

Direct patterning in sub-surface of stainless steel using laser pulses

Z. L. Li,^{1,*} T. Liu,¹ C. C. Khin,¹ A. C. Tan,¹ L. E. Khoong,¹ H. Y. Zheng,¹ W. Zhou²

¹ Singapore Institute of Manufacturing Technology, 71 Nanyang Drive, 638075 Singapore

² Precision Engineering and Nanotechnology Centre, School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang Avenue, 639798 Singapore

*zlli@SIMTech.a-star.edu.sg

Abstract: This paper reports for the first time on the direct creating microcavities in sub-surface of stainless steel using a single Nd:YAG laser pulse. The low peak power density is used in the process, which is in the order of 1 MW/cm². The formation of the microcavities in the sub-surface of stainless steel is an evidence of volume expulsion during laser-metal interaction. Direct patterning in the sub-surface of stainless steel is demonstrated by realizing a series of microcavities to form a pre-designed pattern. Potential applications of sub-surface patterning in metal, such as security marking, micro-heater, micro-insulator and micro-sensor, are discussed.

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OCIS codes: (220.4000) Microstructure fabrication; (140.3390) Laser materials processing; (140.3945) Microcavities; (160.3900) Metals

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1. Introductions

Direct laser writing and patterning inside transparent materials is a well known technology. Using this technology, sub-surface structures are realized by focusing a laser beam into a bulk transparent material. Depending on laser power density and material property, numbers of different laser technologies have been developed for various applications. Optical engraving of three dimensional figures is one of the typical applications [1]. In this technique, laser power density is controlled to be greater than the material damage threshold to induce microcracks [2], which results in an opaque portion and forms desired marks inside the transparent material. It has been found that focusing femtosecond (fs) laser into glass at power density above the refractive index change threshold of the glass results in an increase of refractive index about 0.04 at the focal point inside the glass [3]. The change in refractive index is the result of permanent structural change through densification [4] or formation of defects [5] caused by the high peak power of fs laser. This finding has led to wide investigations on direct laser writing waveguides and optical devices inside transparent materials [5,6]. When fs laser irradiating photosensitive Ag^+ doped Foturan glass at power density below the refractive index change threshold followed by thermal annealing, nano Ag particles are precipitated and confined at the focal point in the glass, which increases refractive index. Using this technique, optical grating [7] and waveguide [8] have been fabricated.

During the laser irradiation of opaque material, such as stainless steel, laser energy is absorbed at the surface and optical penetration depth l_a is the reciprocal of absorption coefficient α ($l_a = 1/\alpha$). The absorption coefficient of stainless steel is 5.45×10^5 /cm at 1064 nm [9], the optical penetration depth is 1.8×10^{-6} cm. Most metals have similar optical penetration depths in the order of 10^{-6} - 10^{-7} cm [10]. Therefore, sub-surface direct patterning in metals by focusing laser beam into the metals is not theoretically feasible. To our best knowledge, so far direct patterning in metal sub-surface by laser or any other techniques has not been reported. In this paper, direct creating of microcavity in the sub-surface of stainless steel using a single laser pulse is demonstrated. Based on the experimental observations, we suggest that the microcavity at sub-surface of metal could be generated by an extremely special drilling process. In normal metal drilling process, material is removed via vaporization and ejection of molten material induced by high peak power density. In our experimental conditions, low peak power density and single pulse were used. Thick melt pool and slow upward ejection speed might result in the ejecting molten material encountered rapid solidification on the half way of the ejection at the end of the laser pulse. As a result, a microcavity was formed at the bottom of the solidified melt pool. Patterns were realized by forming a series of microcavities at the sub-surface of stainless steel. After surface polishing of the laser markings on the surface, the pattern could only be revealed by non-destructive inspection techniques, such as x-ray imaging. The concealment of the patterns can be applied as security marking in automotive, aerospace and military industries.

