Paper:

Fabrication and Control of Fine Periodic Surface Structures by Short Pulsed Laser

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Ultrashort-pulsed laser irradiation is a more efficient approach to the fabrication of fine surface structures than traditional processing methods. However, it has some problems: the equipment expenses usually increase as the pulse shortens, and the process principle has not been clarified completely, although the collisional relaxation time (CRT) is assumed to be a major factor. In this study, a 20-ps pulsed laser was employed to fabricate nanometer-sized periodic structures on a stainless steel alloy, SUS304. The pitch length of the fabricated fine periodic structures was similar to the laser wavelength, and the results suggested that periodic structures could be fabricated within a limited range of the laser fluence. In order to expand the effective fluence range (EFR) and to control the pitch length, laser irradiation was carried out with different workpiece temperatures and the laser wavelengths. In this way, CRT was extended and EFR was expanded by cooling the workpiece, and the pitch lengths were approximately equal to the laser wavelengths. As a result, two things were found: it is easier to fabricate the fine periodic structures by cooling the workpiece, and it is possible to control the pitch length of the fine periodic structures by changing the laser wavelength.

Keywords: short-pulsed laser, collisional relaxation time, effective fluence range, temperature, laser wavelength

1. Introduction

In recent years, energy saving, high functionality, and high value manufacturing have been demanded by a variety of manufacturers, so many studies are being done in order to meet these demands. Most light and chemical reactions occur on the surfaces of materials, and current research has revealed that fine surface structures can provide materials with new functionalities [1], such as improved optical functions [2], wettability control [3], and improved friction characteristics [4]. As a result, many researchers have been trying to fabricate such structures to attain desirable functions. For the creation of these functional surfaces, cutting, electric discharge machining, and photolithography have been widely studied, but these methods have their disadvantages, including long processing times, complicated processes, and difficulty in processing three-dimensional shapes. In this study, ultrashort-pulsed laser irradiation was adopted to solve these problems. Long-pulsed or continuum wave lasers remove material by heating and evaporating the irradiated matter [5]. On the other hand, ultrashort-pulsed lasers enable the fabrication of fine structures via laser irradiation alone, without thermal effects [6]. The principle of fine structure fabrication via this method has not been clarified completely, but it has already been reported that periodic structures are fabricated on the workpiece surfaces when the laser pulse duration is shorter than the collisional relaxation time (CRT) [7]. CRT has been assumed to be a major factor in surface structure fabrication in many studies, so mainly ultrashort-pulsed lasers have been used. On the other hand, as the pulse duration becomes shorter, equipment cost increases dramatically and laser irradiation becomes more unstable. In industry, therefore, it is better to use as long pulse-duration laser, as fine structures can be fabricated. Nevertheless, long pulse-duration laser increases thermal effects and makes fabricating fine structures difficult.

The objective of this study is to evaluate the potential of fabricating nanometer-sized periodic structures (hereinafter referred to as nanostructures) by using a short-pulsed laser process and to develop the process to fabricate clear and sharp structures. To do this, the short-pulsed laser was employed. The pulse duration used was 20 ps, relatively long within the category of ultrashort-pulsed laser. As the workpiece material, a stainless steel alloy, SUS304, was employed since it is a commonly-used industrial material often used for molds and mechanical parts. It is advantageous in industry to fabricate functionality-giving nanostructures on such a widely-used material.

First, basic experiments were conducted with short-pulsed laser to ascertain the conditions under which nanostructures can be fabricated.

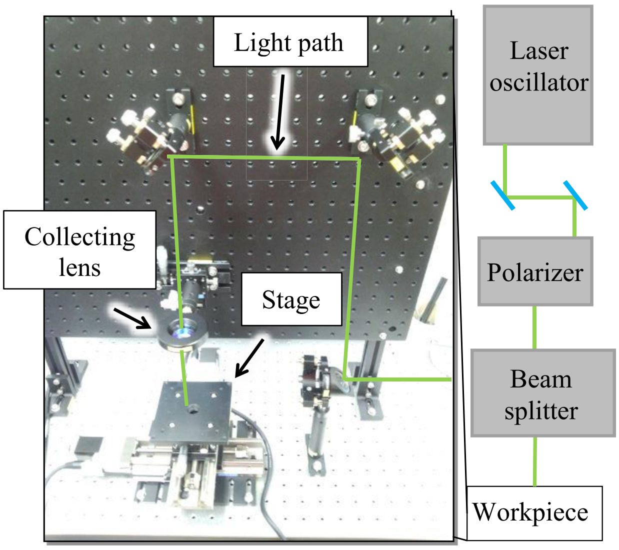


Fig. 1. Short-pulsed laser process unit and optical system.

