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Selective appearance of several laser-induced periodic surface structure patterns on a metal surface using structural colors produced by femtosecond laser pulses

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ABSTRACT

Ripples with a subwavelength period were induced on the surface of a stainless steel (301 L) foil by femtosecond laser pulses. By optimizing the irradiation fluence of the laser pulses and the scanning speed of the laser beam, ripples with large amplitude (∼150 nm) and uniform period could be obtained, rendering vivid structural colors when illuminating the surface with white light. It indicates that these ripples act as a surface grating that diffracts light efficiently. The strong dependence of the ripple orientation on the polarization of laser light offers us the opportunity of decorating different regions of the surface with different types of ripples. As a result, different patterns can be selectively displayed with structural color when white light is irradiated on the surface from different directions. More interestingly, we demonstrated the possibility of decorating the same region with two or more types of ripples with different orientations. In this way, different patterns with spatial overlapping can be selectively displayed with structural color. This technique may find applications in the fields of anti-counterfeiting, color display, decoration, encryption and optical data storage.

1. Introduction

In past several decades, laser induced periodic surface structures (LIPSSs) on the surfaces of various materials (including metals, semiconductors and dielectrics) with periods approximately equal to the laser wavelength have been investigated extensively by using pulsed lasers such as nanosecond and picosecond lasers [1–5]. In recent years, this study has been extended to femtosecond (fs) lasers and it has been known that LIPSSs (e.g., ripples) can be created on the surface of a material when the laser fluence near the ablation threshold of the material [6–14]. In most cases, the periods of LIPSSs are found to be slightly smaller than laser wavelength [8–14]. In addition, the ripples usually appear to be perpendicular to the polarization of laser light. The interference between the incident laser light and the surface scattered wave was proposed many years ago to explain the formation of LIPSSs [3]. Recently, it is suggested that the surface plasmon plaritons (SPPs) excited by fs laser irradiation play a crucial role in the formation of LIPSSs [11,14,15].

Very recently, LIPSSs with periods significantly smaller than laser wavelength have attracted considerable interest because of their potential applications [16–21]. While the LIPSSs with periods approximately equal to the laser wavelength are called low spatial frequency LIPSSs (LSFLs), the LIPSSs with periods much smaller than laser wavelength are referred to as high spatial frequency LIPSSs (HSFLs). Apparently, HSFLs induced by fs laser pulses cannot be interpreted by using the physical models described above [3,4]. So far, several mechanisms have been proposed to explain the formation of HSFLs induced by fs laser pulses, such as self-organization [22], second harmonic generation (SHG) [16], excitation of surface plasmon polaritons [23], and Coulomb explosion [24] etc. However, the actual physical mechanism responsible for the generation of HSFLs is still in debate. Very recently, we proposed a physical mechanism to explain the HSFLs formed on metal surface [25].

Due to their potential applications, periodic surface structures have attracted great interest in color display, anti-counterfeiting, decoration, sensing, and optical data storage etc [26–28]. For color display, LSFLs are usually employed because the periods of LSFLs fabricated by fs lasers with a central wavelength at 800 nm can effectively diffract white light and exhibit vivid structural color. For example, the use of LSFLs for color marking or color display has been successfully demonstrated [29–31]. In comparison, HSFLs may exhibit advantages in optical data storage because of their small periods. The subwavelength period and polarization dependent orientation of ripples makes them attractive for material and

