The Influence of Laser Parameters on the Coloring Effect and Microstructure of Stainless Steel Surface

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Abstract. Nanosecond pulsed, green (532 nm) laser was used to irradiate SUS 304 stainless steel. Black, brown, gray, pink and other colors were obtained on the surface of polished stainless steel by adjusting the three process parameters: scanning interval ($L_s$), scanning speed ($v$) and defocusing distance ($D_{df}$). Scanning electron microscope (SEM) showed that there were three kinds of structures on the surface of stainless steel: columnar protrusions, droplets and cracks. The results indicated that the three process parameters affected the surface coloring effect and the microstructure formation of stainless steel by changing the total accumulated laser fluence in the scanning area.

1. Introduction
The rapid development of laser technology makes it widely used in metal processing [1-5]. Laser coloring is a kind of surface irradiation technology that makes use of pyrolysis reaction to make the metal matrix react with surrounding gas at high temperature and produces metal oxide [6]. A large number of metal and alloy materials form a variety of structures of oxidation growth layer after laser irradiation, many of which can be distinguished by the color layer can be used as a logo label and unique authenticity markings [6]. Lasers irradiated on metal surfaces are divided into nanosecond, picosecond and femtosecond lasers according to pulse width. There are many reports that three kinds of lasers can be used to fabricate color and surface structure on metal materials. In the past decade, femtosecond lasers have been widely used in the preparation of micro/nano structure on metal surface. The color and optical properties of metal surface have been changed by periodic micro-grating on metal surface and laser induced periodic surface structure (LIPSS) covered by nanostructure [7].

In this paper, we reported a study on coloring stainless steel surface using a nanosecond pulse laser. Black, brown and other color patterns were obtained on the surface of stainless steel by single irradiation with pulsed laser and different surface micro/nano structures are prepared. The influence of three process parameters include defocusing distance, scanning speed and the scanning interval on coloring and surface structure were analyzed. The results showed that their changes would lead to changes in the total accumulated laser fluence in the scanning area, thus affecting the color and morphology of the stainless steel surface.

2. Experiment
SUS304 stainless steel, an austenitic stainless was used for the study and the chemical composition
was shown in Table 1. The sample dimensions were 100 x 100 x 1 mm and the surface has been polished. Prior to the laser irradiation, the specimens were cleaned in deionized water. Ultrasound in ethanol was used to degrease the specimen surface and laser shortly irradiated after preparation.

The experiments were carried out by a Solid-state Laser (Draco-EP15A-SHG-S), which emits pulses at the central wavelength of 532 nm. Laser repetition rate (1-100 kHz) and pulse width (10-200 ns) can be adjusted according to different experimental requirements. The beam was focused onto a specimen using a f160 f-theta lens and the focal spot diameter is about 20 μm. The microstructure and topography of the sample surface irradiated by laser were observed by metallographic microscope (CX40M) and the microscopic morphology of the specimens was characterized by SEM (SU1510).

Table 1. The chemical compositions of the SUS 304 stainless steel.

| Element (Wt. %) | C   | Cr  | Ni  | Si  | Mn  | P   | S   | Fe       |
|----------------|-----|-----|-----|-----|-----|-----|-----|----------|
|                | 0.04| 18.35| 8.11 | 1   | 1.15 | 0.044 | 0.03 | Balance  |

3. Results and Discussion

3.1. Surface Colors and Metallographic Micrograph

The surface colors of the sample are shown in Figure 1. Many colors were successfully prepared on the surface of stainless steel by adjusting the process parameters. This is the same as many previous studies[8-10], which all obtained different colors by adjusting the laser parameters. Figure 2 shows the microscopic images of the above colors under a metallographic microscope. The black sample presented severe ablation under the microscope, and the observation area was in the form of focal mass. The observation area of the gray sample showed a large number of worm-like structures, and the entire substrate was in the grayish white color. The brown sample was composed of very dense colored dots. The path of laser irradiation can be clearly observed in bright brown, and the ablation area was the crescent. The dark red, light blue and pink samples showed a large number of cracks under the microscope.

![Figure 1](image1.png)

Figure 1. Different colors obtained on the surface of stainless steel by laser irradiation, (a) black (b) brown (c) silver gray (d) light brown (e) dark red (f) light blue and (g) pink.

![Figure 2](image2.png)

Figure 2. Microscope images of different colors, which corresponds to the seven colors shown in Figure 1 (The magnification is X500).
Different colors showed different morphologies under the microscope mainly because of their different process parameters, which leads to different degrees of reaction of the laser in the process of radiation samples, accompanied by molten, cooling, vaporization and other phenomena of the material[11].