Table 1. Laser irradiation condition.

Wavelength	1064 nm
Pulse duration	20 ps
Frequency	50 Hz
Beam spot size	2500 μm
Irradiation number n	1–1000 shot(s)
Energy density E_d	0.011 – 0.296 J/cm^2
Workpiece	SUS304
Unprocessed Ra	0.182 nm
Material temperature	293 K

Second, low-temperature environment and short-wavelength laser experiments were conducted, and the effects of heat and laser wavelength were evaluated to develop the short-pulsed laser process.

2. Fabrication of Fine Periodic Surface Structures Via Short Pulsed Laser

Basic experiments were conducted using the 20-ps pulsed laser, which was expected to be the longest pulse duration that could fabricate fine structures. If the pulse duration is much longer than the CRT, which depends on the material but is generally about 10 ps, thermal effects increase and structures are obviously melted.

2.1. Methodology

A picosecond laser oscillator (EKSPLA, PL 2250-50-P20) was used in this experiment, the setup of which can be seen in **Fig. 1**. Laser irradiation conditions are listed in **Table 1**. SUS304, a widely-used industrial material, was applied to provide wettability and to improve the friction property. Laser pulses with a Gaussian beam profile were directed at a fixed point on the workpiece, and the irradiation number n and energy density E_d were changed.

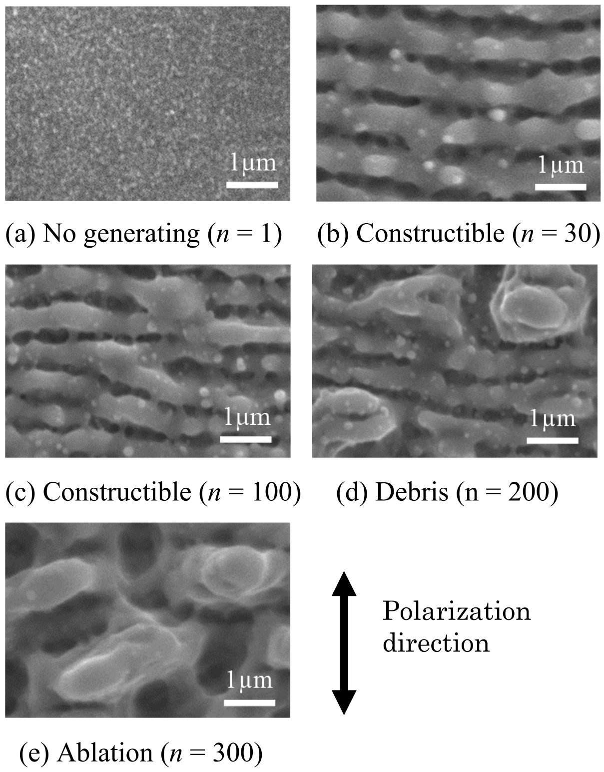


Fig. 2. SEM image of the irradiation surface with E_d= 0.296 J/cm^2.

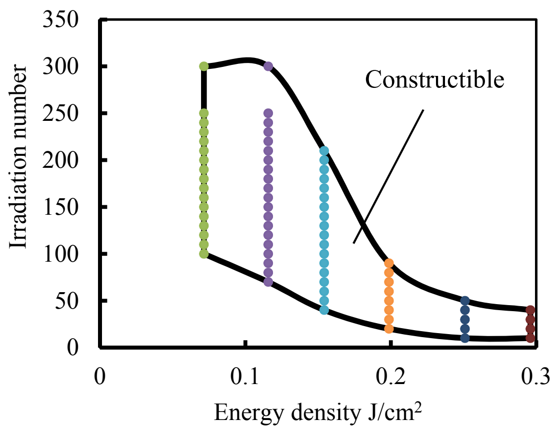


Fig. 3. Relationship between n and E_d when structures were fabricated.

