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Very low electron temperature in warm dense matter formed by focused picosecond soft x-ray laser pulses

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We investigated the optical emission from the ablating surfaces induced by the irradiations of soft x-ray laser (SXRL) pulses with the aim of estimation of the maximum electron temperature. No emission signal in the spectral range of 400–800 nm could be observed despite the formation of damage structures on the target surfaces. Hence, we estimated an upper limit for the electron temperature of 0.4–0.7 eV for the process duration of 100–1000 ps. Our results imply that the ablation and/or surface modification by the SXRL is not accompanied by plasma formation but is induced by thermo-mechanical pressure, which is so called a spallative ablation. This spallative ablation process occurs in the low electron temperature region of a non-equilibrium state of warm dense matter. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4901943>]

I. INTRODUCTION

The interactions of short pulse lasers with matter are interesting subjects not only in applications such as surface fabrication but also in physical phenomena for study.¹ Lasers having short pulse widths have abilities to make the ablation and/or modification structures on material surfaces accompanying the creation of high temperature, high pressure, and excited states of electrons.² Until now, we have reported on the ablation and modification structures on material surfaces induced by the irradiation of soft x-ray laser (SXRL) pulses, which is a plasma based coherent soft x-ray source. On Al surface, unique conical structures with nanometer scale diameters and heights were created, when the fluence of the SXRL pulse was lower than the ablation threshold.³ On Au and Cu surfaces, ripple-like structures were formed by a single SXRL pulse irradiation;⁴ however, on Si surface, a damage structure could be recognized after the accumulation of SXRL pulses.⁴

The ablation and modification structures induced by the SXRL pulses were compared to those from the other laser pulses. The fluence for the ablation threshold of LiF by the SXRL pulse was lower than those of other lasers with longer pulse widths or longer wavelengths.⁵ The modification structures on Au induced by the accumulation of multiple SXRL pulses were similar to those obtained by multiple pulse irradiations of a Ti:sapphire laser,⁶ but it is revealed that the total fluence of the SXRL pulses was essentially lower than

those of the Ti:sapphire laser pulses.⁴ Because of the long attenuation length of the SXRL for Si, deep holes were formed on Si, which were different from other substances,⁴ and those structures were also different from those created by Ti:sapphire laser pulses.⁷ The differences between the SXRL and optical lasers were the attenuation length, absorption coefficients (these were changing with the laser wavelength), fluence, and repetition rate. The low ablation or modification threshold of materials for the SXRL pulse has the possibility for efficient surface machining. It is widely thought that the penetration of short pulse laser radiation into matter during an ablation process accompanies heating of the electrons in the matter. During the heating, the density is of the order of solid state density, so such a process occurs in a non-equilibrium state with relatively high electron temperature, so called warm dense matter (WDM). However, the details of the surface ablation/modification process have yet to be clearly resolved.

We have also studied the surface ablation/modification processes theoretically. Spallation phenomena are predicted for the interaction between SXRL pulses and material surfaces.^{3,8–11} The spallation process describing the ablation mechanism is as follows; the nucleation of interatomic size foams occurs under the SXRL pulse irradiation, these foams swell, a free surface separates from the substance, and finally a spallative layer runs away. The driving force for the spallation is an increasing pressure appearing inside the heating layer. Under a relatively low fluence region near the ablation threshold, it is considered that the first change of the surface structure is due to a splash of a molten layer, because the

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dependence of the molten depth on the fluence agrees with experimental results.^{11,12} The theoretical approach for an interaction model of a SXRL pulse with material surfaces plays a crucial role in the explanation of the nanometer scale surface modification in experiments. However, there is still large gap between the experimental results and the theoretical predictions. One of the difficulties in the theoretical description is the characteristic time of equilibrium states between electrons and ions, which is comparable to the ablation time including heat transfer, phase transitions, and appearance of shock waves. Therefore, experimental information, such as electron temperature, is very important to describe the fundamental physical process of the interaction with condensed matter.