3.2. Microstructure of Sample Surface

In order to further examine the differences of samples, the treated samples by laser and the untreated samples were observed in SEM. A mark of surface polish with untreated sample was shown in Figure 3(a). The corresponding color of Figure 3(b) is black. The SEM image shows that the dense columnar protrusion structures are formed on the sample surface. The illustration at the upper right is the magnification picture, which clearly indicates that these structures are composed of many small groups of particles. The SEM image of brown is shown in Figure 3(c). After laser irradiation, the structure of the sample surface is similar to that of water droplets spreading out. In the center of the irregular structure is the dense sand grain. Figure 3(d) presents a SEM micrograph of a pink sample, which is representative of the typical surface topography. Pervasive, interconnecting, through-thickness cracks were observed[6]. However, these cracks do not affect the appearance of color on the sample surface. In Guo's study[12], various surface microstructures also appeared on the surface of samples with different laser parameters.

Figure 3. SEM images showing surface microstructure of different samples, (a) untreated stainless steel (b) black sample (c) brown sample (d) pink sample.

The main reason for the above different microstructures is that the total accumulated laser fluence in the scanning region is different and the total accumulated fluence $\Phi$ is defined as [13]:

$$\Phi = \phi N = \frac{a^2 Ef}{\nu L_S}$$

(1)

Where the laser fluence is given by

$$\phi = \frac{2E}{\pi \omega_0^2}$$

(2)
The number of effective laser shots is

\[ N = \frac{\pi a \omega_0 f}{\sqrt{\frac{2}{v}}} \sqrt{\frac{\pi a \omega_0}{L_s}} \]  

(3)

where \( E \) is the pulse energy, \( f \) is the laser repetition rate, \( v \) is the scanning speed, \( L_s \) is the scanning interval and \( a \) is a correction factor due to the larger damaged area and therefore larger regions of laser overlap[14]. This correction factor was determined from semi-logarithmic plots used to determine the laser spot size[15]. The intra-line component of \( N \) encompasses the shot overlap within a local region, where \( f / v \) is the distance traveled between successive laser pulses in a single laser line. The inter-line component, in comparison, considers the geometrical overlap, \( a \omega_0 / L_s \), between successive laser lines. The process parameters of several colors obtained in the experiment are shown in Table 2. It can be seen that \( L_s \) and \( v \) in accordance with each color are different. According to the formula (1), \( \phi \) is directly related to \( a, E, f, L, s \) and \( v \), where the laser spot size (laser spot size is related to the distance from the focus) and \( L_s \) directly affect the overlap rate. Therefore, three parameters lead to different total accumulated laser fluence in the scanning area, thus affecting the microstructure of the coloring surface.

Table 2. The process parameters corresponding to different colors.

| Colors  | Scanning interval | Scanning Speed | Repetition Rate | Defocusing distance |
|---------|-------------------|----------------|----------------|-------------------|
|         | \( L_s \) (mm)    | \( v \) (mm/s) | \( f \) (kHz)  | \( Dd \) (mm)     |
| Black   | 0.005             | 50             | 25             | 0                 |
| Brown   | 0.02              | 400            | 25             | 0                 |
| Gray    | 0.005             | 100            | 25             | 4                 |
| Light brown | 0.005       | 500            | 25             | 0                 |
| Dark red| 0.005             | 1650           | 25             | 4                 |
| Light blue | 0.005        | 2400           | 25             | 4                 |
| Pink    | 0.01              | 350            | 25             | 6                 |

4. Conclusion

The work presented in this paper attests to the power of nanosecond pulsed laser surface treatment for stainless steel coloring. Three process parameters are studied in this paper, which are the defocusing distance, the scanning speed, and the scanning interval. The colors obtained on the sample surface have black, brown, gray, light brown, dark red, light blue and pink. At the same time, three kinds of microstructures include columnar protrusions, droplets and cracks, were observed in SEM. Experimental results show that the surface microstructure and coloring effect are closely related to three laser parameters, which can change the total accumulated fluence in the scanning area. The results provide a strong basis for further development and applications related to laser-assisted surface coloring. Unlike other chemical techniques used for metals surface coloring, the advancement of this process will allow for the nanosecond laser to rapidly process into specific microstructures with different optical colors on the steel substrate in a very simple fashion. Since the relationship among part of laser treatment parameters and stainless steel colors were discovered in this paper, it could be said that more colors can be found on the stainless steel surface as long as these parameters are properly adjusted in the following experiments. The applications of the proposed technique are far-reaching and could potentially impact the advancement of metal coloring industry.
Acknowledgments
The authors would like to gratefully acknowledge the financial support provided by Tianjin Municipal Education Commission, China under the Grant 2017KDYB20, Tianjin Natural Science Foundation Grant 17JCYBJC42400.

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