2.2. Results

The possibility and conditions of nanostructures fabricated using short-pulsed laser irradiation were considered. **Fig. 2** illustrates the scanning electron microscope (SEM) image of the irradiated surface with $E_d = 0.296$ J/cm^2. With $n = 1$, structures were not generated due to the low irradiation count. With $n = 30$, fine periodic structures were seen perpendicular to the direction of polarization, though debris thrown about by the irradiation was deposited on the structures when $n = 100$ and 200. Finally, the structures melted and disappeared when n was increased to 300. As indicated by these figures, short-pulsed laser is able to fabricate fine periodic structures perpen-

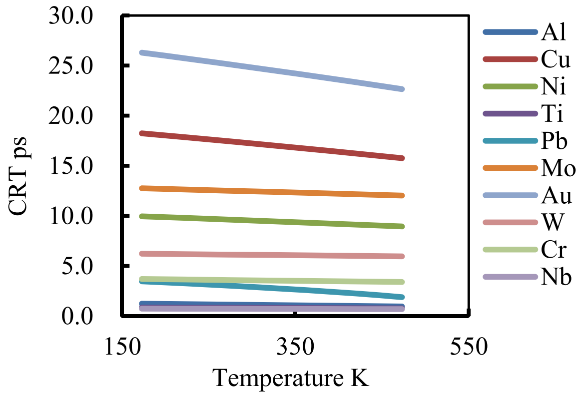


Fig. 4. Collisional relaxation time.

dicular to the direction of polarization under several conditions. On the other hand, structures lacked sharpness and seemed to be affected by heat.

Figure 3 shows the relationship between n and E_d when fine periodic structures were fabricated perpendicular to the polarization direction. This result suggests that there is an effective range for structure fabrication in terms of irradiation number and energy density, defined as an effective fluence range (EFR). This result also shows that the irradiation number necessary for fabricating fine periodic structures decreases with an increase in energy density. It suggests that excessive irradiation number and energy density cause the electric field intensity to increase and periodic structures to disappear due to the attachment of significant amounts of debris to the surface.

3. Controlling Fine Periodic Surface Structures by Changing the Wavelength and Temperature

CRT is the time from the start of rise in electron temperature to the equilibration of the temperature between electron and lattice [8]. It is assumed to be a governing factor in whether the process phenomenon is thermal or athermal. Laser with a relatively long pulse duration close to CRT needs high energy to fabricate fine structures, and its heat will melt structures. CRT is expressed as follows [7]:

$$\mathrm{CRT} = C_e' \frac{T_e}{\gamma} \quad \text{(1)}$$

$$T_e^2 = 2\frac{C_i}{C_e'}(T_m - T_0) \quad \text{(2)}$$

where C_e' is the electronic specific heat, T_e is the electron temperature, γ is the electron and photon bonding parameter, C_i is the lattice specific heat, T_m is the melting point, and T_0 is the lattice ion temperature before irradiation. Based on Eqs. (1) and (2), the relationship between CRT and material temperature is shown in **Fig. 4**. This suggests that CRT is affected by the material temperature, so it is expected that CRT can be extended by cooling the material. On the other hand, it is expected that shortening the wavelength of the laser from 1064 nm to 532 nm decreases the energy required for the fabrication of nanostructures. This is because a shorter laser wavelength increases its incident energy, and structures will be sharpened under low temperature with a short wavelength since it has been reported that the pitch length of a structure fabricated by laser irradiation is close to the laser wavelength. Therefore, the effects of material temperature and laser wavelength were evaluated through experiments.

Table 2. Laser irradiation condition.

Wavelength λ	532, 1064 nm
Pulse duration	20 ps
Frequency	50 Hz
Irradiation number n	1–1000 shots
Energy density E_d	0.002–6.72 J/cm^2
Workpiece	SUS304
Material temperature	223 K, 293 K

