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Induced hydrophobicity in micro- and nanostructured nickel thin films obtained by ultraviolet pulsed laser treatment

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Abstract

Nickel thin films were deposited on Ti6Al4V and surface treated with nanosecond pulse ultraviolet laser in ambient conditions using a fluence of 3.68 J/cm². A twofold to threefold increase in contact angle was observed for water, ethylene glycol and diiodomethane following laser treatment, with recorded contact angles for water in excess of 130°. No additional treatment was used to hydrophobize the surface. Three different micro- and nanometric levels of roughness were generated on the thin film surface, which can be related to the rastering parameters of the laser and to the increase in apparent contact angle. Melt droplets were identified as a main contribution to the nanometric roughness level, indicating that melting and melt ejection play an important role in surface nanostructuring.

Keywords:

ultraviolet laser, nickel, hydrophobicity, micropatterning, nanopatterning

1. Introduction

Hydrophobic surfaces, especially superhydrophobic ones, are of interest because of their excellent repelling, anti-sticking and self-cleaning characteristics [1–6]. Surface micro- and nanopatterning is widely used to create

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hierarchical structures needed to induce surface hydrophobicity and superhydrophobicity. Traditional approaches generally rely on structuring an already hydrophobic surface or on hydrophobizing a rough hierarchically-structured surface, employing a broad range of techniques and materials [1, 7–16]. Micro- and nanopatterning can also be achieved with lasers, however their use as a procedure to induce direct hydrophobicity and superhydrophobicity in metallic surfaces is only emerging. Recent studies have used picosecond to nanosecond pulsed lasers with infrared to ultraviolet wavelengths to successfully induce superhydrophobicity in platinum [17], brass [17–19], iron and stainless steels [20–25], titanium and Ti6Al4V [17, 20–22], silicon [26], copper [18, 22], cobalt [22], aluminium [22, 27] or carbon nanotubes [28]. No such surface patterning has been reported on nickel to date.

2. Experimental section

Annealed Ti6Al4V samples (15 x 15 x 2 mm) were ground using silicon carbide paper, and further wet-polished using a suspension of 3 μm size diamond particle. A final polishing step using a solution containing 0.04 μm size alkaline silica particle dispersed in hydrogen peroxide was employed.

Nickel thin films were deposited from pure nickel targets by means of glow discharge Ar^+ plasma-sputtering at GNS Science in Lower Hutt, Wellington. Three different Ti6Al4V substrates were nickel-coated simultaneously in the sputtering chamber, obtaining an average thin film thickness of approximately 900 nm.

Thin films were further laser-treated in standard room conditions at the Photon Factory in the University of Auckland with a KrF excimer UV laser (Xantos XS, Coherent) of 248 nm emission wavelength and a pulse duration of 5 ns. The experimental assembly provides a beam with spatial profile of 6 x 3 mm and beam divergence of 2 x 1 mrad. The four parameters that could be varied included input power, mask size, repetition rate, and number of pulses. To ensure full treatment of the surface a rastering configuration was adopted, whereby the laser spot was scanned over the surface line by line ensuring that a fixed number of pulses per region was delivered following each laser pass. To compensate for any slight misalignment of the laser system and the material response, the distance between consecutive rastered lines was set to be 2 μm less than the size of the square mask, thus potentially giving rise to an overlapping of 1 μm on each side of the line. The complete laser parameters of the experiment are provided in table 1.

Table 1: Experimental laser parameters.

Base substrate	Ti6Al4V
Sputtered coating, thickness (nm)	Ni, 900
Mask size (μm)	50 x 50
Number of pulses per area (μm^{-2})	4×10^{-3}
Repetition rate (Hz)	500
Input power (mW)	46
Fluence per pulse (J/cm^2)	3.68
Rastering step (μm)	5
Vertical line separation (μm)	48

Surface treated samples were cleaned in water with detergent and blown-dried with ethanol, and subsequently contact angle measurements were performed with a KSV CAM 200 goniometer using a sessile drop technique assembly equipped with a CCD camera. Drops of water, ethylene glycol or diiodomethane were poured with a syringe onto the laser treated surfaces and the contact angle was measured using the CAM 200 software to fit the shape of the drop to a curve and calculate the angle via the intersection with a baseline that matches with the substrate. Due to sensitivity in the substrate recognition by the software, baselines had to be fit manually in some cases. Thirty values of contact angle were taken within the minute after the droplet was deposited and the average was calculated.

A FEI (Philips) XL30 S-FEG scanning electron microscope (SEM) was used to obtain surface images using secondary electrons. Scanning Probe Microscopy (SPM) images were taken with a cube corner nanoindentation tip (Hysitron TI950 TriboIndenter) by rastering the tip over the surface while exerting a very low force of $2 \mu\text{N}$. In this way, topographical information about the surface can be extracted.

3. Results

The effect of UV-laser treatment on surface morphology of nickel thin films can be observed in figures 1 and 2. Freshly sputtered nickel thin films show grainy cauliflower-like features homogeneously distributed throughout the surface, while laser-treated films display three distinct roughness levels.

