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Experimental and analytical investigation of cemented tungsten carbide ultra-short pulse laser ablation

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Abstract

Ultra short pulse laser processing of hard materials, such as cemented tungsten carbide, requires an accurate and agile experimental and analytical investigation to obtain adequate information and setting parameters to maximize ablation rate. Therefore, this study presents a systematic approach which, first, experimentally searches for the variables with the most significant influence on the objective using a design of experiments method; and second, analyzes by means of existing ablation theory the interaction of the material and laser taking into account the Beer-Lambert law and incubation effect. Therefore, this places a basis for future analytical-experimental validation of the examined material.

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1. Introduction

The development of ultra-short pulse laser ablation permits the introduction of this technology in areas dominated by traditional technologies, such as grinding [1, 2]. This trend, mainly, caused by the advantages of ultra-short pulse sources [3], e.g., minimal thermal side-effects, industrial robustness and precision, is complemented by the development of hard and ultra-hard materials like cemented tungsten carbide (also known as hard or heavy metal), polycrystalline diamond (PCD), polycrystalline boron nitride (PCBN) and monocrystalline diamond (MCD). Such materials are difficult, costly and time demanding in their traditional processing [1, 4, 5, 6]. Nevertheless, the laser processing of such materials demands adequate setting parameters and levels for their effective ablation [7]. This would usually lead to an extensive experimental investigation on the material whose results cannot due to the laser-material interactions be applied to other materials or even to the same material when having different component percentages [8]. Therefore, this paper presents an approach that includes an experimental and analytical path to accelerate, first, by design of experiments (DoE), the identification of the influential variables, second, to set the basis of the ablation process and laser-material interaction by a theoretical analysis of existing models, and third, to allow the validation of the model by future experimental results.
2. Experimental approach: identification of the most influential parameters

2.1. Experimental setup

In this investigation, a blank of cemented tungsten carbide is laser processed. It consists of a chemical composition of 12% Cobalt (Co) and 88% tungsten carbide (WC) with a grain size of 0.5 μm. It is important to mention that this work takes into account the whole composite material (cemented tungsten carbide) and not the single components (Co and WC).

The laser beam source is a Picosecond LUMERA Hyper Rapid 50 at a wavelength $\lambda = 1064$ nm and an average beam power of $P = 50$ W at pulse frequency $f = 1000$ kHz [9]. The pulse has a length of $t_{pl} = 10$ ps and the focal diameter is of $d_w = 30$ μm. All experiments are executed ablating a square cavity of 4 mm$^2$ on the blank. For each experiment, the area is irradiated 50 times and it is focused on the surface of the material (50 layers without focus adjustment).

2.2. Methodology

Based on the variable search by Shainin [10], the DoE method concentrates on the identification of the most significant variables based on parameter comparison and the Pareto principle. The objective of the method is to identify and/or confirm the most influential parameters on the ablation of cemented tungsten carbide. The response for this investigation is the ablation rate $Q_n$ [mm$^3$/s] derived from the ablation depth [mm] and the goal is to maximize it. First, the parameters are selected and the settings have to be chosen and listed into two groups; the first includes the settings that are expected to show positive (+) results, i.e., high ablation rate; and the second is grouped according to the levels that are expected to show low ablation (-), as presented in table 1.

Table 1. Choice of parameters for ablation rate $Q_n$

| Factors | Settings |
|---------|----------|
| A Laser beam power [W] | Worst $Q_n$ | Best $Q_n$ |
| B Burst-mode [number of bursts] | 5 | 1* |
| C Pulse overlap and track overlap [%] | 50 | 90 |
| D Scanning processing strategy [-] | bidirectional | Meandering |

* Single pulse

According to DIN 32540, which includes the factors laser pulse frequency, scanning speed, and hatch distance. For $PO = 50 \%$: $f = 800$ kHz, $v_s = 10800$ mm/s and $\Delta_y = 0.01349$ mm, respectively; and for $PO = 90 \%$: $f = 800$ kHz, $v_s = 2160$ mm/s and $\Delta_y = 0.0024$ mm, respectively.

