Lasers in Manufacturing Conference 2017

Investigations on ultra-short pulse laser processing of ceramics using statistical methods

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Abstract

Since ultra-short pulse lasers have been commercially available, they have proven to be a perfect tool for micro processing. The precise and gentle processing caused by an almost non-thermal ablation is suited for a wide variety of materials, even for dielectric, brittle-hard substrates such as ceramics.
However, many questions regarding the beam-material-interactions have still not been entirely clarified. In order to reveal and optimize the processes happening during surface ablation, investigations on alumina have been executed. The particular aim was to examine a large number of parameters regarding their influences on the ablation process as well as their interactions among each other. In the case of ultra-short pulse laser processing, there are, in addition to the material properties, more than ten process parameters, for instance pulse energy, wavelength or repetition rate.
In order to reduce the scope of experiments to a minimum, Design of Experiments (DoE) has been applied to 2.5D surface ablation. Subsequently, a sensitivity analysis of the experimental data has been performed to identify significant parameter correlations. To describe the ablation process in a realistic way, different meta-models of optimal prognosis were created, which have been evaluated by quality and physical validity.
The results show how the process parameters influence the removal rate and the surface characteristics, such as roughness and morphology. On the one hand, the experiments contribute to the basic process understanding, for example how the surface evolves as the material deepens or how the threshold fluence shifts with increasing scanning repetitions. On the other hand, an optimization of the target parameters has been achieved. Whereas the removal rate has been decisively maximized by a proper selection of the line distance, the roughness could be minimized for a certain pulse overlap which strongly depends on the fluence.

Keywords: ultra-short pulses; ablation; surface structuring; DoE; ceramics

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1. Introduction

Technical ceramics such as alumina (Al_2O_3) are used for a wide range of industrial applications, for instance in electronics, mechanics or medical technology. Their special properties, such as wear resistance, stiffness and electrical neutrality, make them the ideal material for various components, but turn mechanical processing into a difficult task. For this reason, modern laser technologies, which enable a contactless and wear-free processing, have been used to process ceramics for some time now. As summarized by Samant and Dahotre, 2009 previous investigations especially focused on cutting and drilling of ceramics using CO_2 and Nd:YAG lasers in particular. However, regarding high-precision applications like three-dimensional structuring, there are still some challenges to be overcome, such as the avoidance of cracks and debris. Ultra-short pulse durations in the pico- and femtosecond range, which lead to non-linear absorption mechanisms and an almost athermal material removal, are especially suited to achieve high surface qualities, even with regard to brittle-hard, dielectric substrates. As shown by Perrie et al., 2005 micro-structures with a particularly high edge quality can be obtained in alumina using femtosecond pulses. Kim et al., 2009 as well as Chen et al., 2013 give further information about threshold fluences, surface morphologies and chemical changes referring to the one-dimensional single and multi-pulse processing of alumina and aluminum nitride.

However, regarding 2.5D surface ablation of ceramics, further investigations are necessary to determine significant parameter correlations as well as process limits. Due to the heterogeneous microstructure of ceramics, the interactions between beam and material emerge from a combination of photothermal and -chemical processes. According to Vora et al., 2012 a multitude of physical phenomena, such as melting, dissociation, plasma formation, recoil pressure and evaporation, can be responsible for the material removal. As shown in figure 1, ultra-short pulse ablation is influenced by a variety of parameters. Besides the material properties and the environmental conditions, the laser parameters as well as the scanner movement influence the process mainly. Nevertheless, it often remains uncertain which of the geometric and energy-related variables describe the process best and how they influence each other as well as the process results. Statistical methods are a suitable means to examine such a large number of parameters simultaneously and systematically. For this reason, they have been used repeatedly in laser materials processing. With regard to ceramics, the response surface method was used by Dhupal et al., 2007 and Kibria

