The purpose of this research is to investigate the femtosecond laser-matter interaction for a tungsten carbide with 10% of cobalt. A femtosecond laser (1030 nm) with a pulse duration of 400 fs have been used for this study. For cumulated fluences between 1.4 and 4 J/cm², Laser-Induced Periodic Surface Structures (LIPSS) could be produced with a low ablation rate. LIPSS had a spatial period of 665 nm and an amplitude of 225 nm. The a-thermal ablation threshold fluence have been found to be from 0.35 to 11 J/cm² in cumulated fluence for a range between 1 to 100 pulses. Thus dimples could be fabricated without any thermal effects. In addition, incubation coefficient and optical penetration depth of tungsten carbide were determined. They are equal to 0.79 and 19 nm respectively.

1. INTRODUCTION

For the last two decades, ultrashort pulsed lasers have been beneficial to handle issues in many applications from manufacturing, military, aerospace and medical industries. Nowadays, this technique is more and more employed to tune the surface properties of a material such as wettability and tribological behavior [1]. In this work, we used laser surface structuring for improving cutting results on tungsten carbide workpieces for turning applications. The desired scope is to decrease both abrasive wear and adhesion on workpieces while machining stainless steel or aluminum alloy. Nevertheless, the interaction of laser pulses with tungsten carbide material has been tackled by only few researchers. Dumitru et al. [2-5] determined the threshold fluence for texturation regime which permits to create nanoscale structures named LIPSS or "ripples". Pfeiffer [6], Calderón Urbina [7] and Neves [8] determined the a-thermal ablation threshold fluence using respectively a femtosecond, picosecond and nanosecond-lasers. For a purpose of improving workpieces made of tungsten carbide by adapting their tribological and physicochemical properties, we need to determine precisely these threshold fluences for performing desired double-scale structures on samples. In fact, we aim to produce nano-scale structures (LIPSS), micro-scale structures and a combination of nano- and micro-scale structures on tungsten carbide workpieces. Knowing the laser-matter interaction is crucial before considering laser material micromachining process. We are proposing to investigate the behavior of tungsten carbide with 10% of cobalt under femtosecond laser pulses at variable peak fluence and number of laser pulses. We will identify the laser-matter interaction regimes for the selected material and give the mapping of fluence domains corresponding to each regime. We will also determine the incubation coefficient and optical penetration depth for tungsten carbide, parameters which are indexed only by one author in literature [7].

2. MATERIALS AND METHODS
The equipment used for laser experiments was a laser source (Tanor HP model from Amplitude Systems, France) with an average power of 100 W, a maximum pulse energy of 500 µJ at a wavelength of 1030 nm and a variable repetition rate adjustable from one shot to 2 MHz. The minimum pulse duration is equal to 400 fs. We used a Gaussian beam which had a radius of 16.67 ± 0.5 µm measured by a beam analyzer. The studied samples were made of tungsten carbide with 10% of cobalt binder and with an average grain size of 0.7 µm. The samples were mirror-polished with diamond particles (from 9 µm down to 3 µm) and washed in an ultrasonic cleaner with ethanol for 5 min before the laser machining. The resulting surface roughness was Ra = 60 nm and Rz = 80 nm. A static matrix of impacts has been performed using different values of peak fluence (from 0.028 to 5.6 J/cm²) and various number of pulses (from 1 to 100) at a repetition rate of 1 kHz. Dimples were examined using a scanning electron microscope (SEM-FEI Quanta® 450 W), an atomic force microscope (AFM) and a confocal microscope (Alicona Infinite focus®). These different techniques permitted either to measure dimples depth and diameter, or to reveal the morphology of the dimples, and to observe and characterize the Laser-Induced Periodic Surface Structures (LIPSS).