Once the factors and its respective settings are determined, the preliminary experiments are executed (first group and second groups with one repetition). The average results of the preliminary experiments are named as reference value $V_-$ and $V_+$, respectively (see table 2). These reference values are the highest and lowest limits of the response, and serve as a point of reference for the contrast of the variables tested in the main experiment. The main experiment then examines each of the variables independently, i.e., each variable against the rest of the variables. First, the variable under examination is set at its negative setting against the rest of the variables in their positive setting, and then, the same variable is set at its positive setting and the rest at their negative setting. In this way each variable is examined in the main experiment. Finally, after running experiments with each of the variables at both settings, the variation is quantified and the most significant variables are identified [10, 11, 12]. Table 2 presents the results and conclusions for cemented tungsten carbide.
Table 2. Main experiment results and conclusions on ablation rate \( Q_n \)

| Factors | Combination | Results | \( V^+ \) | \( V^- \) | Variation | Average | Conclusion |
|---------|-------------|---------|----------|----------|-----------|---------|------------|
|         |             | [mm³/s] | [mm³/s]  | %        | %         |         |            |
| A Laser beam power | A.R.       | 0.0144  | 0.0078   | 0.0226   | 84        | 59      | 2 Red X    |
|         | A.R.       | 0.0148  |          |          |           |         |            |
| B Burst-mode                 | B.R.       | 0.0043  | 0.0078   | 0.0226   | 45        | 47      | 3 Pink X   |
|         | B.R.       | 0.0333  |          |          |           |         |            |
| C Pulse overlap and track overlap | C.R.       | 0.0173  | 0.0078   | 0.0226   | 121       | 98      | 1 Red X    |
|         | C.R.       | 0.0058  |          |          |           |         |            |
| D Scanning processing strategy | D.R.       | 0.0083  | 0.0078   | 0.0226   | 6         | 21      | less relevant |

Factor B “pulse and track overlap” [13] (i.e. scanning speed, frequency and hatch distance) and factor C “laser beam power” resulted as the most influential factors on the maximization of the ablation rate. Factor B “burst-mode” shows less but still interesting influence on the response. Figure 1 depicts the ablated blank.

2.3. Conclusion

Variable search method helps to identify influential laser-processing parameters for materials that do not present sufficient information, in terms of e.g., mechanical, chemical and optical properties, to achieve a pure analytical investigation. This DoE result suggests more attention to power and overlap than to the others.

Fig.1. (a) Ablated cemented tungsten carbide blank, (b) detail of the ablated fields of 4 mm²

3. Analytical approach: Beer-Lambert law and incubation effect

In this part of the investigation a theoretical analysis is carried out for single and multiple-pulse ablation on cemented tungsten carbide. Laser power and the relationships of pulse overlap (e.g., frequency, scanning speed and number of pulses) are taken into account.

3.1. Theoretical background

In the process of ultra-short pulse laser ablation, several (multiple) pulses are thrown on the same spot. This causes that each subsequent pulse changes the properties of the material and consequently its threshold fluence by a material-related factor. This phenomenon is known as incubation effect [14, 15, 16] and is given by the equation 1:

\[
F_{th} = F_{th}(1) \cdot N^{s}
\]

Where,

- \( F_{th} \) Threshold fluence
- \( F_{th}(1) \) Threshold fluence of one pulse
- \( N \) Number of pulses
- \( s \) Incubation factor
Important to consider in the analysis of the ablation behavior of this paper is the adaptation of the Beer-Lambert law to laser radiation by Darif and Semmar, which states that any electromagnetic radiation going through a material, diminishes exponentially with the thickness for a given wavelength, as shown in (2) [17].

\[
G_t(y, t) = I(t) \cdot (1 - R(T)) \cdot e^{-\frac{y}{\delta}} 
\]  

(2)

Where,

- \(G_t(y, t)\) Heat source (in depth \(y\) over time \(t\))
- \(I(t)\) Laser pulse intensity (over time \(t\))
- \(R(T)\) Reflectivity (at a temperature \(T\))
- \(\delta\) Optical penetration depth

In this way, the Beer-Lambert law describes the penetration of the pulse energy through the material and determines the necessary laser power to cross the ablation threshold.

