# Nano-meter scale modifications on material surfaces induced by soft x-ray laser pulse irradiations

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## ABSTRACT

To study the interactions between a soft x-ray laser (SXRL) and various materials, we irradiated Al, Au, Cu, and Si with the SXRL beam pulses having a wavelength of 13.9 nm and duration of 7 ps. Following the irradiation, the induced structures were observed using a scanning electron microscope and an atomic force microscope. With single pulse irradiation, conical structures were observed on the Al surface, and ripple-like structures were formed on the Au and Cu surfaces. The conical structures were destroyed under multiple SXRL pulse irradiation. On the other hand, the developments of modified structures were observed after multiple pulse irradiations on the Au and Cu surfaces. On the Si surface, deep holes, that seemed to be molten structures induced by the accumulation of multiple pulse irradiations, were found. Therefore, it is concluded that the SXRL pulse irradiations of various material surfaces cause different types of surface modifications, and the changes in the surface behaviors are attributed to the differences in the elemental properties of each materials, such as the melting point and the attenuation length of x-rays.

**Keywords:** soft x-ray laser, surface modification, ablation threshold, modification threshold, surface machining

## 1. INTRODUCTION

Laser pulses with short durations of pico-seconds or femto-seconds have the ability to make high temperature, high pressure, excite states of electrons in materials, and emit rays, as a result of various interactions with materials. Soft x-ray lasers (SXRLs) are attractive x-ray sources for scientific studies, because of their short wavelength (photon energy of a soft x-ray is higher than that of visible light), short pulse duration, and high spatial coherence.[1] An SXRL has been developed at the Japan Atomic Energy Agency (JAEA). The SXRL pulse has a short wavelength of 13.9 nm, a duration of 7 ps, and a fully spatial coherence,[2–4] a significant amount of research on applications using the unique features of the SXRL pulse have been done.[5–11] When we investigated the characteristics of the focal patterns of SXRL pulses by means of color center formation in a lithium fluoride (LiF) crystal,[12] we found ablation structures,[13] and we also studied the ablation property theoretically.[14,15] The ablation property of a LiF crystal induced by the SXRL pulse was very interesting, because the ablation threshold was much smaller than those obtained with other lasers having longer

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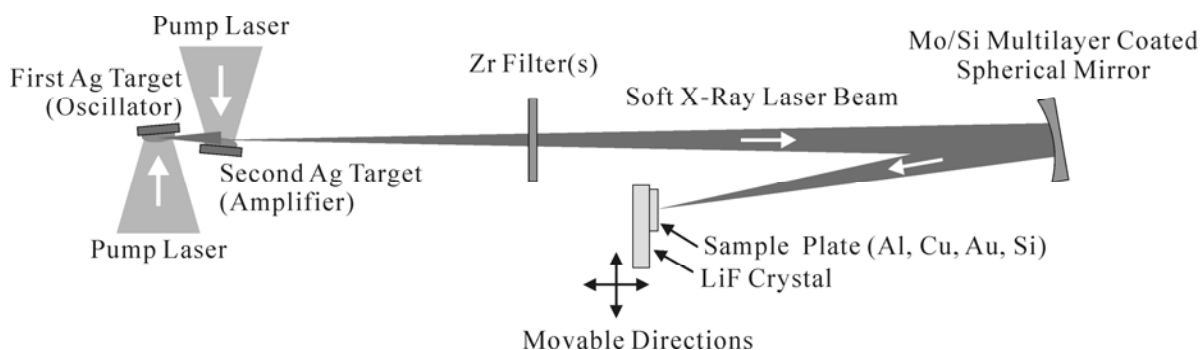
**Figure 1.** Schematic diagram of experimental setup for soft x-ray laser pulse irradiation.
Soft x-ray laser pulse generated from Ag plasma mediums is focused on sample surface by spherical mirror.