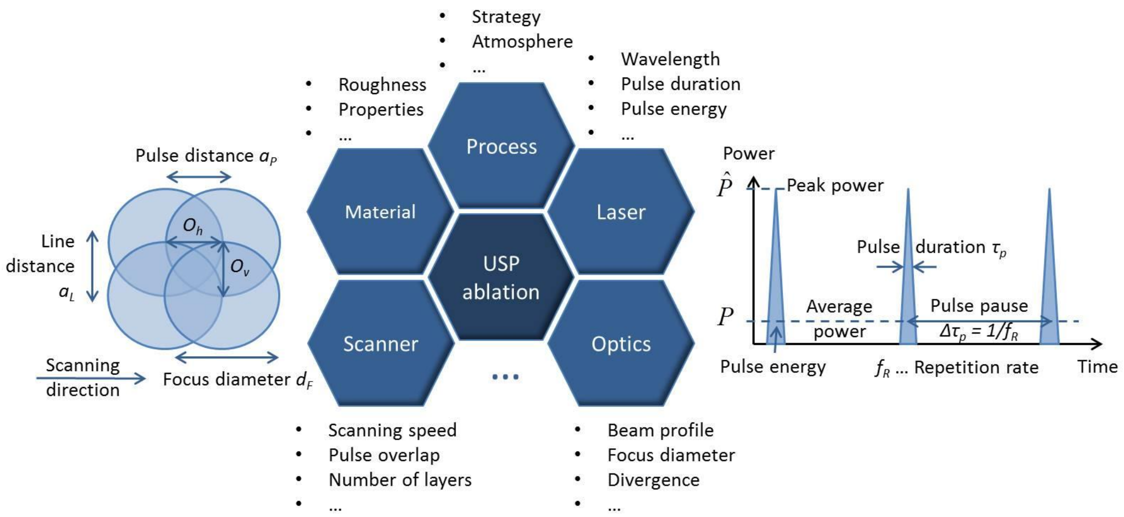

Fig. 1. Overview of parameters affecting ultra-short pulse ablation

et al., 2012 to optimize the micro-grooving of aluminum titanate and the micro-turning of alumina, respectively.

2. Statistical Methods

To investigate the 2.5D surface ablation of alumina the software *optiSLang* by *Dynardo* has been used to execute a Design of Experiments (DoE) and a subsequent sensitivity analysis. As defined by Siebertz et al., 2010 a sensitivity analysis serves to determine the correlation between the variance of input variables and the variance of output variables. The influence of the input variables on the result is identified in order to distinguish between relevant and less relevant parameters. However, existing variance based methods require huge numerical or experimental effort due to a large number of simulation runs or experiments and are not sufficiently accurate to describe multidimensional problems. Therefore, metamodels are used to compute the responses in dependence of the input variables with the help of approximation functions. There are several methods to generate metamodels such as polynomial regression, moving least squares or kriging, which minimize the deviations of the predictions compared to the true data points. As described by Roos et al., 2007 it is often not clear which method is most suitable for which problem. Therefore, *Dynardo* developed the Metamodel of Optimal Prognosis (MOP), which has been introduced by Most and Will, 2008. This selects the best combination of input parameters as well as an optimal metamodel with the help of an objective quality measure, the so-called Coefficient of Prognosis (CoP).

The approximation quality of a metamodel is usually estimated with the Coefficient of Determination (CoD, R^2). However, as shown by Most and Will, 2008, this measure behaves too optimistic for a small number of data points or an increasing number of input variables. Furthermore, it is only applicable to polynomials, which makes the use of more complex but possibly more accurate approximation methods difficult. In contrast to that, the CoP is model independent and does not over-estimate the approximation quality when the number of samples is relatively small. It is defined as follows

$$CoP = 1 - \frac{SS_E^P}{SS_T} \qquad (1)$$

with SS_E^P as the sum of squared prediction errors and SS_T as the total variation of the outputs. The errors are estimated by means of cross validation. In this procedure, the data points are divided into a training group for generating the metamodel and a test group for measuring the quality of the model. In this way, the model quality is determined only by the data points which are not used to create the approximation model. As a result of the MOP, an approximation model is obtained that contains the highest CoP for each output parameter as well as the corresponding most important variables.

