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Investigating and understanding the effects of multiple femtosecond laser scans on the surface topography of stainless steel 304 and titanium

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ABSTRACT

The majority of studies performed on the formation of surface features by femtosecond laser radiation focuses on single scan procedures, i.e. manipulating the laser beam once over the target area to fabricate different surface topographies. In this work, the effect of scanning stainless steel 304 multiple times with femtosecond laser pulses is thoroughly investigated over a wide range of fluences. The resultant laser-induced surface topographies can be categorized into two different regimes. In the low fluence regime ($F_{\Sigma line,max} < 130\,J/cm^2$), ellipsoidal cones (randomly distributed surface protrusions covered by several layers of nanoparticles) are formed. Based on chemical, crystallographic, and topographical analyses, we conclude that these ellipsoidal cones are composed of unablated steel whose conical geometry offers a significant degree of fluence reduction (35–52%). Therefore, the rest of the irradiated area is preferentially ablated at a higher rate than the ellipsoidal cones. The second, or high fluence regime ($F_{\Sigma line,max} > 130\,J/cm^2$) consists of laser-induced surface patterns such as columnar and chaotic structures. Here, the surface topography showed little to no change even when the target was scanned repeatedly. This is in contrast to the ellipsoidal cones, which evolve and grow continuously as more laser passes are applied.

1. Introduction

Femtosecond (fs) laser surface processing is a high-precision technique used to impart micro- and nano-sized features to a material surface. The resulting features can be broadly categorized into laser-inscribed and laser-induced structures [1]. Laser-inscribed structures consist of machined features such as grooves and holes, whose dimensions are equal to or greater than the effective laser beam diameter [2,3]. On the other hand, laser-induced surface structures that are formed under laser irradiation have feature sizes smaller than the effective beam diameter, and the latter has been the subject of extensive research in recent years. Laser-induced structures that have been discovered on metals include laser-induced periodic surface structures (LIPSS) [4–8], bumps/cones [9–29], holes [9,11,21,23,30], undulating grooves [9,11,21], melt-like [23,31], and chaotic structures [9,11,32]. These laser-induced structures can be used to alter the wettability of a material, resulting in a more hydrophobic or hydrophilic surface [12,13,33,34]. Other uses of such laser-induced topographies include surface coloration [35,36], surface-enhanced Raman scattering (SERS) [37], reduced cell growth [38,10], and improved broadband optical absorption [19,29,39] for applications such as photovoltaics [40]. Comprehensive reviews of fs-laser surface texturing on metals has been provided by Ahmmed et al. [1] and Voroboyev and Guo [41].

In their recent work, Ahmmed et al. [9] investigated the surface topographies formed on titanium, stainless steel, aluminum and copper by fs-laser irradiation over a wide range of processing parameters. They observed that when an irradiated area is rescanned multiple times at a particular experimental setting, the surface features become increasingly regular and well-defined. Similarly, Noh et al. [16] fabricated highly regular arrays of bumps and pillars on NAK80 mold steel by repeating their laser scan

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30 and 50 times, respectively. Kam et al. [13] observed the formation of two distinct types of micro-cones on stainless steel 316L, which only appeared after the target was scanned more than one hundred times. Finally, studies by Zuhlke et al. [23–26] have expounded on the dominant formation mechanisms of these laser-induced surface structures, notably via stop-motion scanning electron microscopy (SEM) imaging. Despite the numerous reports on the fabrication of surface features by employing multiple laser scans, it is still unclear how re-scanning a previously-irradiated target influences the final surface topography.

Therefore, the present work investigates the effect of multiple laser scans on the resultant surface structures on stainless steel 304 over a large range of fluences ($32\,\mathrm{J/cm^2} < F_{\Sigma\mathrm{line,max}} < 1096\,\mathrm{J/cm^2}$) and number of scans (up to 30). We report the changes in surface topography that occur during repeated scanning, which, in turn, provide an improved understanding of the underlying processes that govern the formation of micro- and nano-scale surface features on laser-irradiated metals.

2. Experimental

2.1. fs-laser processing

We used an amplified Ti:Sapphire laser (Coherent Libra) with a wavelength of 800 nm, a repetition rate f_p of 10 kHz, and a pulse duration of <100 fs to produce laser-induced surface features on stainless steel 304 (0.030″ thickness, McMaster-Carr) samples polished to a roughness of $R_\mathrm{a} = 43$ nm. These samples were manipulated under the horizontally-polarized Gaussian laser beam by a linear x–z translation stage (Zaber Technologies, Inc.) in a raster scan pattern. The target was maintained at a distance of $\Delta y = +1.5$ mm from the focal point, meaning that the machining plane was located between the focal point and the 100 mm focusing lens; this yielded a theoretical $1/e^2$ beam diameter ω_o of 58 μm. A variable attenuator composed of a half-wave plate and a polarizing beam splitter reduced the output power of 4 W to the desired processing power P. The raster scanning speed was set at $v = 4$ mm/s, which corresponds to a horizontal pulse displacement of $\Delta x = 0.4$ μm. Since the width of ablated lines (ω_eff) laser-etched onto a target varies depending on the experimental conditions (P, v, Δy, sample material and gaseous environment), the vertical displacement Δz was chosen such that the vertical line overlap, φ_line, was maintained at 80% relative to ω_eff, i.e. $\Delta z = (100\% - \varphi_\mathrm{line})\omega_\mathrm{eff}$. The number of laser scans, or passes applied to each patch is denoted by N_s. After fs-laser micromachining, the steel samples were cleaned with acetone in an ultrasonic bath for 5 min to remove any residual nanoparticle debris.