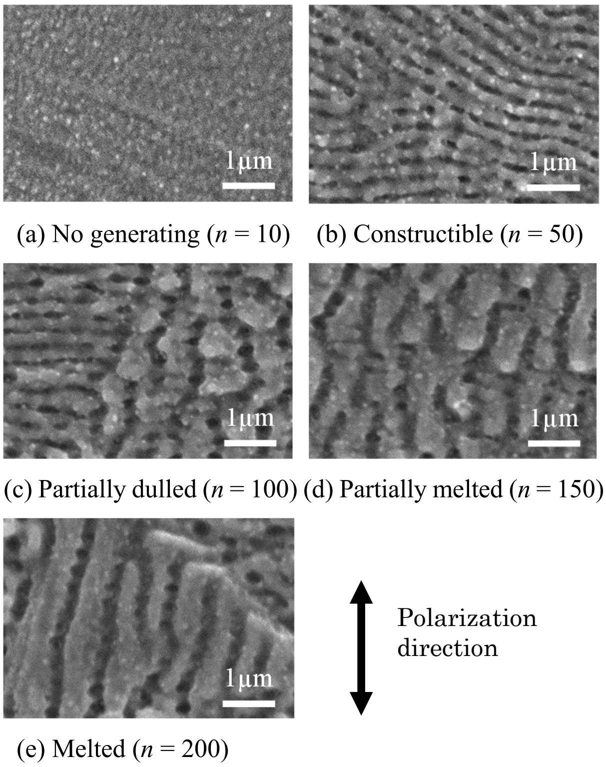
3.1. Methodology

Experiments were conducted using a device similar to that in the previous section. **Table 2** shows the laser irradiation conditions. Laser with a wavelength of 532 and 1064 nm was beamed at a fixed point on the workpiece, the temperature of which was set at 223 K and 293 K before irradiation.

3.2. Results

Figure 5 shows examples of SEM images of the irradiated surface with $\lambda = 532$ nm and $E_d = 0.0042$ J/cm^2 at 293 K. When $n = 10$, structures were not generated due to low energy density and low irradiation number. With $n = 50$, a fine periodic structure was seen, but it was not perpendicular to the polarization. It is considered that one cause may have been the crystal grain boundary, and this will be the subject of future research. With $n = 100$, structures had lines parallel to the direction of polarization, and the structures were partially or completely destroyed due to melting when n was increased to 150 and 200. As indicated in these figures, it was confirmed that a 532 nm-wavelength laser is also able to fabricate fine periodic structures under several conditions. **Fig. 6** is a TEM cross-sectional view of the irradiated surface with $\lambda = 532$ nm and $E_d = 0.004$ J/cm^2 at 293 K. Structures are shown to have a pitch length of close to 400 nm and a depth of about 300 nm. On the other hand, there is some oxide film, which means that the surface melted and oxidized as thermal effects.

Figure 7 shows examples of SEM images of the irradiated surface with $\lambda = 1064$ nm and $E_d = 2.83$ J/cm^2 at 223 K. This result shows that structures were constructed and the debris was gradually deposited on them, and then ablation occurred and the structures were destroyed as the irradiation number increased, similar to the phenomenon observed at 293 K. **Fig. 8** shows examples



(a) No generating ($n = 10$) (b) Constructible ($n = 50$)
(c) Partially dulled ($n = 100$) (d) Partially melted ($n = 150$)
(e) Melted ($n = 200$)

Fig. 5. SEM image of the irradiation surface with $\lambda = 532$ nm, $E_d = 0.0042$ J/cm^2 at 293 K.