3. THEORY/CALCULATION

For a Gaussian beam profile shown in Figure 1a), the spatial variation of laser fluence can be expressed as [9]:

\[ F(r) = F_0 \exp \left(-\frac{2r^2}{\omega_0^2}\right) \quad , \quad F_0 = \frac{2E}{\pi \omega_0^2} \] (1)

where \( r \) is the distance from the laser beam center, \( F_0 \) is the peak fluence at the center (\( r = 0 \)), \( \omega_0 \) is the focused beam radius at 1/e² of \( F_0 \) and \( E \) is the incident laser pulse energy. In case of \( N \) static impacts, when the peak fluence \( F_0 \) is above the ablation threshold fluence \( F_{th}(N) \), a dimple appears by ablation process as presented in Figure 1b).

\[ \text{Fluence} (\text{J/cm}^2) \]

\[ F_0 \quad 0 \quad \omega_0 \]

\[ F_0 \quad 0 \quad \omega_0 \]

\[ r \quad (\mu m) \]

\[ D \quad (\mu m) \]

\[ h_N \quad N \quad \alpha^{-1} \cdot \ln \left(\frac{F_{th}(N)}{F_{th}(1)}\right) \]

\[ N \quad \alpha^{-1} \cdot \ln \left(\frac{F_{th}(N)}{F_{th}(1)}\right) \]

\[ D \quad = \quad \text{depth} \]

\[ F_{th}(N) = F_{th}(1) \cdot N^{\xi^{-1}} \] (3)

where \( \xi \) is the response of the material to cumulative ablation shots. When \( \xi = 1 \), the threshold fluence is independent of the number of pulses and there is no incubation effect. When \( \xi < 1 \), some residual energy is left in the material between each pulse, thus decreasing the a-thermal ablation threshold fluence while increasing number of pulses. For the a-thermal ablation regime, the ablation depth depends on the optical penetration depth \( \alpha^{-1} \), where \( \alpha \) is the absorption coefficient of the material at the given laser wavelength. The ablation depth for 1-pulse is given by [13]:

\[ h_1 = \alpha^{-1} \cdot \ln \left(\frac{F_{th}(1)}{F_0}\right) \] (4)

The ablation depth \( h_N \) for \( N \) pulses shown in Figure 1c) is expressed as:

\[ h_N = N \cdot \alpha^{-1} \cdot \ln \left(\frac{F_{th}(N)}{F_{th}(1)}\right) \] (5)

4. RESULTS AND DISCUSSION

As defined in Equation 2, the relationship between the measured dimple diameter squared (\( D^2 \)) and the logarithm of peak fluence can be used to deduce the laser beam spot radius \( \omega_0 \) and a-thermal ablation threshold fluence for \( N \) pulses. Figure 2 shows the relationship between the square of the dimple diameter and the logarithm of peak fluence for dimples produced by 10,50 and 100 pulses at a repetition rate of 1 kHz. Applied peak fluences were calculated using measured waist size of the focused beam using Equation 1. Dimple’s diameter were measured using confocal microscope (Alicona Infinite focus®). The laser beam spot radius \( \omega_0 \) is calculated from the slope of the line of best fit to the data in Figure 2 for each \( N \) pulses. An average \( \omega_0 \) is found to be 15.75 ± 0.08 µm which is close to the measured beam spot size (16.67 ± 0.5 µm). Once beam size is calculated from experimental analysis, from this point forward, applied peak fluence will be calculated using this new value of beam radius. As the a-thermal ablation threshold fluence changes with number of pulses applied to the same spot, by an extrapolation to \( D^2 = 0, F_{th}(N) \) for
each $N$ can be determined. From Figure 2, a-thermal ablation threshold fluences were found to be 0.19 J/cm², 0.14 J/cm² and 0.11 J/cm² for $N = 10, 50$ and $100$ respectively.

Figure 3 shows the evolution of a-thermal ablation threshold fluence as a function of number of pulses. For a-thermal ablation regime, the range of the threshold fluence is found to be between 0.11 to 0.35 J/cm² from $N = 100$ to 1 pulses, as seen on Figure 3. It corresponds to a range of 0.35 to 11 J/cm² in cumulated fluence ($N \times F$). A-thermal ablation threshold fluence for a single-pulse is found to be $F_{th}(1) = 0.35$ J/cm².