2. Experimental

A Lumonics JK 702H Nd-YAG laser delivering maximum energy of 3 J with 0.5-20 ms pulse duration was used in the experiments. The pulse energy has near Gaussian distribution and was controlled to deliver a single pulse onto a specified location. The laser beam was focused with a lens of focal length 76.2 mm. In order to control the peak power density, the focal plane was set at different positions above the sample surface as shown in Fig. 1. The Z is the distance between the laser focal plane and sample surface. Z is zero when the laser beam focal plane is on the sample surface. An N_2 gas was employed through a coaxial conical nozzle to prevent possible oxidation reaction, but was controlled from blowing away the molten material. Austenite stainless steel with thickness of 1 mm was selected as sample. The sub-surface microcavities created by a series of single laser pulse were examined by scanning electron microscopy (SEM), optical microscopy, non-destructive computed tomography (CT) scan and x-ray imaging. To examine the melt pool, the sectioned samples were electrolytic etched in a mixture of oxalic acid (10 g) and H_2O (100 mL) at 6 V d.c. for approximately 30-40 seconds.

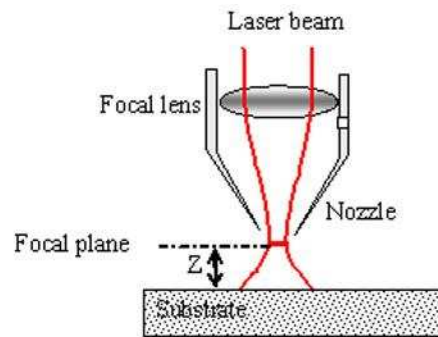


Fig. 1. Experimental set-up.

3. Results and discussions

The experiments were carried out at laser beam pulse energy of 0.43 J and pulse duration of 0.5 ms. The Z was increased from 1.5 mm to 3.1 mm. Increasing Z results in a larger beam radius on the samples surface, in turn the laser peak power density (which is the pulse energy divided by the product of the pulse duration and the cross-sectional area of laser beam) on the sample surface is reduced. Figure 2 shows optical microscopy top views of the samples after single laser pulse shot at various Zs. It was observed that ablation occurred and microcraters were formed when Z was in the range of 1.5 to 1.7 mm as shown in Fig. 2(a). The peak power density was calculated as 4.07 MW/cm^2 when Z was 1.7 mm. Figure 2(b) shows top view of the samples when Z was further increased to the range of 1.9-2.9 mm. Instead of microcraters formation, microbumps were observed. The sizes of the microbumps were gradually increased when Z was in the range of 1.9-2.5 mm. The peak power densities were calculated in the range of $1.90\text{-}1.22 \text{ MW/cm}^2$ (details are shown in following paragraph). The sizes of the microbumps were gradually decreased when Z was in the range of 2.7-2.9 mm. Although the beam radius on the sample surface was increased with Z, however, the energy at the beam peripheral was lower than melting threshold of the metal. Therefore, peak power density could not be calculated. Circular waves on the microbumps revealed the fluid flow motion encountered by the molten materials during the process. When Z was 3.1 mm, Fig. 2(c) shows that the laser pulse only generated a melt mark and molten material motion waves did not appear.

The experiments were also carried out at pulse energy altered from 0.5 to 0.17 J and pulse duration was 0.5 ms at $Z = 2.1 \text{ mm}$. The trend of microbumps sizes changing was the same as

Z change (not show here). This confirmed that the major effect of change Z on the sample surface was same as change laser energy. When peak power density was reduced by increasing Z or reducing pulse energy, the temperature raised in the sample was decreased. These led three levels of material response to a laser pulse shot: high speed molten material ejection (crater), middle momentum molten upward motion (microbump) and melt mark. The crater formation and melt mark are always observed in laser drilling and welding/melting process. However, the circular waves on the microbump are not common. Further investigations were focused on this phenomenon and effects on microstructures.

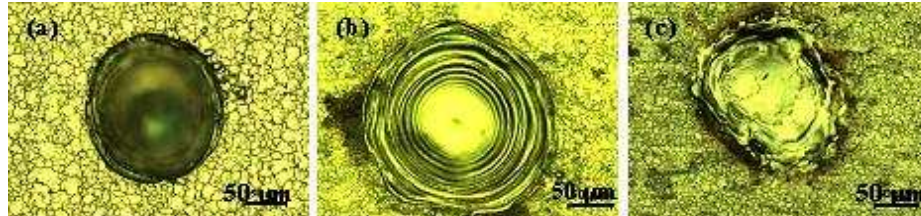


Fig. 2. Optical microscopy top view of samples after one laser pulse shot at various Z ranges: (a) 1.5-1.7 mm, (b) 1.9-2.9 mm, and (c) 3.1 mm. Figure 3 shows the SEM analyses of the top views of the samples irradiated at the Z range in Fig. 2(b). The melt spot diameters were measured to be 0.24, 0.26, 0.27 and 0.3 mm for Z = 1.9, 2.1, 2.3 and 2.5 mm, respectively. The peak power densities at each Z were calculated as 1.90, 1.68, 1.52 and 1.22 MW/cm², respectively. The sample irradiated at Z = 1.9 mm had relatively more sputtering, and the amount sputtering gradually decreased as the Z increased to 2.5 mm. Only small amount of sputtering was observed on the sample surface for Z = 2.5 mm.