2.2. Surface analysis

2.2.1. SEM and EDS

The laser-induced surface topographies were analyzed and imaged using SEM (FEI Inspect F50). Stop-motion SEM imaging (Section 3.1), first introduced by Zuhlke et al. [23], was performed as follows. Once the fs-laser had scanned the target in a raster pattern, the latter was cleaned with acetone in an ultrasonic bath for 5 min and then imaged with SEM. The sample was then returned to the translational stage and carefully re-aligned to the same starting position as before, where the second laser pass was subsequently applied ($N_\mathrm{s} = 2$). The sample was cleaned again and imaged with the SEM at the same location and magnification. This process was repeated for each additional laser scan applied, resulting in a series of SEM images that tracked the growth of ellipsoidal cones with each scan. Energy dispersive X-ray spectroscopy (EDS) on metallic specimens (Section 3.2) was conducted using the same SEM, which was equipped with a 60 mm^2 silicon drift detector (Octane Super, EDAX®), and analyzed using the TEAM™ EDS Analysis System. A 20 kV acceleration voltage was used during the EDS analysis, which corresponds to an X-ray emission depth on the order of 1–1.2 μm for stainless steel 304 and 2 μm for titanium.

2.2.2. ECCI and EBSD

In order to obtain crystallographic information pertaining to the material within the ellipsoidal cones (Section 3.3), the cones were sectioned along the transverse and longitudinal planes as follows. For the transverse section, we filled the irradiated area containing ellipsoidal cones with superglue to prevent them from fracturing. After drying, the specimen was grinded and polished using standard metallographic procedures until the cones were abraded to approximately half of their original height. The last polishing step was performed using a colloidal suspension of 50 nm silica particles. For the longitudinal section, we cut the laser-irradiated steel sample using a high-precision diamond saw through the middle of the target patch. After careful mechanical polishing (identical to the procedure followed for the transverse section), the specimen cross-section was milled in a flat ion milling system (Hitachi IM3000) with Ar^+ ions for several hours, using an accelerating voltage of 6 kV, a ion probe current of 80 μA and an angle of incidence of 80°.

Electron channeling contrast imaging (ECCI) micrographs were recorded with a retractable solid-state photo-diode backscattered electron detector (PD-BSE) attached to a Hitachi SU-8000 cold-field emission scanning electron microscope (CFE-SEM) (Hitachi High-Technologies Canada Inc.). A beam energy of 5 kV and a probe current of approximately 1 nA were used for imaging, and the beam convergence angle at the specimen surface was approximately 12.5 mrad. The electron backscatter diffraction (EBSD) analysis was conducted with the same CFE-SEM with an accelerating voltage of 20 kV and 4–5 nA probe current. The EBSD system from HKL (Oxford Instruments US) consisted of a Nordlys II EBSD camera controlled by the Flamenco software from the Channel 5 package. A 2×2 camera binning acquired the EBSD patterns, which resulted in 672 pixels × 512 pixels pattern images. Integration of two frames with 30 ms dwell time yielded the raw pattern images, and up to 8 Kikuchi bands were used for the orientation calculations. The crystallographic phase used for indexing was iron fcc (space group 225, Fm -3 m) with a lattice parameter of 3.66 Å. The EBSD maps were post-processed using Tango from the same software package.

2.2.3. 3D confocal microscopy

The heights and diameters of ellipsoidal cones were measured using 3D confocal microscopy (Olympus LEXT OLS4000) (Section 3.4). Since each laser-irradiated patch contained several ellipsoidal cones, we determined h and d for multiple cones. Furthermore, each ellipsoidal cone was measured using two orthogonal baselines as their base area was elliptical.

2.3. Model computations

Under a raster scanning scheme, the four independent laser processing parameters (P, v, Δy, φ_line) can be condensed into one variable, $F_{\Sigma\mathrm{line,max}}$, using the accumulated fluence profile (AFP) model, first developed by Eichstädt et al. [42]. This irradiation model calculates the total deposited laser energy on the substrate by summing the fluence distribution of Gaussian pulses in both horizontal and vertical displacements, i.e. Δx and Δz, respectively. Using $F_{\Sigma\mathrm{line,max}}$ to report the processing parameters is convenient because it encompasses P, v, Δy, φ_line into one variable. Nevertheless, the peak fluence ($F = 8P/\pi\omega_\mathrm{o}{}^2 f_\mathrm{p}$) and pulses-per-spot ($\mathrm{PPS} = \omega_\mathrm{o}{}^2/\Delta x\Delta z$) have been included in the figure captions in this paper to facilitate comparisons with literature. The derivation