To study the ablation/modification mechanism experimentally, especially in the soft x-ray range, spatial, temporal, and spectral analysis experiments are necessary. Unfortunately, we can scarcely find articles dealing with ablation processes, such as time-resolved observations.^{13–15} Now, we have initiated an experimental study pursuing investigation of the surface ablation/modification process by a SXRL with a short pulse width. In this article, we report on the observational results of the optical emission in the visible spectral range from the irradiated target surfaces and also on the discussion of an upper limit of the electron temperature during the ablation process. We will suggest a plasma-less ablation process in the interaction between the SXRL pulse and matter.

II. EXPERIMENTAL SETUP

The SXRL for the irradiation experiment is a full coherent soft x-ray source, which has been developed at the Japan Atomic Energy Agency.¹⁶ The experimental setup was composed of three parts: the SXRL irradiation component (source and optics), the target, and the observational component (lens and optical camera). A schematic diagram of the experimental setup is shown in Fig. 1.

The parameters of the SXRL were a wavelength of 13.9 nm with a bandwidth of $\sim 10^{-4}$, and 7 ps pulse width. The SXRL pulse generated from Ag plasma mediums using an oscillator-amplifier configuration with double Ag targets

was focused on the target surface using a Mo/Si multilayer coated spherical mirror with a radius of curvature of 1000 mm, which was placed at a distance of approximately 2600 mm from the SXRL output. A 100 μm thick glass plate made from quartz (SiO_2) and a 0.1 μm or 0.2 μm thick Zr filter were placed in front of the spherical mirror. The glass plate is transparent to optical light (visible spectral range) and opaque for the SXRL beam. The Zr filter reduces almost all of the scattered optical radiation from the laser produced Ag plasma. The glass plate and Zr filter could be removed from the SXRL penetration axis independently. The transmittance of 0.1 μm or 0.2 μm thick Zr filter for 13.9 nm was approximately 70% or 48%, respectively. The Mo/Si multilayer coating was optimized for soft x-rays of 13.9 nm at normal incidence, and the reflectivity of this spherical mirror was approximately 50%. This mirror could also reflect the visible light, in the spectral range of 400–800 nm, with an efficiency of 30%–40% because of a Si top layer coating. When we adopted a 0.2 μm thick Zr filter, the typical energy and fluences on the target surfaces, which were measured as an averaged value in the modification zone, were estimated to be around 48 nJ/pulse and 10–30 mJ/cm^2 , respectively.^{3,4} In the same geometry, if the glass plate was only adopted without Zr filters, the optical light with an energy of approximately 90 nJ/shot, which was two times larger than the energy of the SXRL pulse, could be focused on the target surface.

LiF, Al, and Cu plates were used as targets. We selected such targets for the following reasons: LiF is a typical dielectric with well-known characteristics, the two others are typical metals. We chose Al as a metal with a simple zone structure and Cu as a metal with a complicated electron spectra having d-electrons. We expected that the surface ablation/modification mechanism for such different solid materials would be similar, because the x-rays are absorbed mainly by internal shells in the material. We used rather thick targets: the thickness of the LiF plate was 2 mm, and those of the Al and Cu plates were 1 mm. All target plates were mounted on a movable sample holder in order to ensure equal focusing conditions. The fresh surfaces were placed at the best focal position of the SXRL beam. We should stress another important