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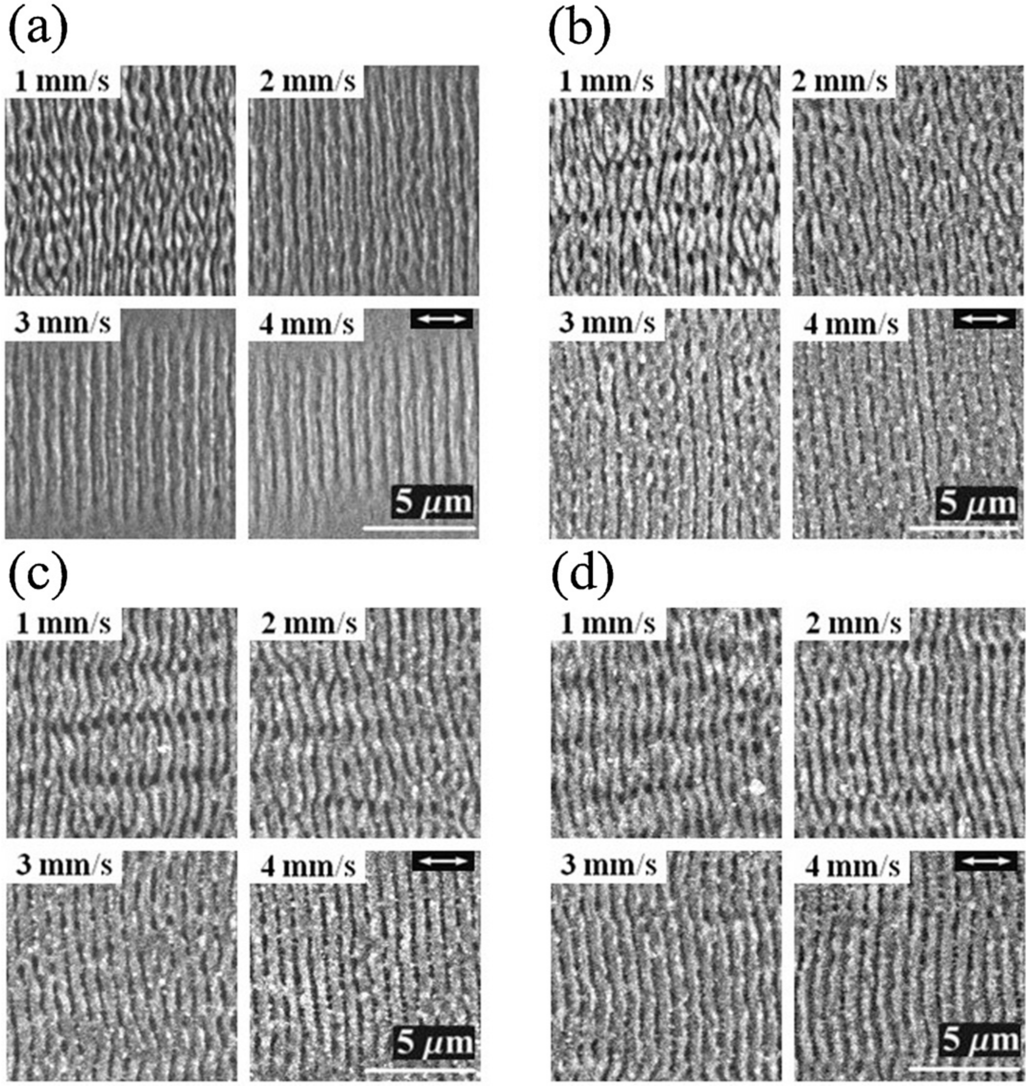

Fig. 1. SEM images of the ripples formed on the surface of the stainless steel by fs laser pulses with different fluences. (a) 0.16 J/cm^2, (b) 0.40 J/cm^2, (c) 0.64 J/cm^2, and (d) 0.80 J/cm^2. In each case, four scanning speeds, which are 1 mm/s, 2 mm/s, 3 mm/s, and 4 mm/s, have been employed to produce ripples. The arrows in the images indicate the polarization of fs laser light which is parallel to the scanning direction.

device applications. From the viewpoint of practical application, it is essential to produce deep and uniform ripples that can be extended to a large area. Therefore, the scanning of focused laser beam is necessary. In this case, the quality of the formed ripples depends strongly on the irradiation fluence of the laser pulses and the scanning speed of the laser beam. How to obtain high-quality ripples that behave as an efficient surface grating through optimizing these two critical parameters has become a major issue to be solved in the fabrication. In addition, it is desirable that the polarization dependence of the orientation of ripples can be utilized to selectively decorate surface with structural colors that are sensitive to incident light coming from a certain direction. More importantly, it is interesting to know whether the same area of a surface can be encoded with laser-induced ripples oriented in different directions.

In this article, we demonstrated the formation of deep and uniform ripples on the surface of a stainless steel foil by scanning fs laser with an optimized energy density and scanning speed. Vivid structural colors that span the entire visible spectrum were observed when illuminating the surface with white light and viewing at different angles. In addition, we showed that the selective decoration of the metal surface with ripples oriented in different directions could be used to display different patterns that are sensitive to illumination direction. Finally, we confirmed experimentally that the polarization dependence of ripples could be utilized to encode the same area with at least two types of ripples oriented in different directions. This polarization multiplexing may find applications in the fields of anti-counterfeiting, color display, decoration, encryption and optical data storage.