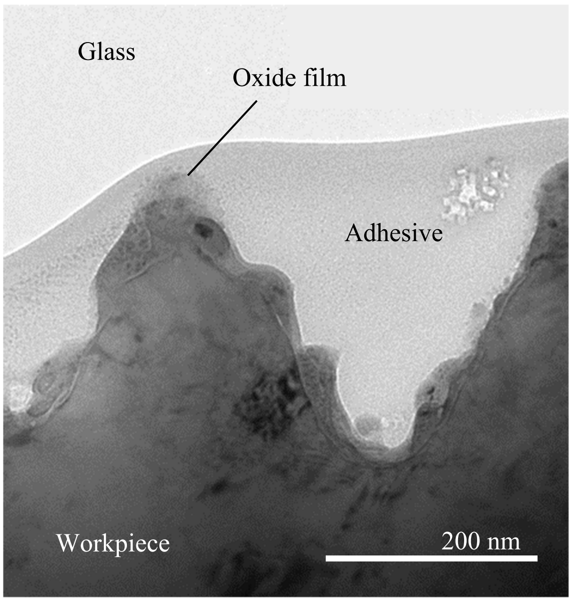
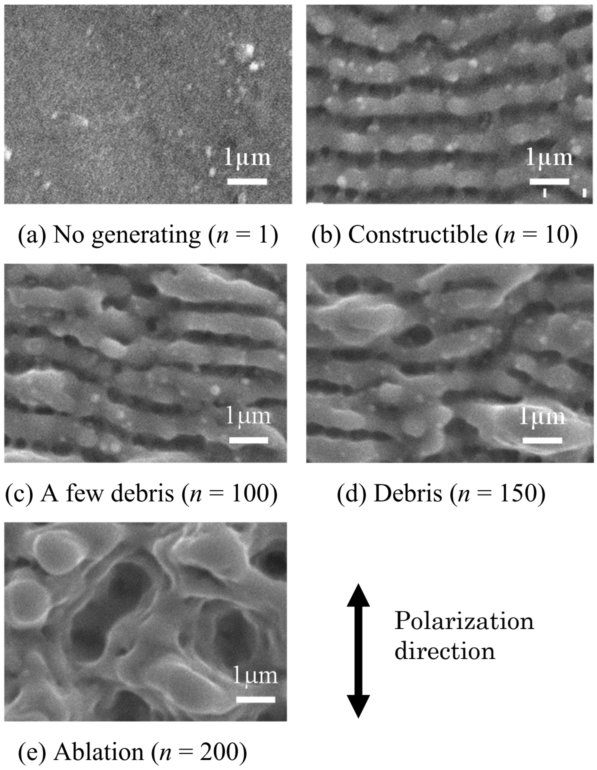
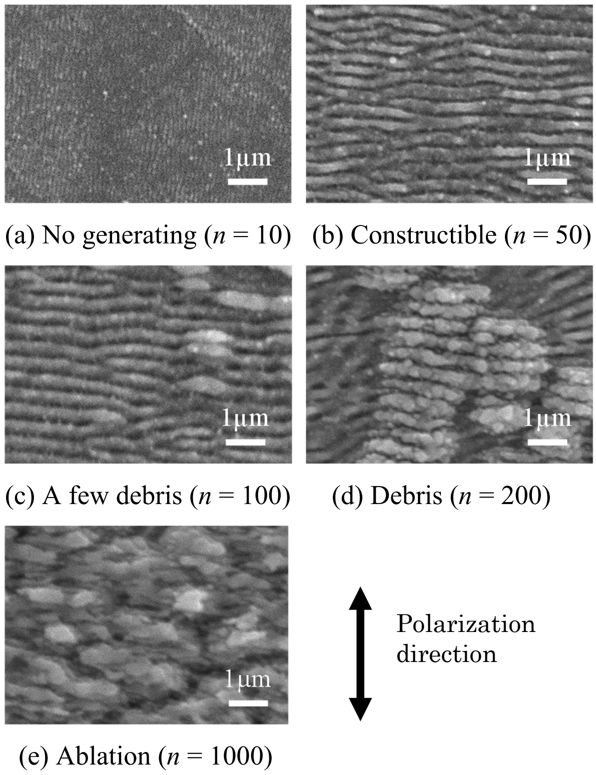


Fig. 6. TEM image of cross section view of the irradiation surface with $\lambda = 532$ nm, $E_d = 0.004$ J/cm^2, $n = 50$ at 293 K.



(a) No generating ($n = 1$) (b) Constructible ($n = 10$)
(c) A few debris ($n = 100$) (d) Debris ($n = 150$)
(e) Ablation ($n = 200$)

Fig. 7. SEM image of the irradiation surface with $\lambda = 1064$ nm, $E_d = 2.83$ J/cm^2 at 223 K.



(a) No generating ($n = 10$) (b) Constructible ($n = 50$)
(c) A few debris ($n = 100$) (d) Debris ($n = 200$)
(e) Ablation ($n = 1000$)

Fig. 8. SEM image of the irradiation surface with $\lambda = 532$ nm, $E_d = 0.044$ J/cm^2 at 223 K.

of SEM images of the irradiated surface with $\lambda = 532$ nm and $E_d = 0.044$ J/cm^2 at 223 K. With $n = 50$, fine periodic structures were seen perpendicular to the direction of polarization. However, debris was gradually deposited on them until the structures disappeared. The structures were constructed clearly and sharply perpendicular to the direction of polarization, in contrast to the structures created at 293 K, and the process was different from the process at 293 K. The reason is considered to be that thermal effects were suppressed and the thermal fluctuation and influence