The first one consists of periodic and relatively dense lines of approximately $3\text{ }\mu\text{m}$ thickness separated by a period of $50\text{ }\mu\text{m}$. The vertical dimensions could not be unmistakably estimated by SPM due to a combination of the height of the features, the intricate topography, and the piezo vertical limit, however recorded values suggest heights in excess of $2\text{ }\mu\text{m}$. The second roughness level is located between the dense lines and consists of periodic thin lines of $50\text{ }\mu\text{m}$ length separated by a period of approximately $5\text{ }\mu\text{m}$, and formed by the alignment of large melted and solidified droplets of roughly $500 - 700\text{ nm}$ size and $300 - 600\text{ nm}$ height. Crater/donut morphologies and dragging of the droplets can also be identified. The third roughness level corresponds to the region between the thin lines, which is mainly formed by melted and solidified droplets of $200 - 400\text{ nm}$ size and less than 300 nm height, either solid or crater-shaped.

Average contact angles measured during the first minute are plotted in figure 3 before and after laser treatment. On freshly sputtered nickel, measured contact angles are $41.5 \pm 4.2^\circ$ for water, $33.3 \pm 0.9^\circ$ for ethylene glycol, and $27.0 \pm 0.4^\circ$ for diiodomethane. The values following laser treatment are $128.2 \pm 6.2^\circ$ for water, $95.7 \pm 5.9^\circ$ for ethylene glycol, and $56.2 \pm 6.0^\circ$ for diiodomethane, which correspond to a twofold to threefold increase in contact angle. The effect of laser treatment can be observed in figure 4 for water, where angles in excess of 130° were measured.

4. Discussion

The roughness levels observed can be understood in terms of the laser interaction with the material surface. Under conditions of low energy deposition rates such as the ones found in nanosecond laser pulses, melting initiates heterogeneously from surfaces and crystal defects [29], and the extent of the heat affected zone can be estimated by the heat diffusion length into the material [30–32], which depends on its thermal diffusivity and the laser pulse duration. This would yield a heat-affected region of approximately 510 nm per pulse for nickel [33, 34], however the total extent of the heat affected region varies because it also depends on the number of pulses and the type of heat conduction [35].

Numerical models have predicted an increased mass flow rate at the edges of the irradiated zone as a consequence of a spatial plasma pressure variation [36], although other mechanisms such as explosive ablation can also generate crater morphologies with grooves of different depths depending on the laser

Figure 1: SEM micrographs of sputter-coated nickel thin film: a), b) before and c), d) after laser treatment.

machining parameters and ablation efficiencies [37–42]. Surface features such as droplets and the use of a moderate fluence $< 7 \text{ J/cm}^2$ [37] point towards pressure-induced melt displacement being the main mechanism behind the generation of the surface morphology, with some melt instability that results in droplet ejection and recondensation on the surface [43]. In this context,

Figure 2: SPM image of laser-treated nickel thin film, clearly displaying the two roughness levels shown in figure 1d).

Figure 3: Average contact angle before and after laser treatment for water, ethylene glycol and diiodomethane on nickel thin film.

the observed craters could be the consequence of bubble entrapment during melt pool solidification [44–46], or bubble formation at the interface between redeposited ejected droplets and the solid substrate [47]. The use of a 50 μm mask would therefore create the features observed experimentally, with 50 μm -period higher walls in parallel to the scanning direction and smaller periodic lines in perpendicular. These latter features arise as a consequence of

Figure 4: CCD camera image of sessile drop experiment for water on sputter-coated nickel thin film a) before and b) after laser treatment. The apparent contact angle measured after laser treatment is in excess of 130° .

the $5\ \mu\text{m}$ rastering laser step and are not to be confused with laser induced ripples, which are sub-wavelength structures that form as a result of laser interference [48–51].

Laser-induced micro- and nanostructuration induces an increase in the apparent contact angle which is independent on the polarity of probing liquid employed, and the ratio between the contact angles before and after laser treatment would rule out the existence a roughness-induced Wenzel state in favour of a Cassie-Baxter state [7, 52]. Even if a Wenzel state was generated due to the cauliflower-like morphology of freshly sputtered nickel thin films, values reported for nickel surfaces [53, 54] confirm that the laser treatment results in an actual increase in apparent contact angle in all cases. However, a change in surface energy as a consequence of laser treatment cannot be completely ruled out [18, 22], an effect that could change the hydrophobicity of the original smooth surface and give rise to a Wenzel state.

5. Conclusions

Ultraviolet laser was used to micro- and nanopattern a nickel thin film sputtered on Ti6Al4V. Three different roughness levels were identified, and two of them could be directly linked to the geometrical rastering parameters (mask size, rastering step). Topographical features such as droplets and craters were related to pressure-induced melt displacement and droplet melt ejection as a consequence of the laser conditions. A twofold to threefold

increase in contact angle was recorded for water, ethylene glycol and diiodomethane following laser treatment, a behaviour which can be explained in terms the micro- and nanopatterning of the surface.

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