wavelengths and/or longer pulse durations. When we irradiated an aluminum (Al) surface with a focused SXRL pulse, we found that conical structures having diameters of approximately 100 nm and average depths of approximately 40 nm were formed on the Al surface.[16] These peculiar nano-meter size structures were the result of the unique interaction between the SXRL pulse and the Al surface. Recently, we studied the ablation properties of gold (Au) both experimentally and theoretically.[17,18] The modifications on the Au surface induced by an SXRL pulse with a fluence of $21 \pm 5$ mJ/cm$^2$, and the deployment of SXRL pulses with fluences of 30–60 mJ/cm$^2$ allowed nano-meter size structuring of the Au surface by removing layers 10–50 nm thick. The energy threshold of the surface modification at 21 mJ/cm$^2$ was almost the same as that described by an extreme ultraviolet free-electron laser (FEL) pulse,[19] and slightly lower than the ablation threshold of 67 mJ/cm$^2$ obtained by an optical Ti:sapphire laser pulse.[20] Hence, we are interested in the interactions between the SXRL and various material surfaces, especially the surface modifications induced by the SXRL pulse irradiation with relatively low fluence, because an understanding of surface modifications is fundamental and also because surface modifications influence technological applications. The low ablation threshold and peculiar surface structures have possibilities for efficient and unique surface machining.

In this paper, we experimentally show the surface modifications induced by SXRL pulses. The results of the surface modifications resulting from the interactions between the SXRL pulses and materials are reported. The samples were Al, copper (Cu), Au, and silicon (Si). We found that the induced structures on various surfaces were considerably different from each other. The possible applications of these modified structures are also discussed.

## 2. EXPERIMENT

The SXRL irradiation experiment was carried out at the SXRL facility at JAEA. Al (1 mm thick), Cu (1 mm thick), Au (0.5 mm thick), and Si (1 mm thick) plates were used as the target samples. Al, Cu and Au are low-Z, middle-Z and high-Z conductors, respectively. Si is a semiconductor. A schematic diagram of the experimental setup is shown in Fig. 1. The SXRL pulse was generated from silver (Ag) plasma mediums, which were used in an oscillator-amplifier configuration with Ag double targets. The characteristics of the generated SXRL pulse from the Ag double targets were wavelength of 13.9 nm (photon energy of 89 eV), bandwidth of narrower than 10$^{-4}$, and duration of 7 ps. The output energy of the SXRL pulse varied with each shot, and the pulse-to-pulse fluctuation was evaluated to be 70–80% from 15 consecutive laser shots. However, the fluctuation of the nearest five shots was relatively small, i.e., less than 22%. The SXRL pulse was focused on a sample surface by using a Mo/Si multilayer coated spherical mirror having a radius of curvature of 1000 mm. The Mo/Si multilayer coating was optimized for soft x-rays with a wavelength of 13.9 nm at an incidence angle of 2°, and it was placed approximately 2640 mm from the SXRL output. Then 0.2 μm thick Zr filters were placed in front of the spherical mirror in order to reduce the scattered optical radiations from the Ag plasmas. All the sample plates were fixed on the surfaces of LiF crystals. Each LiF crystal with its target sample was mounted on a
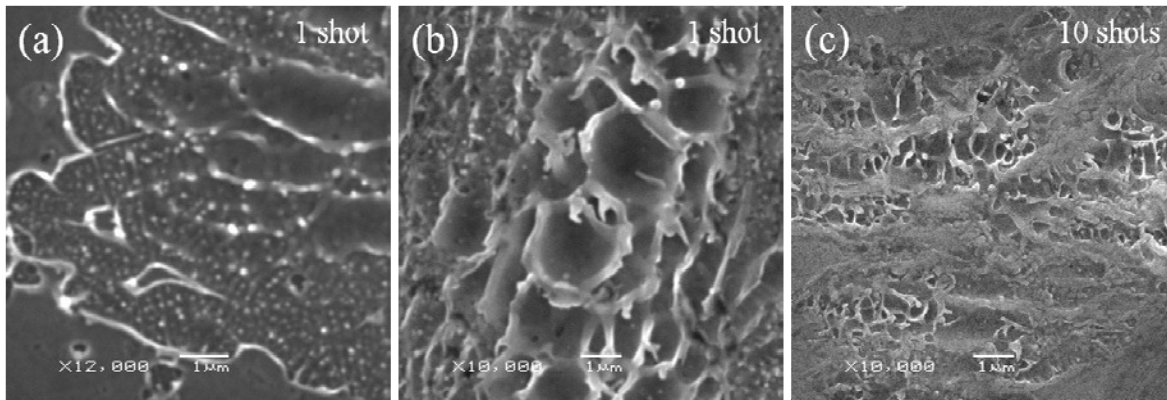
**Figure 2.** SEM images of structures on Al surfaces induced by single shot irradiation of an SXRL beam with (a) two Zr filters (0.2 μm + 0.2 μm) and (b) a single Zr filter, and (c) 10 shot irradiation with a single Zr filter.

sample stage that had two movable directions. The sample was moved after the prescribed number of shots along the propagation direction of the incident SXRL beam around the best focal position, and it was also moved perpendicular to the propagation direction in order to be irradiated on a fresh surface. All target surfaces were well synchronized to ensure identical focusing conditions. The distribution of the formed features on each sample surface represents the distribution of the gain structures in the Ag plasmas, because the spherical mirror reconstructs a reduction image of the SXRL source. The complete irradiation process was carried out in a vacuum chamber.