2. Experimental

In experiments, we used a Ti: sapphire fs amplifier (Legend, Coherent) that delivers 800 nm, 90 fs, and horizontally polarized pulses at a repetition rate of 1 kHz. The metal we studied was 301 L stainless steel that is widely used in industry. It was mechanically polished and washed in an ultrasonic cleaner with acetone. The surface roughness of the stainless steel sample was less than 10 nm. It was fixed on a three-dimensional motorized translation stage (7SC3-4, 7-Star) with a position accuracy of 625 nm. The diameter of the unfocused laser beam with a Gaussian profile is ~ 8 mm. The M^2 factor of the laser beam is ~1.16. It was focused normally on the sample by using a lens (SPX025AR.16, Newport) with a focusing length of 150 mm. The diameter of the laser spot on the sample surface was measured to be ~40 μm. A half-wave plate (10RP02-46, Newport) in combination with a polarizer (10P109AR.16, Newport) was employed to control the laser fluence. The surface morphology of the sample after fs laser treatment was examined by scanning electron microscope (SEM) (S-3700, Hitachi). In addition, the amplitude of the fabricated ripples was also characterized by atomic force microscope (AFM) (Cypher, Asylum Research). The structural colors originating from the diffraction of light by the formed ripples were observed by shining a white light from a light emitting diode (LED) on the surface of the sample.

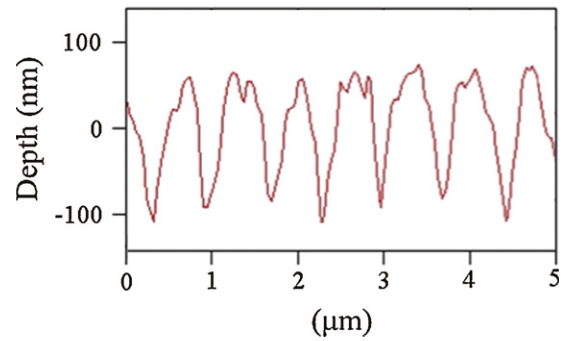

Fig. 2. AFM measurement showing the amplitude of the ripples fabricated by fs laser pulses with a laser fluence of 0.16 J/cm^2 and a scanning speed of 4 mm/s.

3. Results and discussion

3.1. Uniform ripples and vivid structural colors

From the viewpoint of practical application, it is desirable to fabricate deep and uniform ripples on metal surface. Based on experimental observations, it is found that two critical parameters for the fs laser have to be carefully controlled. They are the laser fluence and the scanning speed. In experiments, we have examined four typical laser fluences ranging from 0.16 to 0.80 J/cm^2. These values are just above the ablation threshold of stainless steel, satisfying the condition for generating ripples on metal surface. At each laser fluence, we varied the scanning speed from 1 to 4 mm/s with a step of 1 mm/s. It is estimated that the average number of pulses irradiated on the laser spot size ranges from 10 to 40. With different laser parameters, the surface of the stainless steel sheet with an area of 5 mm × 5 mm was processed by scanning the laser beam line by line with a step size of 50 μm. After processing, we used a white light source to illuminate the processed areas. It was found that the metal surface treated by fs laser exhibited vivid structural colors. By changing the irradiating or viewing angle, it was revealed that the structural colors spanned the entire visible spectrum.

We have compared the structure color of the surfaces processed with different laser parameters. It was found that the structural color became brighter and more vivid when we decreased the laser fluence and increased the scanning speed. In order to gain a deep insight into this phenomenon, we have examined by SEM the morphology of the surface treated with different laser parameters, as shown in Fig. 1. It can be seen that pronounced ripples with sub-wavelength periods could be obtained in all cases. In addition, it is noticed that the surface ripples appear to be coarse and irregular for large laser fluences and slow scanning speeds. In these cases, the ripples were segmented and the ablated material was re-deposited on the surface. In sharp contrast, clearer, longer, straighter and more uniform ripples were obtained by using a low laser fluence of 0.16 J/cm^2 and a high scanning speed of 4 mm/s. In this case,