3. Experimental Approach

The experiments were carried out with a mode-coupled solid state laser (Lumera Laser, Hyper Rapid 25) with a pulse duration of 9 ps and a wavelength of 1064 nm. Via external frequency conversion the second and third harmonic are generated, so that 532 nm and 355 nm are available as well. The beam, which has a Gaussian shaped intensity profile, is deflected by a galvo scanner and focused with F-Theta objectives of varying focal lengths. To remove the material, test fields of 5 mm x 5 mm have been filled with parallel scanning lines. The direction of the lines has been randomly rotated after each layer to achieve a homogeneous surface treatment. The evaluation of the ablation process is based on the output parameters profile depth and roughness, which have been measured with the help of a laser scanning microscope

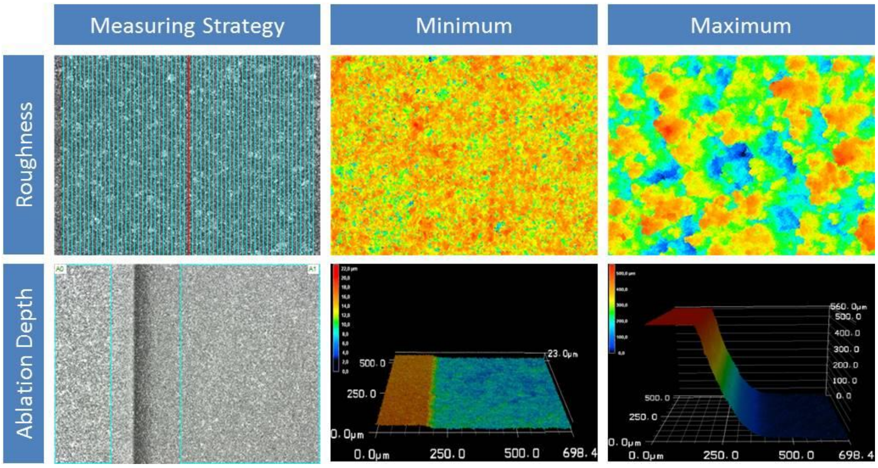

Fig. 2. Measurement of the main output parameters – roughness and ablation depth

(Keyence, VK-X100). The roughness has been determined by a multiple line scan and the profile depth by measuring the step distance between initial and processed surface, as can be seen in figure 2. To evaluate the efficiency of the process the ablation rate was calculated as the ratio of ablated volume and processing time. Furthermore, the ablation per layer has been determined as the ratio of profile depth and the number of processed layers.

Using the so-called Latin Hypercube Sampling (LHS), an experimental design with 100 parameter combinations was set up. This special DoE scheme, which has been described by McKay et al., 1979 is especially suitable for the analysis of complex nonlinear systems. The LHS is a stochastic sampling method, in which the parameter settings are spread randomly and uniformly over the whole design space within the lower and upper bounds. By means of stochastic evolution strategies undesired correlation errors between the input variables are minimized. In contrast to deterministic sampling schemes, such as full factorial or fractional factorial designs, the number of samples does not increase exponentially with increasing dimension and the input values are not limited to only two or three levels in each dimension. Therefore, the LHS can be used to obtain a maximum of information with little effort, even if nonlinear parameter correlations are considered.

Table 1. Overview of input and output parameters with corresponding value ranges

Input Parameters				Output Parameters	
actuating variables		controlled variables			
Power *P* [W]:	0.3 … 20	Fluence *F* [J/cm²]:	0.1 … 32	Roughness *Ra* [µm]:	0.42 … 3.8
Wavelength *λ* [nm]:	355; 532; 1064	Pulse distance a_P [µm]:	1 … 15	Ablation rate *A* [mm³/s]:	0 … 7.8
Scanning speed v_S [mm/s]:	200 … 3000	Horizontal overlap O_h [%]:	-33 … 99	Ablation depth *t* [µm]:	0.24 … 543
Line distance a_L [µm]:	1 … 15	Vertical overlap O_v [%]:	-33 … 99	Ablation/layer *ApL* [µm]:	0.018 … 31.5
Focal length *f* [mm]:	40; 80; 100; 250	Focus diameter d_F [µm]:	12 … 100		
Number of layers *N:*	1 … 20				

Table 1 gives an overview of the investigated input and output parameters and their value ranges. While some inputs can be changed directly by the machine settings, others result from physical connections. Within the scope of the sensitivity analysis, all listed parameters have been taken into account to identify their significance for the removal process.