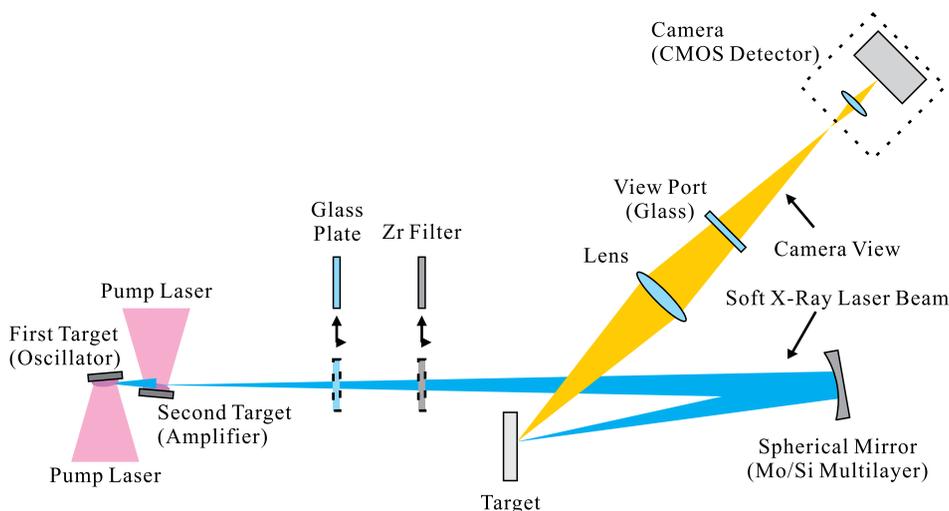


FIG. 1. Schematic diagram of the experimental setup.



FIG. 2. Acquired optical images of the SXRL pulse irradiating the LiF surface by the CMOS detector without a filter, with a $0.2\ \mu\text{m}$ thick Zr filter and with a $100\ \mu\text{m}$ thick glass plate.

advantage of the use of LiF crystal as a target. Because of the small surface modification (permanent surface alteration or damage by melting) threshold of the LiF surface by the SXRL pulse irradiation $\sim 10\ \text{mJ}/\text{cm}^2$,⁵ it is easy to make a damage structure with a single shot of the SXRL pulse. In addition, stable color centers (CCs) can be formed by the SXRL pulse irradiation on a LiF crystal.¹⁷ These CCs enable us to find the irradiated positions easily. By observation of the visible photoluminescence patterns from the CCs after the irradiation process, we can confirm the existence and shapes of the focus patterns with a sub-micrometer spatial resolution. For the SXRL beam, the CC formation threshold is estimated to be around $0.1\ \text{mJ}/\text{cm}^2$.^{12,17}

Emission light in the visible spectral range from a target surface induced by the SXRL irradiation or scattered optical light was observed by an optical camera. The optical image of the emitted light was transferred from vacuum to air by an optical lens ($f = 100\ \text{mm}$) and a glass viewport. The lens and viewport were made from quartz. Then the emission image was captured with an optical camera. A complementary metal oxide semiconductor (CMOS) device was used as an image sensor of the camera. The size of the CMOS sensor was $23.6\ \text{mm} \times 15.6\ \text{mm}$, and the pixel size of the sensor was $4.8\ \mu\text{m} \times 4.8\ \mu\text{m}$. This optical system observed radiation in the spectral region from $400\ \text{nm}$ to $800\ \text{nm}$ (in the photon energy region of $1.54\text{--}3.08\ \text{eV}$). The detectable energy threshold was estimated to be approximately $0.016\ \text{nJ}/\text{pixel}$ ($1.6 \times 10^{-11}\ \text{J}/\text{pixel}$).

The SXRL irradiation was performed in a vacuum, and the camera with the CMOS detector was in air.

III. RESULTS AND DISCUSSION

Figure 2 shows the acquired optical images from a LiF surface irradiated by the SXRL pulse with the optical camera. When no filter was applied (shown as no filter), a bright lighting signal on the target surface was observed. In this case, not only the SXRL pulse but also the optical radiation from the Ag plasma reached the LiF surfaces. However, when a $0.2\ \mu\text{m}$ thick Zr filter was inserted, we could not recognize any signals even by adjusting the brightness and/or contrast of these images. In this case, only the SXRL pulse reached the surfaces. If the Zr filter was removed and the glass plate was inserted in the SXRL beam, the SXRL pulse was blocked and the LiF surface was irradiated by optical radiation from the Ag plasma. In this case, a bright lighting signal could be observed.

Figure 3(a) shows the beam patterns of the SXRL pulses recorded on the LiF target surface at the best focus position obtained with a fluorescent microscope. Every SXRL pulse irradiated position, except in the case of the insertion of a glass window, shows fluorescence signals due to the formation of CCs. When the glass window was inserted in the SXRL beam axis, no fluorescence signal was observed, even though an accumulation of three pulses was applied. The focused optical light (especially the visible portion) from the Ag plasma will not be able to produce CCs, since the band gap of LiF is about $14\ \text{eV}$, which is larger than the photon energy of the visible light range. This is because the glass window could not transport optical emission having an energy range larger than $8\ \text{eV}$ (wavelength range shorter than $160\ \text{nm}$).