The total energy of the SXRL pulse reaching the sample surface was approximately 48 nJ, because the transmittance of the Zr filter and the reflectivity of the Mo/Si mirror at a wavelength of 13.9 nm were approximately 48% and 50%, respectively, and the average output energy of the SXRL pulse was estimated to be approximately 200 nJ.[21] At this wavelength with normal incident irradiation, almost the entire incident energy of x-ray photons was absorbed by the samples. The SXRL pulse intensity profiles and stabilities were confirmed using a LiF detector. The modulation of the intensity for the two shots did not exceed approximately 10%. The positional stability of the SXRL pulses on the target surfaces was observed by the reproducibility of the distribution of laser spot images.[22] After the single and multiple SXRL pulse irradiations, it was confirmed that the SXRL pulse irradiations reproduced almost the same images on LiF. Using the LiF detector technique to estimate the quality of the SXRL pulse and its focusing properties, we found that the SXRL energy concentration in the best focusing areas reached approximately 60%.[18] The residual parts of the SXRL pulse would result from the intensity of the background, which might arise from the oscillator plasma of the first Ag target. Approximately 40% of the focusing SXRL energy was concentrated in the center of the best focusing spots,[16] as a result, the SXRL energy at the center of the best focal spot was expected to be 12 nJ.

After the irradiation experiments, the sample surfaces were observed with an optical microscope to confirm whether the irradiated positions were modified or not. To analyze the details of the induced structures, which could not be seen with an optical microscope, the structures were also observed with a scanning electron microscope (SEM) and an atomic force microscope (AFM). Before the SEM observation of Si, the Si surface was coated with a thin Au layer in order to drain the electrons.

## 3. RESULTS AND DISCUSSION

The SEM images of the structures on the Al surfaces induced by SXRL pulse irradiations near the focal positions are shown in Figs. 2(a)–2(c). When a single Zr filter was used, fluences of 24 mJ/cm$^2$ and 12 mJ/cm$^2$ were estimated at the
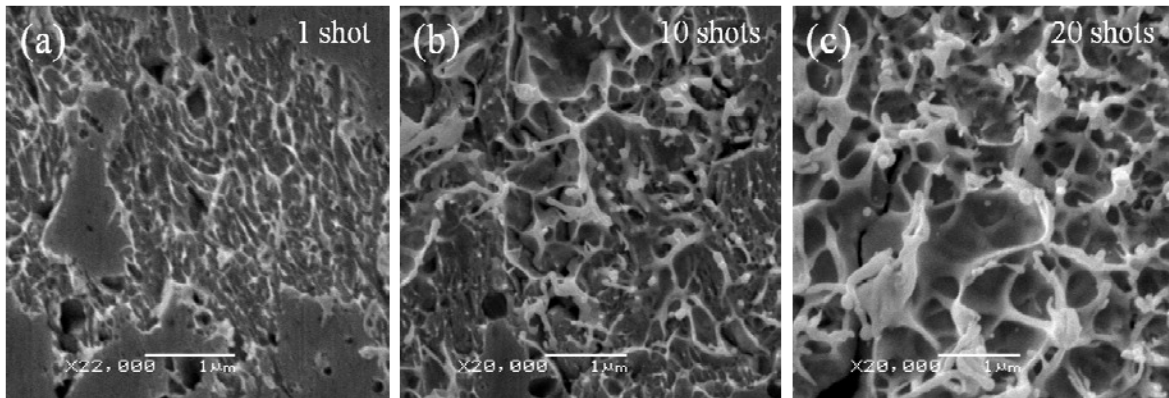
**Figure 3.** SEM images of structures on Au surface induced by (a) single shot, (b) 10 shot, and (c) 20 shot irradiation of SXRL beam with a single Zr filter.