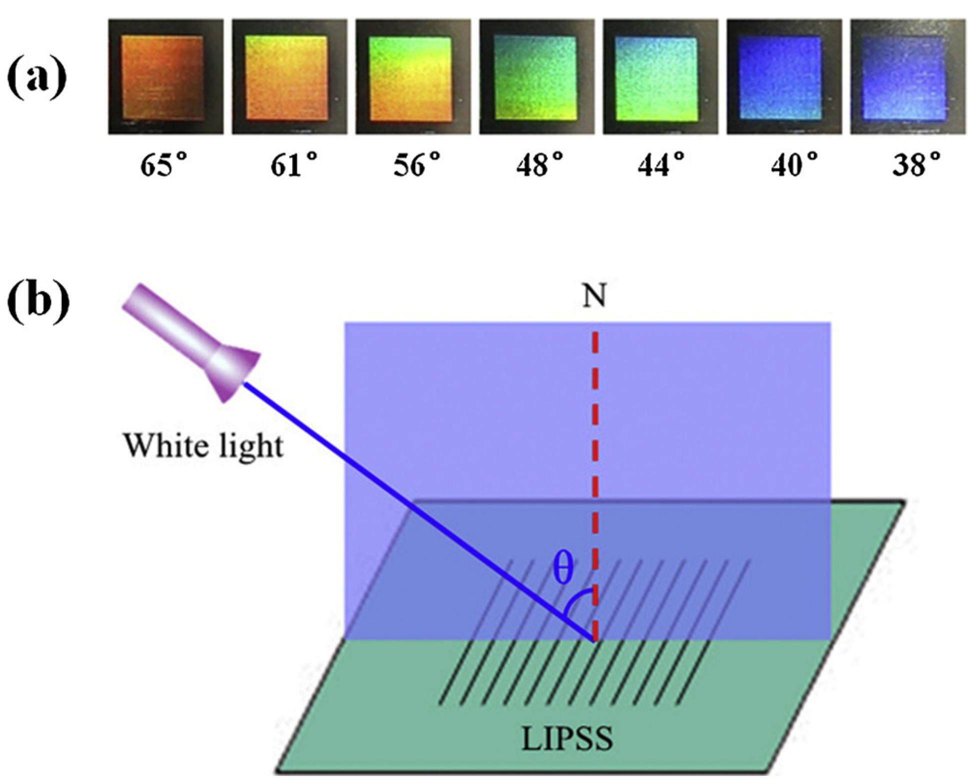

Fig. 3. (a) Variation of structural color observed by shining white light on surface modified stainless steel at different incidence angles. In each case, the incidence angle of the white light is indicated. The ripples on the surface of the stainless steel were produced by using optimum laser fluence and scanning speed. (b) Irradiation schematic.