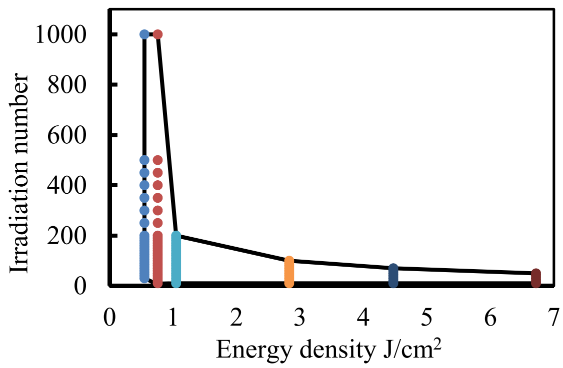
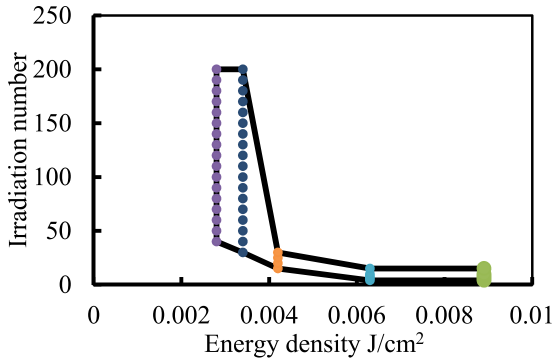
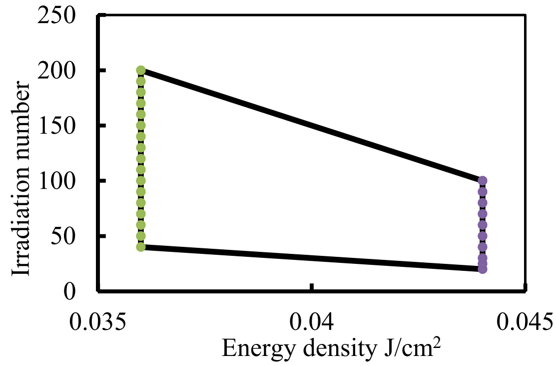


Fig. 9. EFR with λ= 1064 nm, 223 K.



(a) Material temperature 293 K



(b) Material temperature 223 K

Fig. 10. EFR with λ= 532 nm at 293 K and 223 K.

of the crystal grain boundary was inhibited by cooling the workpiece.

Figure 9 shows the EFR with $\lambda = 1064$ nm at 223 K. These results show that the range of E_d for structure fabrication is expanded immeasurably over that at 293 K, and the EFR is much larger than that at 293 K. **Fig. 10** illustrates the EFR with $\lambda = 532$ nm at 223 K and 293 K. This figure indicates that the upper limits of E_d and n in terms of EFR are obviously smaller than those when $\lambda = 1064$ nm. This difference is caused by the change in wavelength. It is estimated by the equation for the absorption coefficient a, the penetration depth d, and the critical density n_{cr} since it is assumed that the ablation begins when a laser beam penetrates the material and the free electron density is built up, reaching the critical density [9–12]. The absorption coefficient, the penetration depth, and the critical density are expressed as [13–14]:

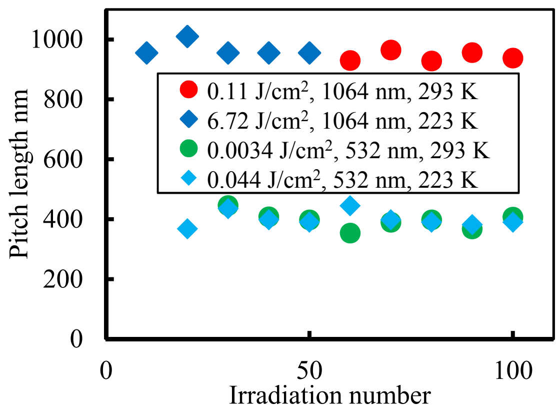


Fig. 11. Relationship between pitch length and n with λ= 532 nm and 1064 nm at 223 K and 293 K.