center of the best focal spots (ablated areas) and the modified parts around the ablated areas, respectively. The total fluence in Fig. 2(a) was reduced to 50% of that in Fig. 2(b), because a Zr filter with a thickness of 0.2 μm has a transmittance of 50%. In Fig. 2(b), several ablation holes are confirmed at the center of the modified structure. It can also be seen that conical structures are formed in the modified areas surrounding the ablation holes. The conical structures are formed in the lower fluence region. The formation of the conical structures on the Al surface induced by SXRL pulse irradiation is a significant phenomenon. The damaged area (including the ablated and modified parts) shown in Fig. 2(a) is smaller than that shown in Fig. 2(b). On the other hand, the fraction of the conical structure area could be increased. In addition, the diameters of the formed conical structures seem to be uniform, meaning that we should be able to control the size or dispersion of the conical structures by controlling the SXRL fluence. From the estimation of fluences in the ablation and modified parts that appear on the Al surfaces, it can be concluded that the ablation and modification thresholds for Al were approximately 24 mJ/cm$^2$ and 12 mJ/cm$^2$, respectively. These estimated values agreed well with those reported in previous work, which were in the range of 7.3–27 mJ/cm$^2$.[16]

The structures induced by SXRL single pulse irradiation on the Au and Cu surfaces were quite different from that on the Al surface. Conical structures did not appear on either surface; instead, ripple-like shallow structures can be seen on the Au shown in Fig. 3(a) and Cu shown in Fig. 4(a). Ablated holes were not confirmed on the Au surface. However, they are confirmed on the Cu surface, and the ripple-like structures were formed around the holes. The modified area on the Au surface was essentially smaller than those on the Al and Cu surfaces. The ablated parts in the modified structures on the Cu surface were similar to that on the Al surface. In addition, these ablated areas on the Al and Cu surfaces were similar to that on the Au surface. Such differences in the modified sizes and the absence of ablated holes show that Au has a higher threshold for ablation and surface modification compared with Al and Cu. Fluences of 20 mJ/cm$^2$ and 30 mJ/cm$^2$ were estimated at the center of the best focal spots for the Au and Cu surfaces, respectively, and 14 mJ/cm$^2$ was expected at the modified parts around the ablated areas on the Cu surface. Hence, the modification thresholds for Au and Cu would be approximately 20 mJ/cm$^2$ [18] and 14 mJ/cm$^2$, respectively. The ablation threshold for Cu was estimated to be approximately 30 mJ/cm$^2$.

In order to study the accumulation effects of laser fluence, we observed the structures formed by multiple SXRL pulse irradiations. The structure induced under multiple SXRL pulse irradiations on the Al surface is shown in Fig. 2(c). After irradiation of 10 SXRL pulses, the conical structures were destroyed and no growth can be seen. Figures 3(b) and 3(c) show the structures formed on the Au surface by 10 and 20 SXRL pulse irradiations, respectively. Also, Figures 4(b) and 4(c) show the structures formed on the Cu surface after irradiations of 5 and 10 pulses, respectively. For Au, the structures induced by the single and multiple SXRL pulse irradiations were very similar to those formed by Ti:sapphire laser pulses with a fluence of 1.1 J/cm$^2$.[20] After single pulse irradiation by the Ti:sapphire laser, a modified structure denoted as "nanoscale roughness" was produced. After irradiation of two pulses, "nanobranches" were formed, and
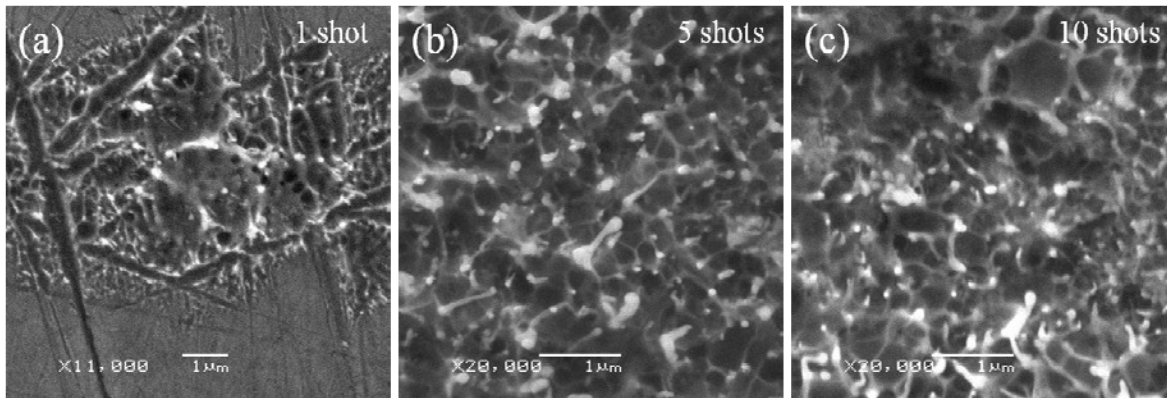
**Figure 4.** SEM images of structures on Cu surface induced by (a) single shot, (b) 5 shot, and (c) 10 shot irradiation of SXRL beam with a single Zr filter.