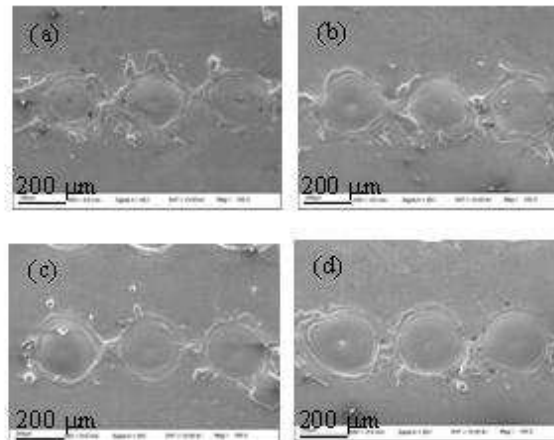


Fig. 3. SEM top views of laser irradiated surfaces at various Zs: (a) 1.9 mm, (b) 2.1 mm, (c) 2.3 mm and (d) 2.5 mm.

Figure 4 shows the longitudinal cross-sectional CT scan images of the samples irradiated at various Zs as shown in Fig. 3. Microcavities in the sub-surface were observed in these four tested samples. Some of the microcavities inside the sample were discrete. The longest microcavities were obtained at Z = 1.9 mm, and the average length was approximately 0.6 mm. The lengths of microcavities were decreased to approximately 0.2 mm as the laser focal plane was set further away from the sample surface at Z = 2.5 mm. These observations show that longer microcavities in the sub-surface were obtained at higher peak power density and accompanied with larger amount material ejection. The transverse cross-sectional CT scan images show the microcavities were circular (not shown here).



Fig. 4. Cross-sectional images of CT scan of microcavities in sub-surface of stainless steel with various Zs.

Figure 5 shows the cross-sectional micrograph of a stainless steel sample after the irradiation of a single laser pulse when Z was 2.5 mm (peak power density of 1.22 MW/cm^2). Figure 5(a) shows a clear boundary of the melt pool and Austenite growth direction. A bump was observed at the top surface with height of about $25 \mu\text{m}$. The bump diameter and depth of the melt pool was 0.3 mm and 0.35 mm , respectively. A conical shape microcavity was observed at the bottom of the melt pool and extended toward the laser beam incident direction. Figure 5(b) is an enlarged image of the region as indicated in Fig. 5(a). The surface of the microcavity was smooth and molten metal flow wave was observed at the top of the microcavity.

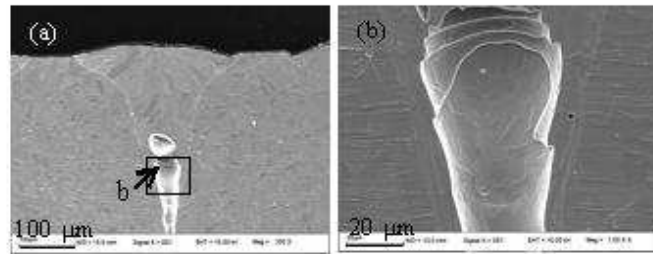


Fig. 5. Typical microcavity created in stainless steel, (b) enlarged images as indicated in (a).