Figure 3(b) shows the enlarged fluorescence images of the SXRL focal spots obtained with a single pulse shot, in the case of no filter, $0.1\ \mu\text{m}$ or $0.2\ \mu\text{m}$ thick Zr filter. In the fluorescence images, we could clearly confirm damage structures on the LiF surface, as shown in Fig. 3(c). The sizes of the damage structures are estimated to be $5\text{--}10\ \mu\text{m}$ (W) \times $12\text{--}15\ \mu\text{m}$ (H). However, any modified structure on the LiF surface was not obtained, when the $100\ \mu\text{m}$ thick glass plate was inserted solitarily. The experimental findings as shown in Figs. 3(a)–3(c) demonstrated that pure optical light irradiation with comparable fluences to the SXRL pulse did not influence the LiF surface.

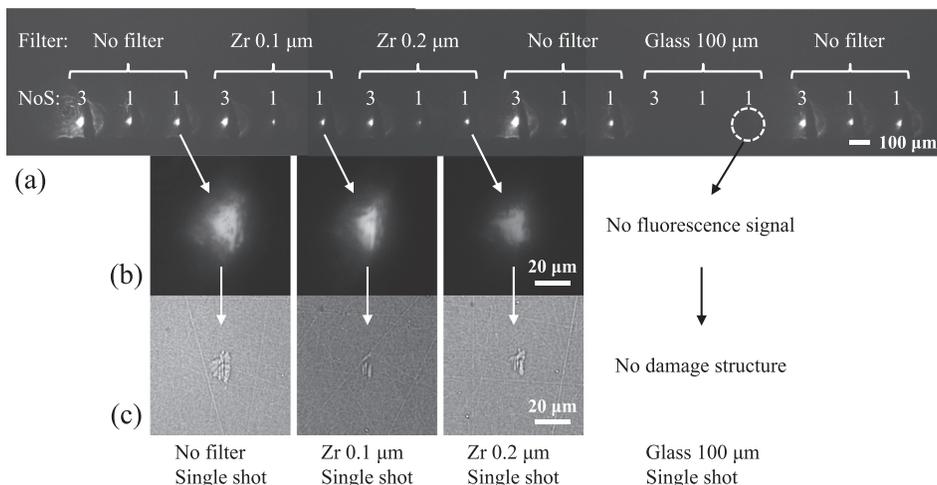


FIG. 3. (a) Fluorescence microscope images on the LiF target surface. NoS means the number of accumulated shots. (b) Enlarged fluorescence microscope images and (c) differential interference microscope images for each single SXRL pulse irradiated position. Scale lengths are indicated in the images.

From the results as mentioned above, we could conclude that the observed bright lighting images shown in Fig. 2 were focused light images radiated from the Ag plasma. This light emitted from the Ag plasma was reflected by the Mo/Si multilayer coated spherical mirror and that was focused onto the target surface. The focused light was scattered from the surface, and then, the optical camera acquired an image of this light. The wavelength range of this light was estimated to be 400–650 nm by an additional spectral measurement.

By using the same setup, we also irradiated Al and Cu plate targets. The surface modification thresholds of Al and Cu by the SXRL pulse are around 20 mJ/cm².^{3,4} Figure 4 shows the acquired optical images from the Al and Cu surfaces irradiated by the SXRL pulse by the optical camera. When the Zr filter was inserted, we could not observe any signals, despite the fact that clear damage on the target surfaces occurred (not shown). The results obtained with the dielectric crystal and the metal plate targets were the same, even though material properties, such as electric resistivity, melting point, x-ray attenuation length, and so on, were different. It is considered that the ablation mechanism for dielectric and metal targets induced by the SXRL pulses can be described using the same model.⁹ Because x-rays are absorbed mainly by internal shells in the materials, it is considered that the x-ray absorptions for dielectrics, semiconductors, and metals are qualitatively similar. In addition, the main condition for surface ablation or modification process mainly depends on the ratio between the laser pulse width and acoustic response time, which is concerned with the heated layer thickness and sound velocity. Hence, other parameters, such as target material, are less significant.⁹

The experimental results mentioned above mean that the photon intensity reaching the CMOS pixels, i.e., integrated energy of visible light during the exposure time of the camera, emitted from the ablation plasma produced by only the SXRL pulse did not exceed the detectable energy threshold of the CMOS detector. The production of WDM having a low electron temperature is a possible plasma-less ablation process in materials whether they are dielectric or metals. We could conclude that the plasma-less ablation process

should be an important process to consider as a SXRL interaction mechanism with all matter.