"spherical nanoparticles" appeared after five shots. The induced structures shown in Figs. 3(a)–3(c) were similar to the "nanoscale roughness", "nanobranches", and "spherical nanoparticles" structures, respectively. Comparing our results to those of Vorobyev and Guo, it can be said that the deposited total energy of the SXRL is essentially lower than that of a Ti:sapphire laser, but the induced structures on Au surfaces are the same. This fact reveals that the threshold value for the formation of nano-meter size structures obtained by an SXRL pulse is smaller than those obtained by other lasers having longer wavelengths. In other words, a laser beam that has a low damage threshold for materials could be use for efficient surface machining. For Cu, the ripple-like structures around the ablated holes formed by single pulse irradiation are also similar to the "nanoscale roughness" structure. The structural differences shown in Figs. 4(b) and 4(c) are small, and these structural patterns look like "nanobranches". If the Cu surface could be exposed to SXRL pulses of 20 shots or more, structures similar to the "spherical nanoparticles" might appear. But the mechanism of surface modification induced by the SXRL pulses, such as the ablation threshold, seems to be different; therefore additional quantitative investigations would be required for deeper understanding.

The depths of the ablated holes appearing on the Al surface shown in Fig. 2(b) were measured to be approximately 400 nm by AFM measurement. Many profiles of ablated holes on the Al surfaces created by single SXRL pulse irradiation were observed,[16] and measured to be in the range 100–400 nm. On the Cu surface, ablated holes with depths of approximately 140 nm were created by a single pulse with a fluence of 30 mJ/cm$^2$. Typical depth profiles of the modified structures on the Al, Au, and Cu surfaces induced by single and multiple pulse irradiations of the SXRL with fluences near the modification thresholds (12 mJ/cm$^2$, 20 mJ/cm$^2$, and 14 mJ/cm$^2$ for Al, Au, and Cu, respectively) have also been reported.[22] The irradiated surfaces of Al, Au, and Cu were modified by the single pulse irradiation, which had a depth of less than 100 nm. After the multiple pulse irradiations, both the depth of the damaged areas and the height of the rims of the modified structures on all the sample surfaces increased with increasing numbers of pulses. Under the ablation threshold, it is possible to create shallow structures by using single pulse irradiation. If the deposited fluence to the surface exceeds the ablation threshold, the ablation holes will appear, even on an Au surface.[18]

When we irradiated the Si surface with SXRL pulses, unique structures were found at the position where multiple 10 shot SXRL pulse irradiation was carried out.[22] Molten structures having depths greater than 600 nm were observed. The SEM image of this sample is shown in Fig. 5. The Zr filter was not used in this exposure, and at the position where only a single pulse was irradiated, no structure was detected. The measured depth was in accordance with the attenuation length of the SXRL beam for Si (590 nm). However, the Zr filter was not inserted in the SXRL beam axis, and thus, a portion of the radiated rays from the Ag target plasmas, such as visible and infrared light, reached on Si surface. The influence of the visible and infrared light on the surface modification could not be determined.