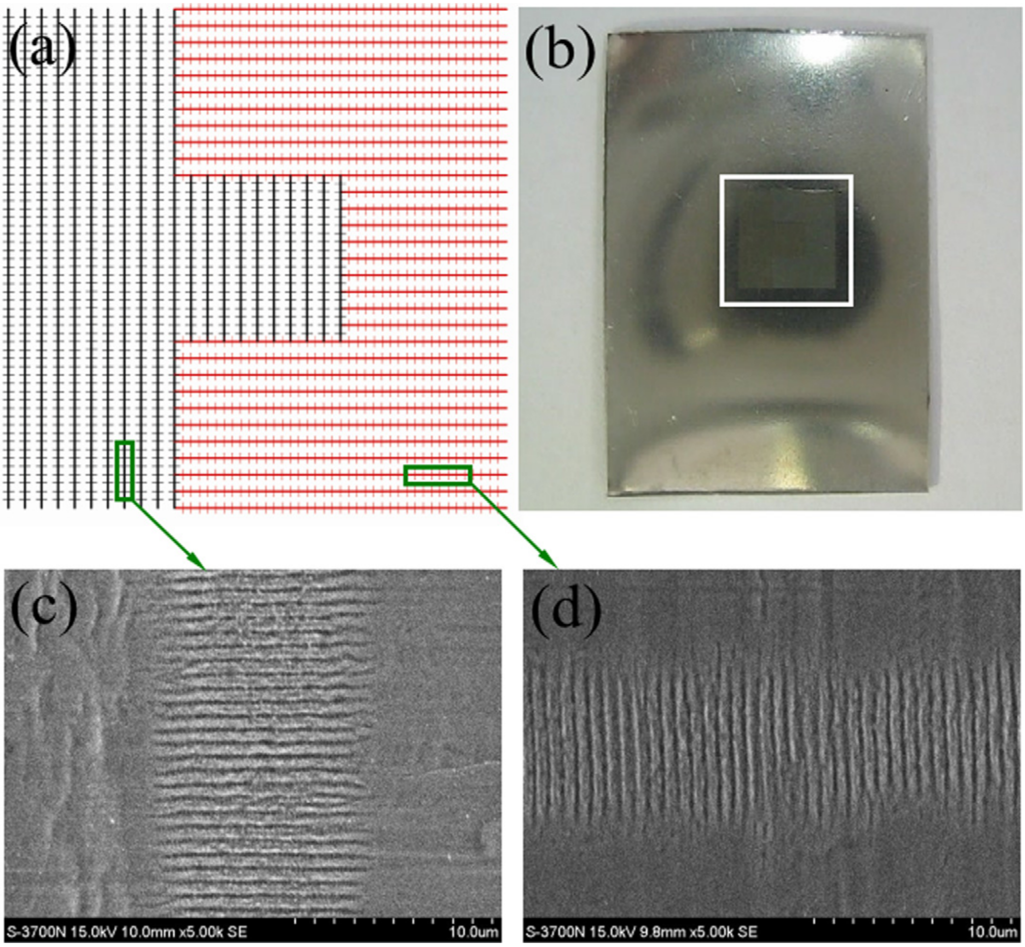

Fig. 4. (a) Schematic of a square region composed of two different patterns containing ripples oriented in two different directions. The black and red lines indicate the scanning traces of fs laser beam in the two patterns, respectively. The laser polarization was chosen to be parallel to the scanning trace. The short and thin lines perpendicular to the scanning trace represent the ripples produced by fs laser pulses. (b) Photograph of the patterns fabricated on the surface of the stainless steel by fs laser pulses. The SEM images of the ripples formed in the two regions are shown in (c) and (d), respectively. (For interpretation of the references to color in the artwork, the reader is referred to the web version of the article.)

amplitude of the created ripples was found to be ~150 nm by AFM measurements, as shown in Fig. 2. The formed ripples behave as an ideal reflective diffraction grating, rendering vivid structural colors. We have measured the diffraction efficiency of the fabricated ripples at 532 nm which act as a diffractive grating by using a solid state laser. It was found that the power of the zero-order diffraction was about 48% of the incident power. In comparison, the reflection of the smooth surface was determined to be about 63%. We also experimentally verified that such vivid structural colors could also be produced on the surfaces of other metals such as nickel, tungsten and aluminum alloy under suitable processing parameters.

Fig. 3(a) shows the variation of structural color resulting from the surface treated with the optimum laser fluence (0.16 J/cm^2) and scanning speed (4 mm/s). In this case, the viewing angle was fixed while white light was irradiated on the surface from left (or right) side with different incidence angles. A schematic showing the surface of the stainless steel, the alignment of the ripples, the incidence plane and angle of the white light is presented in Fig. 3(b). Since the laser polarization was chosen to be parallel to the scanning direction (which is horizontal), ripples perpendicular to the scanning direction (i.e., oriented in vertical direction) were created. As white light was irradiated on the surface from up (or down) side, the structural colors disappeared completely. It implies that the ripples were consistently oriented with respect to each other. For other processing parameters, the diffraction efficiency from the ripples degraded to some extent and the structural color was not vivid because long ripples with the same orientation were not obtained, as can be seen in Fig. 1. Consequently, the complete disappearance of structural color did not occur when white light is incident on the surface from up (or down) side due to the degradation in the alignment of the ripples.Essentially, structural colors mainly originate from basic optical processes such as thin-film interference, multilayer interference, diffraction grating, photonic crystals, light scattering, and so on [32,33]. In our case, the structure colors originate from periodic ripples that act as a reflective diffraction grating. Constructive interference between the reflected beams of light at specific angles for a given wavelength and not for other wavelengths results in the creation of structural colors. So we can easily understand why the surface treated with the optimum processing parameters can exhibit vivid structural colors when the incidence plane of white light is perpendicular to the ripples. In this case, the fabricated ripples are regularly aligned in the same direction. When the incidence plane of white light is parallel to the ripples, the diffraction condition is not fulfilled and no structural color was observed.