The penetration of the SXRL pulse into the target in an ablation or a modification process is accompanied by heating of the electrons, so such a process occurs in an extremely non-equilibrium state in the system. Calculations have predicted an electron temperature of 0.1–10 eV, which depends on the laser fluence.^{8–12} To consider the radiation from a heated or molten layer, we suppose a black-body radiator as an object. Suppose that the temporal behavior of the target electron temperature, T_e , can be described by a Gaussian

$$T_e = T_m \exp(-t^2/t_{em}^2), \quad (1)$$

where T_m is the maximum electron temperature and t_{em} characterizes the emission duration. It should be noted that dependence (1) is an approximation. The real dependence $T_e(t)$ must be asymmetrical, because the increasing rate of the temperature is determined by the temporal profile of the SXRL pulse, while the decreasing rate of the temperature is caused by another physical process, namely by electron-ion relaxation. However, we believe that the Gaussian function in Eq. (1) can be used to approximate the dependence of target temperature in time.

Taking into account the imaging properties of the camera system described above, i.e., the observable spectral region, we can calculate the total energy of the optical photons reaching a camera cell as follows:

$$E_{cell} = \int_{-\infty}^{+\infty} 2 \cdot 10^4 \left[\frac{T_m \exp(-t^2/t_{em}^2)}{13.6} \right]^4 dt \times \int_{1.54 \exp(t^2/t_{em}^2)/T_m}^{3.08 \exp(t^2/t_{em}^2)/T_m} \left[\frac{x^3 dx}{\exp(x) - 1} \right]. \quad (2)$$

In (2), E_{cell} is in J and T_m is in eV, and E_{cell} depends on two parameters. Figure 5 shows the dependencies of E_{cell} on T_m for different values of t_{em} . In Fig. 5, the experimentally determined detectable energy threshold is also shown. From the consideration of the calculated curves, if T_m is larger than 0.7 eV and t_{em} is longer than 100 ps, we should observe an emission signal from the irradiation surface by the system. When E_{cell} is smaller than the detectable threshold value, no image should be observed. In this case, we can determine an upper limit of the target temperature. The model calculations show that the spallation process goes on for more than 100 ps.^{3,11} For $t_{em} \sim 100$ –1000 ps, for example, the temperature is estimated to be $T_m < 0.4$ –0.7 eV.

Here, we give a brief estimation of the temporal calculation for the electron temperature. We have mentioned above that the real dependence $T_e(t)$ will be asymmetrical. This means that the use of a symmetrical Gaussian function, generally speaking, can lead to errors in the temperature measurements. It is possible to roughly estimate this effect by using a strongly asymmetrical function for the time dependence of temperature. For this estimation, if we use the following function with the same emission width t_{em} as Eq. (1):

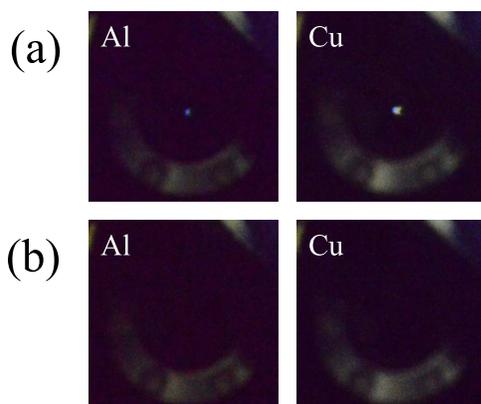


FIG. 4. Acquired images of the SXRL pulse irradiating metal target surfaces by the CMOS detector (a) without a filter and (b) with a 0.2 μm thick Zr filter.