The physical mechanisms of forming microcavity in the sub-surface of metal could be complex and are not fully understood yet. The process probably is an extremely special case of laser metal drilling. During a normal laser drilling process, immediately after a laser pulse started, the energy is absorbed at the surface of the material and the surface material is heated up. The energy is conducted into the bulk metal forming a melt layer [11] and the depth and temperature of the melt layer are depended on the peak power density. As the temperature increases, the material is removed by vaporization and molten material ejection. In generally, there are two common mechanisms could be involved in driving the molten material ejection. Firstly is the recoil pressure that induced during the vaporization of material. The temperature at surface is highest and decreases exponentially towards the depth. Vaporization at the surface generates a recoil pressure which increase with the surface peak temperature [12] and laser energy. When the recoil pressure exceeds the surface tension and hydrostatic pressure in the melt pool, molten material at the surface of the melt pool is expelled. As the pulse proceeds, the molten material is expelled from the ablation front along the wall [13] to form a microcrater. In our current work, the recoil pressure induced material ejection is unlikely the dominant mechanism for the formation of microcavities in the sub-surface of stainless steel. This is because the microcavity was observed at the bottom of the melt pool indicating that whole volume of the melt pool could have been forced upward. The second possible mechanism is the volume expulsion. After surface vaporization occurs, the surface may be cooled due to the high latent heat of vaporization. The vaporization process may result in the inversion of positive vertical thermal gradient into negative one [14,15]. In other words, the internal temperature is higher than that of at the evaporating surface. The sub-surface superheating leads to nucleation of gas phases in the melt pool. The nucleation rate of gas phase increases exponentially with temperature [16]. Figure 2 shows three phenomena occurred at various laser peak power densities. If the peak power density is sufficiently high,

e.g. $\geq 4.07 \text{ MW/cm}^2$ at $Z \leq 1.7 \text{ mm}$, the high superheating temperature results in large amount of gas nuclei. The shock wave at sub-surface pushes the high-momentum fluid ejection. A crater is generated by volume expulsions as shown in Fig. 2(a). If the peak power density is sufficiently low ($Z = 3.1 \text{ mm}$), the low surface temperature and slow vaporization rate produce a thick melt pool in the substrate. Figure 2(c) shows that the low-momentum fluid is re-solidified at the end of the pulse. In experimental conditions as shown in Fig. 2(b), the peak power density, e.g. $1.90\text{--}1.22 \text{ MW/cm}^2$ at the range of Z between 1.9 and 2.5 mm , was insufficient to generate a microcrater but was enough to drive the fluid upward ejection. However, the ejection flow velocity of the molten material can be slow [13,17,18]. The volume expulsion might not be complete at the end of the laser pulse due to the slow upward motion. The remaining molten metal was rapidly re-solidified on the half way of the ejection. As a result, microcavities were formed at the bottom of the melt pool as shown in Fig. 5. The location of the microcavity evidenced that entire volume of melt pool was expelled upward during a laser pulse. In the range of peak power density indicated in Fig. 4, higher peak power density (e.g. 1.90 MW/cm^2) resulted in a deeper melt pool with relative faster momentum motion speed, in turn, more molten materials were ejected and longer microcavities were formed. In the condition of lower peak power density (e.g. 1.22 MW/cm^2), shallower melt pool was formed with slower momentum motion speed. Shorter microcavities were formed near to the surface. The discrete microcavities observed in Fig. 4 may suggest that the initial flow velocities of the top and bottom portion of the melt pool were different due to temperature gradient. The former was faster and separated with bottom portion during ejection.

Table 1. Stainless steel grade 316 material properties^a

Property	Value	Unites
Vaporization point, T_v	3270	K
Density, ρ	8283	Kg/m ³
Specific heat capacity, C_p	468	J/kgK
Latent heat of melting, L_m	300	kJ/kg

^aFrom J. Appl. Phys. **72**(8), 3317 (1992).

Using a simplified approximation, which is a lumped heat capacity calculation, penetration velocity of the laser energy into the material can be estimated by assuming the heat flow is one-dimensional and all absorbed laser energy is used to induce phase change of the metal. As such, the penetrate velocity can be estimated by [19]: $V = P_0 / \rho [L_m + C_p(T_v - T_0)]$, where P_0 is peak power density. ρ and C_p are material density and heat capacity. L_m is latent heat of melt. T_v and T_0 are material vaporization and room temperatures respectively. Since volume expulsion was assumed to be the possible mechanism in forming the microcavities, partial nuclei may occur. The material temperature was assumed to be the vaporization temperature T_v . The material properties are listed in Table 1 [12]. The measured reflectivity of the metal was 66%. If incident pulse energy was 0.43 J , the absorbed energy was 0.15 J . The penetration (melt) depth in 0.5 ms was estimated as $0.24, 0.21, 0.19$ and 0.15 mm when Z changed from $1.9, 2.1, 2.3$ and 2.5 mm , respectively. It was considered that these simplest calculations could have overestimated the penetration depths, however, the melt depths of experiments were approximately $0.79, 0.65, 0.6$ and 0.35 mm respectively. It was more than 2 times greater than the estimated depth. This indicated that the heat penetration mechanism is much complex. Investigation of the physical mechanisms involved is still being carried on.