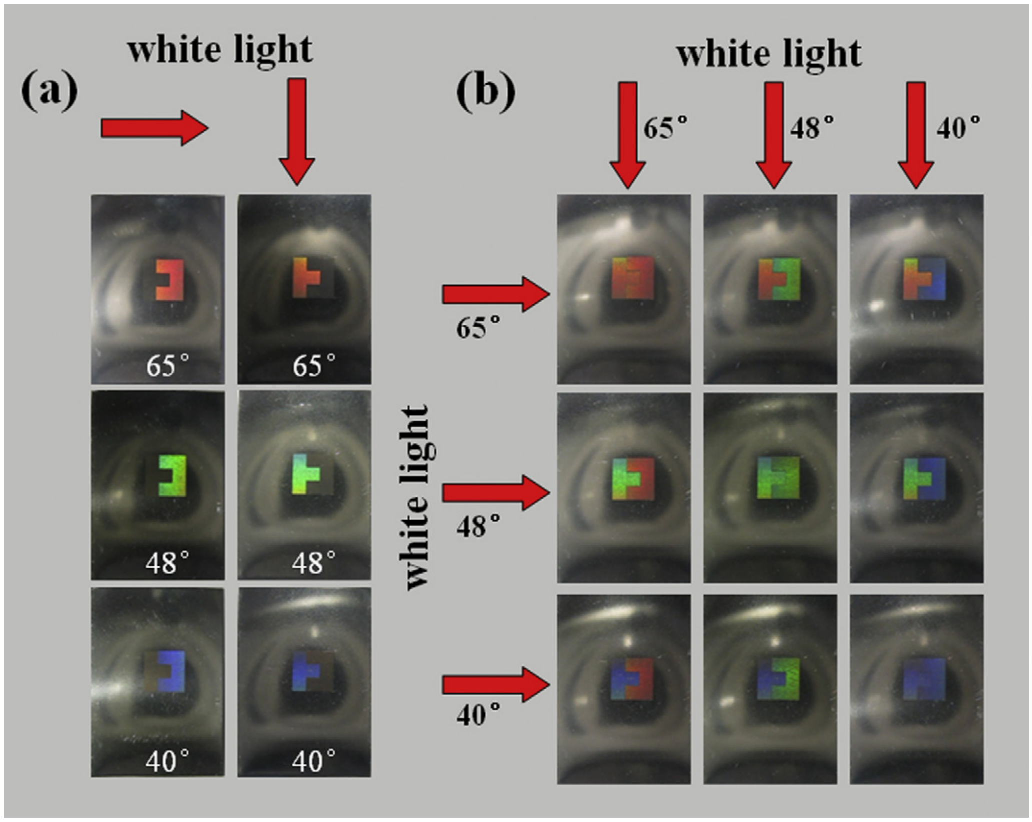

Fig. 5. Selective display of the two patterns separately (a) and simultaneously (b) with different structural colors generated by irradiating white light on the surface of the laser-treated area. In each case, the incidence angle of the white light is indicated. Different structural colors can be observed by changing the incidence angle of the white light. Both patterns exhibiting same or different structural colors can be displayed by simultaneously irradiating the laser-treated area with white light at different incidence angles from two perpendicular directions, as indicated by the red arrows. (For interpretation of the references to color in the artwork, the reader is referred to the web version of the article.)

3.2. Selective display of different patterns without spatial overlapping

Obviously, the structural colors originating from the ripples induced by fs laser on metal surfaces may be applied to color display, decoration, anti-counterfeiting, optical data storage and so on. As an example, we demonstrated experimentally the selective display of two patterns without spatial overlapping. As depicted in Fig. 4(a), we decorate a square region on the surface of 301 L stainless steel with surface ripples. The square region is divided into two regions in which the ripples are designed to be oriented in horizontal and vertical directions, respectively. In order to achieve best diffraction efficiency, the ripples on the surface were induced by scanning fs laser with an irradiation fluence of 0.16 J/cm^2 and a scanning speed of 4 mm/s. The spacing between two neighboring scanned lines is chosen to be 50 μm. Therefore, the horizontal ripples in the left region were produced by scanning fs laser with vertical polarization vertically. Similarly, in the right region, horizontally polarized laser resulted in vertical ripples. The photograph of a fabricated sample is shown in Fig. 4(b). We can easily identify the square region which has been processed by fs laser. Without special attention, however, it is not easy to distinguish the left and right regions which have been decorated with ripples aligned in different directions. The SEM images of the fabricated ripples in the left and right regions are shown in Fig. 4(c) and (d), respectively. It can be seen that the ripples in these two regions are perpendicular to each other. When white light is irradiated on the surface from left or right side with different incidence angles, the right region exhibits vivid structural color while the left region remains to be dark. The situation is reversed when white light is irradiated from up or down side. Red, green and blue colors can be observed by changing the viewing or the irradiating angle, as shown in Fig. 5(a). When we chose to irradiate white light from both directions with different incidence angles, a combination of left and right patterns decorated with different structural colors can be displayed, as shown in Fig. 5(b).

3.3. Selective display of different patterns with spatial overlapping

As described above, we can induce uniform ripples oriented in different directions in different regions on metal surface by utilizing the polarization dependence of the ripple orientation. Similar work has been carried out by Dusser et al. who have successfully demonstrated the use of polorization dependent ripples for color marking [29]. In this case, however, there exists only one type of ripples in a macroscopic region. Thus, it is interesting to know whether two or more types of ripples can be produced in the same macroscopic region. If possible, this would result in the possibility of selective display of different patterns that are spatially overlapping.