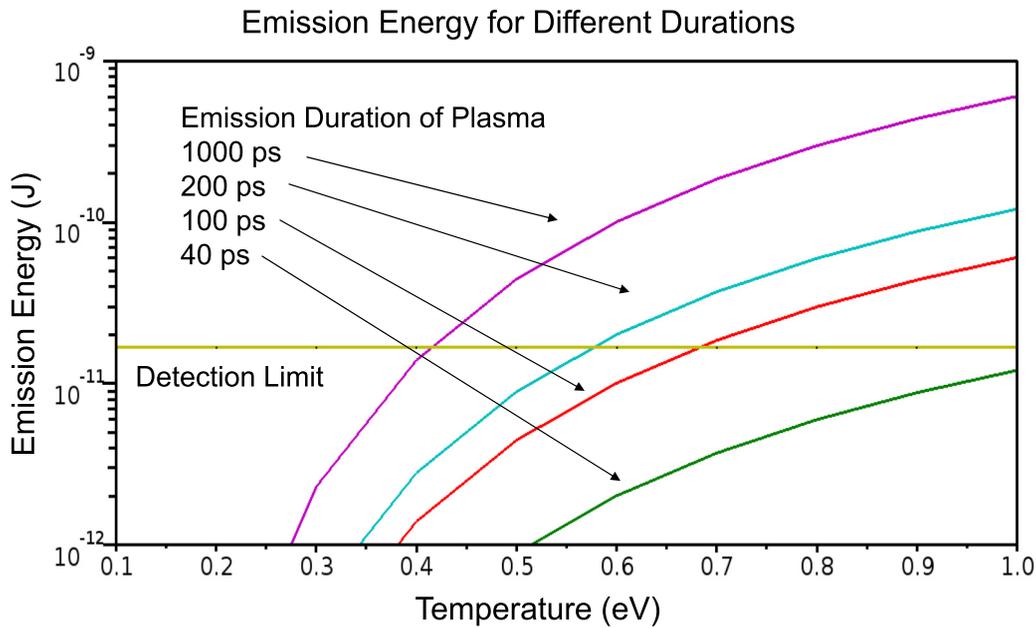


FIG. 5. The dependencies of the emission energy E_{cell} on the temperature T_m for different values of duration t_{em} . The sensitivity of CMOS detector is indicated by the straight line.

$$\begin{cases} T_e = 0 (t < 0) \\ T_e = T_m \exp(-t^2/4t_{em}^2) (t \geq 0), \end{cases} \quad (3)$$

then it is clearly seen that using Eq. (3) gives the almost same result for E_{cell} as the use of Eq. (1). We consider that the main inaccuracy of the temperature measurements is caused by the inaccuracy of t_{em} . However, even in the changing of t_{em} from 100 ps to 1000 ps (by 10 times) changes T_m only from 0.7 eV to 0.4 eV, i.e., less than a factor of two.

Our experimental and calculated results are in good accordance with the theoretical predictions. The electron temperature of around 1000 K (~ 0.1 eV) was calculated for a LiF target irradiated by the SXRL pulse with a fluence of 10 mJ/cm².⁹ If the fluence of 65 mJ/cm² is supposed for the SXRL pulse, then the total pressure reaches around 15 GPa, which is generated by the electron temperature of around 1 eV.¹¹ Under a low fluence region of 10–30 mJ/cm² for the SXRL pulse, an electron temperature of less than 1 eV is considered reasonable, which is, however, high enough for nanometer scale surface modification on the target surface.

IV. CONCLUSION

In this study, we demonstrated that the focused picosecond soft x-ray laser pulses could induce ablation of a dielectric and metals at very low electron temperature. The any light emission signal in visible spectral range from LiF, Al, and Cu surfaces accompanying the surface ablation/modification caused by only the SXRL pulse irradiation was not observed. These results allowed us to estimate the maximum electron temperature of the ablating matter to be 0.4–0.7 eV for the duration of 100–1000 ps, which is a quite low electron temperature region of WDM. We can conclude that our experimental study confirms the theoretical model of an atomistic simulation, which predicts the splash of molten

layers occurring under a low electron temperature of less than 1 eV creating nanometer scale surface modifications due to spallative ablation. It is important to consider the plasma-less ablation and/or surface modification process as a possible SXRL interaction mechanism with matter.

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