Among the few works giving results about the a-thermal ablation domain and thresholds, Neves et al. found the upper limit of the range of a-thermal ablation zone to be at 22.92 J/cm² in cumulated fluence starting their investigations from 3.82 J/cm² whereas Tan et al. worked in the range between 0.49 to 4.11 J/cm² without defining these values as the limits of the ablation domain [14]. More complete work have been published by Dumitru et al. [4, 5], Pfeiffer [6] and Calderón Urbina [7]. Dumitru and Pfeiffer found the lower limit around 0.4 J/cm² and Calderón Urbina around 0.2 J/cm² both for a single-pulse. The variations can be explained, in one hand, by the difference of pulse duration applied, which is 10 ps for Calderón Urbina and about 100 fs for Pfeiffer and Dumitru. On the other hand, it can also be due to wavelength employed during experiments, which is 1064 nm for Calderón Urbina et al., 532 nm for Neves et al. and around 775-800 nm for the other authors. However the order of magnitude of these values are close to the measured one in the present work notwithstanding the different composition of studied materials.

Incubation coefficient $\xi$ has been determined from slope of the line of best fit to the data in Figure 4, and found to be equal to 0.79. According to Calderón Urbina et al., the incubation coefficient for tungsten carbide is given as 0.85 for a matrix with 12% of cobalt and a grain size of 0.5 µm [7].

According to Equation 5, when plotting depth of ablated dimples as a function of the logarithm of peak fluence $F_{0}$ which is calculated using the new value of beam spot size (15.75 µm), as illustrated on Figure 5, optical penetration depth ($\alpha^{-1}$) can be determined. Dimple's depth were measured using a confocal microscope (Alicona Infinite focus®). We
found an average optical penetration depth equal to 19 ± 2.8 nm. According to Calderón Urbina and Pfeiffer, optical penetration depth for tungsten carbide is equal to 22.5 nm measured with different lasers and carbide compositions [7].

The LIPSS appear above a threshold value of fluence. Figure 7 displays LIPSS with low ablation rate produced at a peak fluence of 0.2 J/cm² and a number of pulses of 20. In our matrix, periodic LIPSS appeared from \( F_0 = 0.2 \) J/cm² for \( N = 7 \), with a mean periodicity of 665 nm and an average height of 225 nm measured using AFM technique. 2D profile of LIPSS is presented on Figure 8. These ripples had a period equal to approximately \( 0.6 \times \lambda_{\text{laser}} \). In the literature, this kind of ripples is called coarse ripples or low spatial frequency (LSF-LIPSS) [15]. The orientation of LIPSS is perpendicular to the polarization direction of the laser beam. From AFM analysis, we could determine the range of texturation regime (LIPSS generation zone) without digging. This range is between 1.4 to 4 J/cm² which is a cumulated fluence \( (N \times F_0) \) for a peak fluence of 0.2 J/cm² and a number of pulses varying from 7 to 20 pulses. For a single-pulse, threshold peak fluence of texturation regime would be between 0.22 and 0.35 J/cm². According to Dumitru et al., the range of texturation regime is between 0.4 to 2.8 J/cm² for a tungsten carbide of 10% of cobalt and a grain size of 0.5 µm [5]. Once threshold fluences for each regime were established, laser-matter interaction domain map for tungsten carbide can be drawn, as shown on Figure 9.
5. CONCLUSIONS

We have investigated the laser-matter interaction for tungsten carbide with femtosecond laser for multiple number of pulses and different fluence values. The main conclusions can be summarized as follows:

(1) Three regimes were identified which correspond to: low interaction zone, LIPSS zone and a-thermal ablation zone. Figure 9 is intended to show these three range zones.

(2) Ripples could be produced starting from \( N = 7 \) at a peak fluence of 0.2 J/cm². They were perpendicular to the polarization of the laser beam and have a period equivalent to 0.6×\( \lambda_{\text{laser}} \).

(3) The single pulse threshold fluence for a-thermal ablation was determined and is equal to 0.35 J/cm² which is close to the values of Dumitru and Pfeiffer.

(4) Incubation coefficient of tungsten carbide is found to be 0.79 in agreement with Calderón Urbina's postulated value.

(5) Optical penetration depth of tungsten carbide has been determined and found equal to 19 nm in accordance with Calderón Urbina and Pfeiffer's results using different tungsten carbide grades.

It is the first time that all the laser-matter parameters for generating ripples or femtosecond laser-matter ablation on tungsten carbide have been measured and communicated to worldwide scientific community.

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