4. Sensitivity Analysis

The results of the sensitivity analysis based on the MOP can be visualized with the help of a CoP-matrix as shown in figure 3. This illustrates the significance of the input variables with respect to the responses at first glance. The single indices refer to the variance contribution of the input variables and thereby show how much every input affects every output. The total CoPs quantify the quality of the approximation models. Unimportant variables are filtered by the system, so that they do not appear in the CoP-matrix. This has been the case for the wavelength for each tested model approach. That reveals that the wavelength has no significant importance for the ultra-short pulse ablation of alumina, at least within the examined value ranges. Due to the physical relations between several input parameters, high input correlations occur, which reduce the model quality and make realistic conclusions difficult. For this reason, it has been necessary to filter some parameters manually so that input correlations can be avoided and physically meaningful models of high prognosis ability are generated. In particular, the best models could be created either by considering power, pulse distance and line distance in combination with the focus diameter or fluence and pulse overlap without taking the focus diameter into account, because this is already included in the aforementioned parameters. Using the second mentioned model, especially roughness-related phenomena can be explained very well. However, since the DoE was set up respecting the actuating parameters, only small CoPs are achieved for this case due to an asymmetric distribution of the experimental points. By contrast, high total CoPs occur by removing the calculated variables fluence and pulse overlap. This model (figure 3) is of particular importance for the explanation of phenomena associated with the depth parameters. In order to investigate the processes leading to ultra-short pulse ablation of alumina, both models have been considered, though. The essential findings are summarized in the following.