$$a = \frac{4\pi k}{\lambda} \quad \text{(3)}$$

$$d = \frac{1}{a} \quad \text{(4)}$$

$$n_{cr} = \frac{4\pi^2 c^2 m_e \varepsilon_0}{\lambda^2 e^2} \quad \text{(5)}$$

where k is the extinction coefficient, λ is the wavelength of the laser, c is the scalar speed of light in a vacuum, m_e is the mass of a stationary electron, ε_0 is the electrical permittivity, and e is the electron charge. Based on Eqs. (3), (4), and (5), the absorption coefficient and the critical density are inversely proportional to the laser wavelength and the square of the laser wavelength. On the other hand, the depth of penetration is inversely proportional to the absorption coefficient. That is, shortening the laser wavelength increases the light that is absorbed into the surface of the material, and this increases the chances for total ablation and causes the upper limit of EFR to decrease. **Fig. 10** also indicates that the lower limit of EFR obviously decreases with the change in the wavelength. Comparison between the results at 293 K and 223 K indicates that the width of EFR at 223 K has a larger range than at 293 K, and this indicates that the fabricated structure at 223 K has less heat influence than that at 293 K. The principle of fine structure fabrication has not been specifically clarified, but these results indicate that a certain amount of energy incident is required for fabrication, and it increases the bottom limitation of EFR as it relates to the decrease in material temperature and the increase in wavelength.

Figure 11 shows the relationship between the pitch length of the periodic structures with $\lambda = 1064$ nm and 532 nm at both 293 K and 223 K. The pitch length of nanostructures fabricated with the 1064 nm wavelength laser were about 900 nm, and fine periodic structures fabricated with the 532 nm wavelength laser had a pitch length of approximately 400 nm at both 293 K and 223 K. The structures were close to the laser wavelength. It is considered that the pitch length of periodic structures depends just on the laser wavelength regardless of changes in the energy density, the irradiation number, the material temperature, and the laser wavelength, which is expressed as [15]:

$$P = \frac{\lambda}{1 \pm \sin\theta} \quad . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \quad (6)$$

where P is the pitch length of fine periodic structures and θ is the laser incident angle. Eq. (6) is defined by the principle of interference of the incident light and scattered light, and it shows that the pitch length is equal to the laser wavelength if laser irradiation is perpendicular to the plane of the workpiece. However, it was reported that nanostructures have a pitch length that is a fraction of the laser wavelength [16–18]. That principle is not able to explain this phenomenon, so the principle of the surface plasmon is proposed. This means that collective vibrations of free electrons occur periodically on the surface of the material, caused by the electronic excitation of the laser irradiation, Coulomb explosion is periodically caused by excitation of surface plasmon and fine periodic structure is constructed. The pitch length of surface plasmon, which means the pitch length of periodic structures, is expressed as [19–21].

$$\frac{1}{3}\lambda \leq P \leq \lambda \quad . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \quad (7)$$

Recently, these two principles have mainly been advocated for structure fabrication with short-pulsed lasers, and this means the pitch length is close to the laser wavelength.

The results in this section verify that a short-pulsed laser with a pulse duration of 20 ps is able to fabricate fine nanostructures, and chilling the workpiece serves to enable sharp, fine structures under a wide range of process conditions and without heat effects. Additionally, it is confirmed that the pitch length depends on the laser wavelength, and EFR can be extended by changing the wavelength and the temperature of the material.

4. Conclusion

1. In this study, the fabrication and control of a fine periodic surface structure by means of short-pulsed laser were investigated. The conclusions of this study are as follows.

2. By changing the material temperature from 293 K to 223 K, thermal effects such as melting, which are a problem for short-pulsed laser, decreased, and EFR expanded. It is easy to fabricate structures by cooling the material since the CRT extends in low-temperature environments.

3. By changing the laser wavelength from 1064 nm to 532 nm, the energy density necessary for fabricating fine periodic structures decreased, and EFR became narrow since the light penetrates deeper into materials as the laser wavelength becomes shorter. The shorter the wavelength is, the deeper light penetrates into materials

4. The pitch length of structures was close to the laser wavelength and depended on it; the length is independent of the changing material temperature and laser wavelength.

5. It is easy to control the fabrication of fine, periodic surface structures by regulating the material temperature and the laser wavelength.

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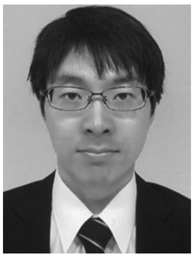

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