To apply the above mentioned theoretical relationships (equations 1, 2) it is important to determine the number of effective pulses on the material when applying single pulse and also with burst-mode.

Formula 3 defines the number of pulses in a determined segment. The number of ultra-short pulses (\(N\)) for a burst-mode level (\(n\)) on the same spot [18] can be calculated from the frequency of laser pulses \((f)\) of diameter \((d_w)\) following a linear direction in a scanning speed \((v_s)\). This paper considers \(n = 1\), i.e., no burst-mode.

\[
N_{(n)} = f \cdot d_w \cdot v_s \cdot n^{-1} \cdot n 
\]  

(3)

Equation 4 is the result of transforming the equation 1 [14] by considering the number of pulses \(N\) for a burst-mode level (equation 3). In this case, the threshold fluence \(F_{th}\) includes the incubation factor for burst-mode ablation.

\[
F_{th} = F_{th}(1) \cdot N_{(n)}^{-1}^{-1} 
\]  

(4)

Input fluence \(F(6)\) is calculated by using the adaptation of the Beer-Lambert law for laser radiation [17] and according to the Gaussian pulse profile for the input power \(P_{in}(5)\) [14, 17, 19, 20].

\[
P_{in} = \frac{E}{S \cdot \tau_H} \cdot e^{-\frac{4(\tau_H - \tau_{nd})^2}{\tau_{nd}^2}} 
\]  

(5)

\[
F = \frac{\sum_{m=1}^{\infty} \frac{E}{S \cdot \tau_H} \cdot e^{-\frac{4(\tau_H - \tau_{nd})^2}{\tau_{nd}^2}}}{1 + 2 + \cdots + m} 
\]  

(6)

Where,

- \(\tau_H\) Pulse length
- \(E\) Pulse energy
- \(S\) Laser spot area
- \(\tau_{nd}\) Half the pulse length \(\tau_H\)

Based on [14], the ablation depth \(Z_{abl}\) for number of pulses on the same spot \(N_{(n)}\) is defined by equation 7 and volume ablation \(V_{abl}\) for an area \(A\) by equation 8.

\[
Z_{abl} = N_{(n)} \cdot \delta \cdot \ln \frac{F}{F_{th}} 
\]  

(7)

\[
V_{abl} = A \cdot f \cdot n \cdot \delta \cdot \ln \frac{F}{F_{th}} 
\]  

(8)

Where,
In this paper the ablation depth $Z_{abl}$ is analyzed.

### 4. Analysis

Using the properties of the material, listed in table 3, the theoretical analysis starts with the calculation of the threshold fluence of the material $F_{th}(1)$ when a single pulse is irradiated. For this, it is necessary to calculate the optical penetration depth $\delta = \alpha^{-1}$ for this case $\delta = 2.25 \times 10^{-8}$ m. Knowing this, it is necessary to calculate the power that is required for 10 ps pulse length to evaporate the carbide. Therefore, the energy for temperature change and phase change are calculated using (9) and (10), respectively. First, the energy needed to rise the temperature to liquidation boundary and the energy to change the phase of the material from solid to liquid, and then, the energy to rise the temperature from liquid to boiling temperature, and the energy to change the phase from liquid to vapor.

\[
E_v = C_p \cdot \rho \cdot \frac{dT}{dt}
\]

\[
E_{ph} = L_H \cdot \rho \cdot t^l
\]

Where,

- $E_v$ Minimum energy required for temperature change
- $E_{ph}$ Minimum energy required for a material phase change
- $C_p$ Specific heat capacity
- $\rho$ Density
- $dT$ Change in temperature
- $dt$ Change in time
- $L_H$ Latent heat
- $t$ Time period