There are many potential applications of patterning of microcavities in the sub-surface of metal. Patterns constructed by a series of microcavities can be created inside a stainless steel

using laser pulses. Figure 6(a) shows the surface of a sample after laser direct patterning and sandpaper polishing. The ejected molten material and micro bumps were removed. The pattern could not be observed by naked eyes and only be revealed by non-destructive inspection techniques, such as X-ray imaging. Figure 6(b) shows x-ray image of the sample in which the pattern “SIMTECH” and “2009” were created inside the metal. The concealment of the patterns can be applied as security marking in automotive, aerospace and military industries.

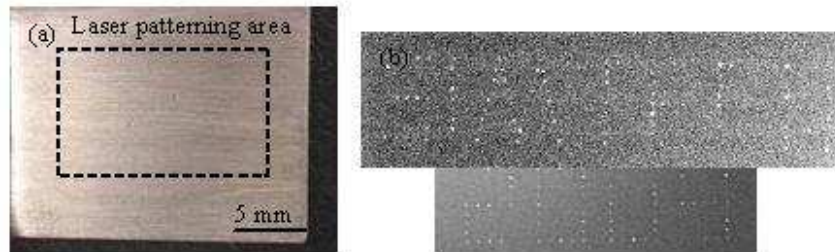


Fig. 6. (a) Optical microscopy of the sample after laser patterning and sandpaper polishing, (b) x-ray image of sub-surface patterning.

The porous metals are known to have many interesting combinations of physical and mechanical properties [20]. Figure 7(a) shows that closed porous surface structure was generated using a totally new fabrication technology - laser pulses. The closed porous surface structure can be patterned in many ways depending on the applications. Figure 7(b) illustrates examples of patterning may be generated inside a metal. The flexible changes in electrical resistivity [21], acoustic [22] and electromagnetic wave propagation and reflection properties [23] in a porous patterned metal may be designed to fabricate micro-devices, such as micro-heater, micro-insulator and sensors, *etc.*

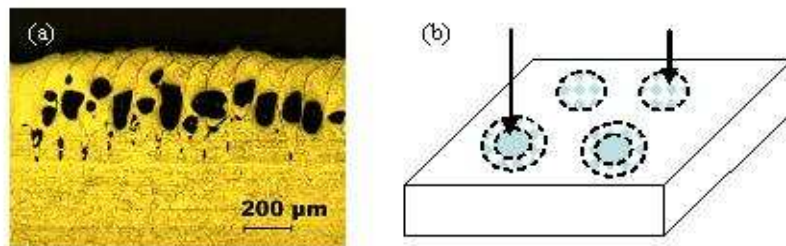


Fig. 7. (a) Closed porous surface structure generated by laser pulses; (b) illustration of patterning of the porous structure

4. Conclusions

It is demonstrated for the first time on direct creating of microcavities in the sub-surface of stainless steel using a Nd:YAG laser pulse. The laser pulse has low peak power density (order of 1 MW/cm^2), which generates a thick melt pool during the pulse. The molten material is driven upward with slow motion speed due to insufficient energy. The material ejection is stopped at the half way of the volume expulsion when the laser pulse ends. The remaining melt material rapidly solidified and created a microcavity at the sub-surface of the stainless steel. The length of microcavity can be controlled by changing the pulse peak power density, which determined the depth of melt pool and the ejection speed of molten material. Our experimental results show that the microcavity in the sub-surface can be formed in other metals, such as mild steels and titanium alloy.

Patterns constructed by serial microcavities in the sub-surface of stainless steel are directly generated. The patterns could be potentially useful in many applications. One example is patterning of the microcavities in the sub-surface of metal for security marking in automotive, aerospace and military industries. The concealment and certain depth of the patterns make the patterns exceptionally difficult to be removed and it is easily to be detected if the modifying of the patterned markings is attempted. Another example is patterning of closed porous structures on metal surface. The metal surface with closed porosity has been fabricated using our totally new fabrication technology - laser pulses. Surfaces with different densities of porosity and patterns can be directly created by programming the laser process parameters. Micro-heater, micro-insulator and micro-sensor may be fabricated by patterning the porous structures. The flexible and simple laser direct patterning in metal sub-surface may lead a new research direction.