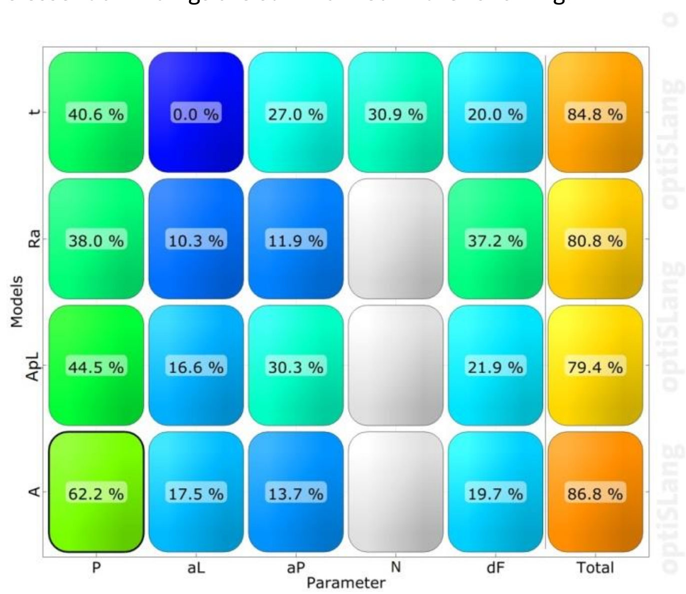

Fig. 3. CoP-matrix of best achieved metamodel

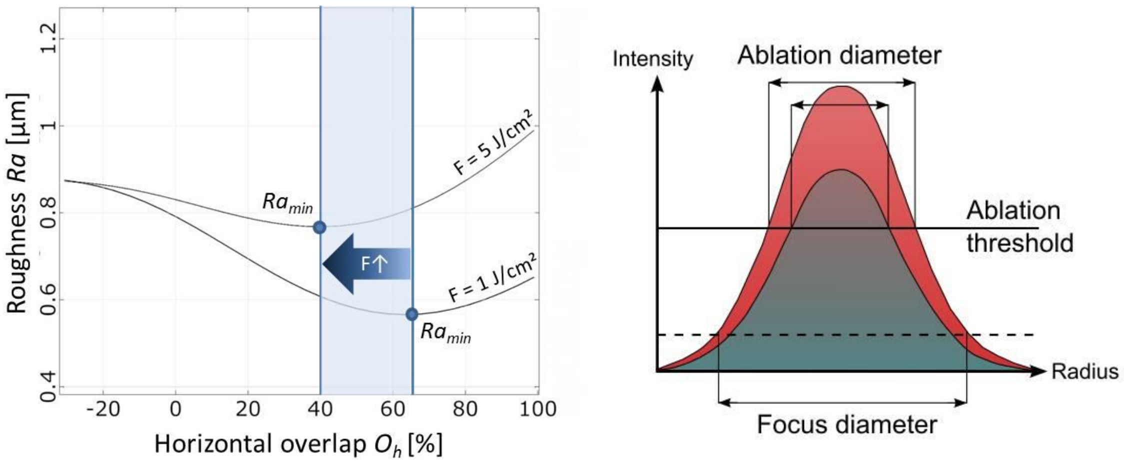

Fig. 4. (a) Fluence dependent shifting of optimal pulse overlap; (b) Fluence dependent changing of effective ablation diameter

4.1. Optimization of roughness

In the majority of cases, the roughness rises if fluence and pulse overlap are increased. However, the metamodeling indicates that there exists an optimal pulse overlap for which the roughness can be reduced. Starting from this point, the roughness rises for both increasing and decreasing overlap. As can be seen in figure 4 (a), this optimum strongly depends on the fluence. The pulse overlap leading to a minimal roughness shifts to smaller values if the fluence is increased. This observation can be explained by the fact that the effective ablation diameter increases with an increase of power as shown in figure 4 (b). That leads to a larger effective overlap at similar pulse distances. However, since the metamodeling is based on the theoretical overlap, a shifting of the values occurs. As a result, small roughnesses and therefore good surface qualities are achievable even for high fluences, just by adapting the pulse distance.

4.2. Optimization of ablation rate

The depth-related output parameters mainly depend on the average laser power. Figure 5 shows that the ablation rate rises steadily with an increase of power. In addition, the pulse distance as well as the line distance have a decisive influence on the effectiveness of the material removal. The response-surface-diagrams reveal that the functional relation between ablation rate and pulse distance significantly changes through a variation of the line distance.

In the case of large line distances (figure 5A), the removal rate increases with decreasing pulse distance. This is due to the increasing pulse overlap, which generates higher ablation depths. The high influence of the pulse distance on the ablation depth can be also seen in the CoP-matrix (figure 3). In addition to the ablation depth, the ablation rate is also determined by the processing speed. Since small pulse distances are generated by the use of low scanning speeds, they generally have a negative effect on the removal rate. However, the steady increase of the ablation rate with decreasing pulse distance shows that this effect plays only a subordinate role.

On the other hand, regarding small line distances (figure 5C), a completely different functional interrelationship between ablation rate and pulse distance can be observed. Especially regarding high power