In the experiments described above, the surface grating is not formed by long ripples that extend to the whole region. Instead, it is formed by arrays (i.e., rows or columns) of short ripples with a length of ∼10 μm. The spacing between the two neighboring rows (or columns) was chosen to be 50 μm. Thus, there exists an untreated area with a width of ∼40 μm in between the two neighboring rows (or columns). It is verified, however, that

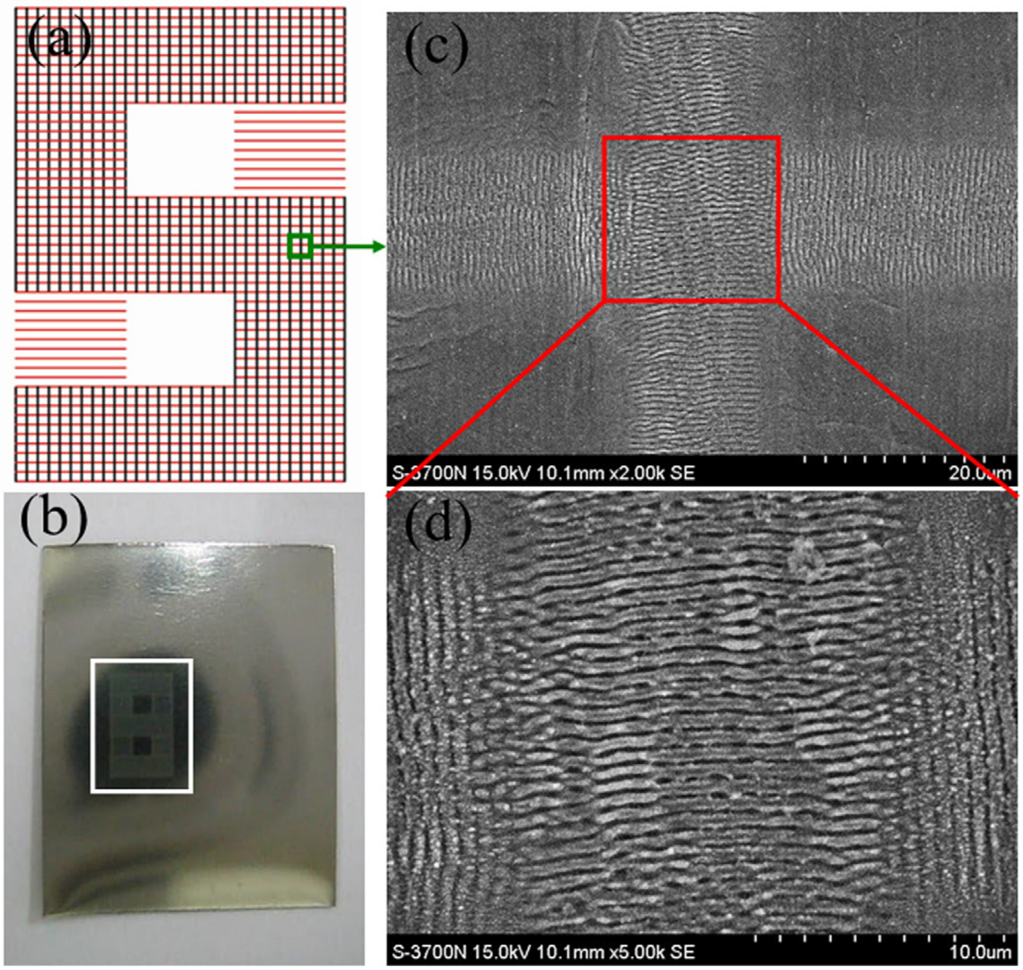

Fig. 6. (a) Schematic of a structure designed for selectively displaying two symbols (5 and 8) with spatial overlapping. The black and red lines indicate the horizontally and vertically scanned lines, respectively. The laser polarization was chosen to be parallel to the scanning direction. (b) Photograph of a structure fabricated on the surface of the stainless steel. (c) SEM image of the intersection of horizontally and vertically scanned lines. (d) The magnified image of the selected region in (c). (For interpretation of the references to color in the artwork, the reader is referred to the web version of the article.)

high diffraction efficiency can still be achieved by using such discrete surface grating. This feature offers us the opportunity to introduce ripples with a different orientation in the untreated area. It implies that one can encode the same macroscopic region with two or more types of ripples (with different incident polarizations).