As a result, it is found that the power to evaporate the material is $P = 8.744 \times 10^{21}$ W m$^{-2}$. For this power, laser pulse length and optical penetration depth, the resulting threshold fluence is $F_{th} = 1960$ J m$^{-2}$. The visualization of the pulse that ablates the material is calculated by the Beer-Lambert law formulas (2) and (5) for a Gaussian distribution as seen in figure 2. The orange area is the ablated area; it shows the power that provides the minimum energy to ablate equal or greater than the ablation threshold (threshold fluence). The other regions represent heated material which stays below the ablation threshold.

![Fig.2. Adapted Beer-Lambert law energy 2D distribution for 1000 kHz, 10 ps and single pulse](image-url)
Table 3: Material properties for the analysis [21, 22, 23, 24, 25]

| Properties                  | Cemented tungsten carbide | Unit           |
|-----------------------------|---------------------------|----------------|
| Thermal conductivity $k$    | 164                       | W m$^{-1}$ K$^{-1}$ |
| Specific heat (solid)$c_p$  | 210                       | J Kg$^{-1}$ K$^{-1}$ |
| Specific heat (liquid)$c_p$ | 292*                      | J Kg$^{-1}$ K$^{-1}$ |
| Mass density (solid)$\rho_0$| 15930                     | kg m$^{-3}$    |
| Mass density (fluid)$\rho_f$| 14500*                    | g cm$^{-3}$    |
| Absorption coefficient $\alpha$| 4.45E+07                  | m$^{-1}$       |
| Absorption index $ab_{max}$ | 3.77                      | -              |
| Reflectivity $R$            | 0.60254                   | -              |
| Melting point $T_m$         | 3143                      | °K             |
| Boiling point $T_b$         | 6273                      | °K             |
| Latent heat of fusion $L_f$ | 4000000                   | J kg$^{-1}$    |
| Latent heat of evaporation $L_H$ | 4020000                  | J kg$^{-1}$    |
| Incubation factor $s$       | 0.85*                     | -              |

* approximated

For the same frequency of 1000 kHz, a multiple pulse ablation with different scanning speeds (i.e. different number of pulses) is shown in figure 3.

![Fig.3](image)

Fig. 3. (a) calculated ablation depth, and (b) its respective threshold fluence for the ablation of cemented tungsten carbide

In figure 3, the effect of incubation $s$, decreasing the ablation threshold, i.e. increasing the ablation depth, by irradiating a higher number of pulses can be observed. These represent the results of the theoretical analysis of formulas (1), (4), (5) and (6). Figure 3(a) shows a decreasing exponential function, where the ablation depth decays approximately from 50% to 15% every 1000 mm/s, from low to high scanning speeds, respectively. Figure 3(b) illustrates an increasing logarithmic function with the threshold fluence varying every 1000 mm/s approximately from 9% to 2%, at low and high scanning speeds, respectively.

5. Results and Discussion

In this paper subsequent two-step approach for the investigation of laser ablation with ultra-short pulses was presented. The first step, known as Shainin’s variable search, identified the parameters with the highest influence on the maximization of the ablation rate when processing cemented tungsten carbide. This method helped to find laser power as well as pulse and track overlap as the most influential variables on the process. Therefore, future studies can concentrate on their study avoiding unnecessary complex experimental designs. The second step, a theoretical investigation, contributed with the
modeling of the ablation process of the material by taking into consideration material properties, the Beer-Lambert law, a Gaussian beam distribution and the incubation effect. Several characteristics of the process, e.g., optical penetration depth, threshold fluence, ablation depth with single and multiple pulses were calculated, among others.

Future work considers results for other combinations of frequency and scanning speed not included in this work and the analysis of volume ablation for the objective of obtaining information to optimize the ultra-short pulse laser ablation of cemented tungsten carbide.

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