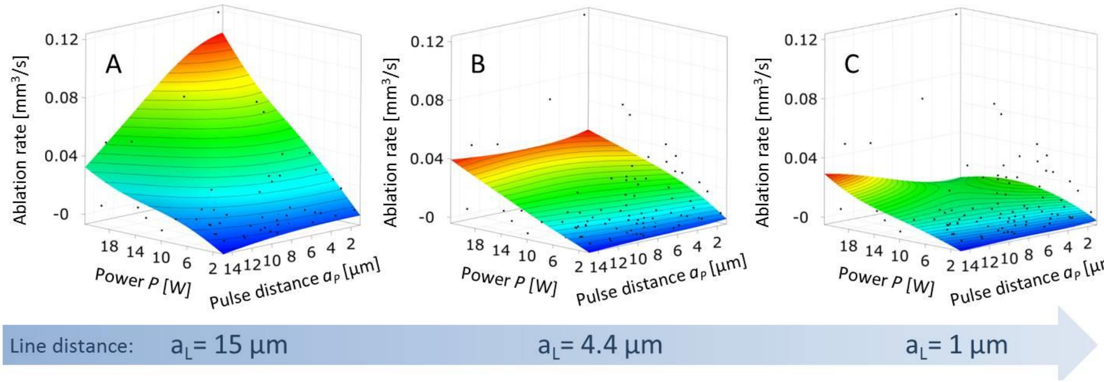

Fig. 5. Ablation rate in dependence of pulse distance and line distance

values, the removal rate now rises with increasing pulse distance, i.e. with decreasing pulse overlap. This seems to contradict the previous observations, but can be justified by the high influence of the line distance on the ablation rate. It has a considerably higher impact on the process efficiency than the scanning speed. Decreasing line distances, as well as small pulse distances, generate higher pulse overlaps and thus lead to an increase of the removal depth. However, small line distances also mean that the area to be processed has to be filled with more scanning lines. Due to the many jumping and marking vectors to be executed, the processing time increases significantly. For this reason, the ablation rate generally decreases with decreasing line distance. The loss of time at small line distances is so crucial that the scanning speed gains in importance. High marking speeds and resulting high pulse distances can now lead to an increase of the ablation rate.

In between, there exists a line distance for which the opposing influences of the pulse distance on the ablation depth and the processing time compensate each other, so that an independence of the removal rate from the pulse distance can be detected (figure 5B).

4.3. Development of material removal into depth

In addition to the surface quality described by the roughness and the process efficiency described by the ablation rate, it also makes sense to take a closer look at the parameter dependencies for the actual material removal indicated by the ablation depth and the ablation per layer. According to the CoP-matrix, the number of layers is the second most important influencing factor for the ablation depth, only surpassed by the laser power. This is easy to understand and just what was to be expected. Much more interesting is the fact that the number of layers has no influence on the remaining responses at all. This indicates that the ablation process continues constantly into depth. Each layer can be processed under the same conditions, irrespective of the number of layers which have already been removed. Such a behavior is a prerequisite for the three-dimensional structuring of material with high shape accuracy. As a result, a high agreement of calculated target contours with processed actual contours can be ensured.

5. Summary and Discussion

Within the scope of the investigations, it could be shown that a sensitivity analysis based on metamodeling can be used to identify most significant parameters as well as physical relations between input and output variables of complex laser processes. The gained knowledge can be used to optimize efficiency and quality of the process and to develop general processing strategies. It should be noted, however, that the models must always be thoroughly examined regarding their physical validity. In particular, a filtering of input parameters has to be executed in order to avoid input correlations and to obtain well-founded models.

In the context of investigating the ablation process of alumina, useful new insights into the beam-material-interactions of ultra-short pulses with technical ceramics could be gained. Fluence and pulse overlap have been identified to be by far the most important influencing parameters, which describe the process best. The results concerning the surface quality show that it is possible to achieve acceptable roughness values even at high laser powers by optimizing the pulse overlap. These are sufficiently good for at least most possible applications. In this case, the general rule of laser materials processing that quality and quantity cannot be reconciled can at least be somewhat softened. Regarding the efficiency of the process, it could be shown that it makes sense to consider pulse distance and line distance separately in order to generate the highest possible ablation rates. This contradicts the so far existing strategy of always equating pulse distance and line distance in favor of a homogeneous energy distribution.

Nevertheless, more detailed investigations have to follow. Due to the technical characteristics of the machinery, such as the availability of varying power ranges for different wavelengths, an asymmetrical design space was used so far. This allowed the involvement of a broad value range, but has shown a negative effect on the model quality, characterized by reduced CoP values. For further investigations, the experimental design has to be changed to validate the collected findings and to increase the model quality. Including further technical ceramics, the influence of the material shall be examined in order to gain an even better understanding of ultra-short pulse laser processes.

Acknowledgements

The presented results have been obtained in a research project (2015 VF0021), which is supported by the free state of Thuringia. A cofinancing is carried out by the Europäischer Fonds für regionale Entwicklung (EFRE). This support is gratefully acknowledged.

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