In order to demonstrate this idea, we have chosen to display two symbols (5 and 8) that are spatial overlapping, as schematically shown in Fig. 6(a). The red and black lines indicate the horizontally and vertically scanned lines in the laser-treated area, which display symbol 8 and symbol 5 respectively. The laser polarization was chosen to be parallel to the scanning direction. It is obvious that the laser-treated area of symbol 5 contains two types of scanned lines (or two types of ripples) while some parts of symbol 8 contain only one type of scanned lines. The processing parameters were chosen to be the same as those described above except that the spacing between two neighboring scanned lines was increased to 70 μm. The photograph of a fabricated sample is shown in Fig. 6(b).

The SEM image showing the surface with a cross formed due to horizontally and vertically scanned lines is presented in Fig. 6(c). A magnified image of the intersection region of the cross is given in Fig. 6(d). In experiments, we first did horizontal scanning followed by vertical scanning in selective regions. In Fig. 6(d), it is noticed that the intersection region exhibits well the horizontal lines while the vertical lines are almost completely smeared out. In Fig. 7, we show the appearance of vivid structural colors which display two different symbols when white light was irradiated on the surface from two perpendicular directions. As expected, one can see either symbol 8 or symbol 5 when the sample was illuminated from different directions. In both cases, the structural color can be changed by varying the irradiating or viewing angle.

Apparently, the technique described above can be easily applied to anti-counterfeiting. There are many advantages of this technique over the conventional anti-counterfeiting techniques. First, the copyright patterns consist of periodic ripples produced on material surface by direct fs laser writing instead of sticky security label. So, it is durable, stable and difficult to duplicate. Moreover, it can present vivid structural colors in the same area or in different areas by irradiating white light from different directions. In addition, the structural colors can be varied remarkably by changing the irradiating or viewing angle. The copyright patterns produced by this technique are simple to observe and effective against unauthorized reproduction. All these unique properties are very attractive in anti-counterfeiting field and have potential applications in many other fields.

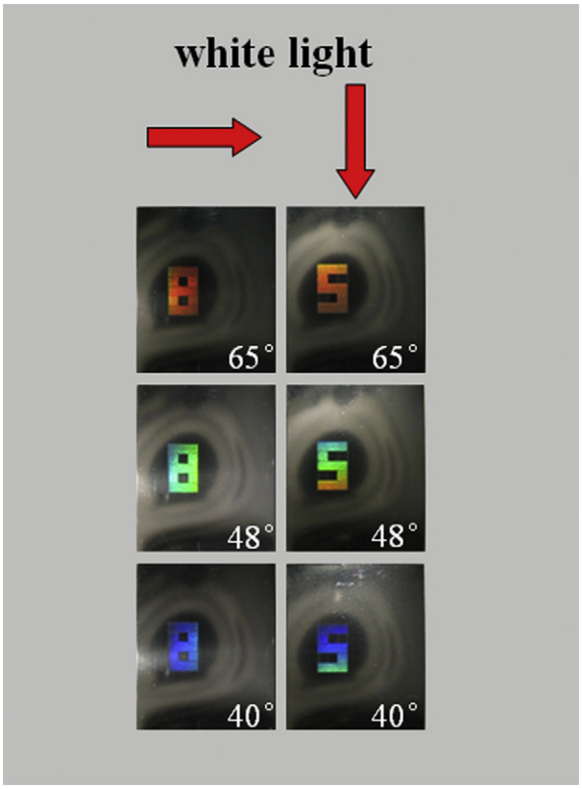

Fig. 7. Selective display of two symbols (5 and 8) with spatial overlapping by irradiating white light form two perpendicular directions, as indicated by the red arrows. In each case, the incidence angle of the white light is indicated. (For interpretation of the references to color in the artwork, the reader is referred to the web version of the article.)

4. Conclusion

In summary, we have demonstrated that metal surface can exhibit vivid structural color in the visible spectrum region under illumination of white light by using fs laser surface structuring technology. It is revealed that deep and uniform ripples that act as an efficient diffractive grating can be produced on metal surface by optimizing the irradiation fluence of laser pulses and the scanning speed of the laser beam. We show that selective display of patterns without spatial overlapping can be realized by utilizing ripples oriented in different directions. By employing arrays of short ripples instead of long ripples as diffractive gratings, we verify that the selective display of different patterns with spatial overlapping can also be achieved by decorating (or encoding) the same region with two types of ripples (or laser polarizations). Our technique provides a simple, effective, controllable approach to creating artificial surface structures on metal surface. Apart from anti-counterfeiting, the unique properties of this technique may find applications in many other fields.

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