The differences in surface behavior induced by the SXRL pulse irradiations would be attributed to differences in the
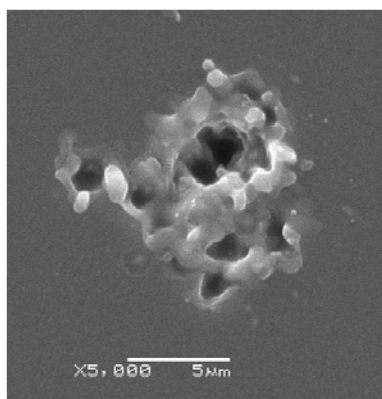
**Figure 5.**  SEM images of structures on Si surface induced by 10 shot irradiation of SXRL beam without Zr filter.

melting points of the sample materials and/or the attenuation lengths for the SXRL beam, because the other physical constants are almost the same. To understand the ablation and modification mechanisms occurring on the Al and Au surfaces resulting from the interaction with the SXRL pulse irradiation, we have previously discussed these mechanisms using the results of theoretical calculation.[15–18] For Al, spallation was proposed as the mechanism of surface modification, and the results of the model calculations were in good agreement with the experimental results. However, it is difficult to explain the formation of conical structures on the Al surface. For Au, the calculation results using the "atomistic model", which takes into account the molecular dynamics of a two temperature system with the inter-ionic electron-temperature-dependent potential, can reveal the modification processes. The modified depths by the irradiation of a single SXRL pulse could be explained by considering the molten layer.

The surface modifications occurring on the surfaces of materials, such as Al, Cu, and Au, resulting from interactions with an SXRL beam having low fluence are very interesting, not only in terms of a fundamental study but also in terms of technological applications. By using an SXRL pulse with a fluence of less than 20 mJ/cm$^2$, nano-meter scale modifications (not ablation) can be obtained on the material surface, as described above. Recently, we used an SXRL pulse to irradiate a nickel (Ni) surface. The Ni surface was also modified by a single SXRL pulse having a fluence approximately 17 mJ/cm$^2$. Judging from our experimental results, an SXRL fluence of 20 mJ/cm$^2$ is a critical value for the nano-meter scale surface modification of various materials. In addition, the small ablation or modification thresholds enable efficient machining on the material surface. Because the wavelength of an SXRL beam is shorter than that of visible light, the SXRL beam has the potential to draw small patterns, such as grooves or channels with nano-meter sale widths. If we construct an interferometer with double pulse interference, a periodical structure having several tens of nano-meter periods will appear on the surface. This periodical structure would work as x-ray diffraction optics. When mirror materials are deposited on the periodical structure, this periodical structure could serve as x-ray diffraction grating. A silicon nitride (Si$_3$N$_4$) membrane having thickness of several hundred nano-meters could be used as a transmittable substrate. A periodical structure fabricated on materials deposited on a Si$_3$N$_4$ membrane would work as a transmission-type light shaping diffuser for x-rays or transmission grating. The probability of fabricating transmission-type light shaping diffusers[23] for x-rays is worth considering. Because they increase the surface area, the conical structures formed on the Al surface could serve as catalytic substrates for chemical reactions, when the surface is coated with catalytic materials such as molybdenum, palladium, platinum, and so on. A nanopillar sheet for culturing biological cells[24] might be used to apply conical structures on Al surface. The formation of deep structures on Si surfaces could lead to novel structures buried within the Si substrate. When we can control the surface modification process by SXRL pulse irradiation, the SXRL beam could facilitate micromachining, which would enable us to fabricate three dimensional nano-meter size structures.

# 4. CONCLUSION

We irradiated Al, Au, Cu, and Si surfaces with the single and multiple SXRL pulses with a wavelength of 13.9 nm and duration of 7 ps, and observed the structures induced on each surface. Conical structures were formed on the Al surface, whereas ripple-like structures were confirmed on the Au and Cu surfaces. We also observed the modified surface structures generated after the accumulation of several SXRL pulses, and the development of these structures as a function of the number of pulses was confirmed. On the Si surface, deep holes were formed as a result of exposure to multiple SXRL pulses. It was found that the surface behavior of different materials upon exposure to SXRL beam irradiation varies significantly. This systematic study under a wide variety of experimental conditions shows that the optimal conditions for the formation of pure nano-meter size structures can be achieved with only single pulse irradiation on the target material using an SXRL beam with very low fluence. The obtained results may become good benchmarks for experiments using x-ray FELs in the near future. There are still many gaps in our understanding of the ablation mechanism induced by short pulse lasers. The present results may provide ideas for the construction of theoretical models that might address these gaps. Finally, our experimental and observed results provide ideas for the machining the surfaces of these materials. The wavelength of the SXRL is shorter than those of a visible or infrared laser, meaning that an SXRL potentially has the ability to draw small patterns. The modified surfaces had an average depth approximately several tens of nano-meters. Hence, the SXRL is a candidate a tool to fabricate nano-meter scale three dimensional structures.

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