some materials, which will consequently lead to progress in the field of micro and nanotechnology. At present, the potential of creating micro and nanoscale layers and elements is not fully used and the lack of knowledge about the interactions of the electromagnetic radiation with many types of technologically significant materials is one of the reasons for this. The practical application of femtosecond micromachining opens the door to an entirely new generation of micro device development, such as MEMS (micro-electromechanical sensors) or Lab-on-a-chip (size of a credit card micro-chemical laboratory - the latest trend in medical diagnostics). There is also a number of candidates for the application area which might drive femtosecond laser technology into mainstream industrial use. Some of the sectors include biomedical devices (stent manufacturing), microoptics (micro-lenses, diffractive elements) and (optical photonics devices waveguides, telecommunications devices). Non-systemized knowledge of the optimal micromachining parameters for a specific material and machining type (cutting, dicing, engraving, etc.), makes implementing to the industrial level very difficult and ineffective and solving this problems might be one of the outcomes of the proposed research.

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