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ORIGINAL PAPER

Ablation of single-crystalline cesium iodide by extreme ultraviolet capillary-discharge laser

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Abstract. Extreme ultraviolet (XUV) capillary-discharge lasers (CDLs) are a suitable source for the efficient, clean ablation of ionic crystals, which are obviously difficult to ablate with conventional, long-wavelength lasers. In the present study, a single crystal of cesium iodide (CsI) was irradiated by multiple, focused 1.5-ns pulses of 46.9-nm radiation delivered from a compact XUV-CDL device operated at either 2-Hz or 3-Hz repetition rates. The ablation rates were determined from the depth of the craters produced by the accumulation of laser pulses. Langmuir probes were used to diagnose the plasma plume produced by the focused XUV-CDL beam. Both the electron density and electron temperature were sufficiently high to confirm that ablation was the key process in the observed CsI removal. Moreover, a CsI thin film on MgO substrate was prepared by XUV pulsed laser deposition; a fraction of the film was detected by X-ray photoelectron spectroscopy.

Keywords: Ablation • CsI • Desorption • Laser • PLD • XUV

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Introduction

Laser ablation has been investigated for several decades. Laser ablation and desorption phenomena are of practical importance in numerous diverse applications. In the present study, we ablated a typical ionic crystal, i.e., cesium iodide (CsI), by short-wavelength nanosecond laser pulses.

In the past, attempts have been made to ablate CsI to prepare thin films by pulsed laser deposition (PLD) [1] with conventional, long-wavelength lasers [2, 3]. There are at least two strong sources of motivation for coating a chosen substrate with CsI. A thin film of CsI serves as (a) a widely used photocathode [4]; and (b) a traditional scintillator material [5, 6]. Recently, the nonthermal ablation of CsI induced by femtosecond visible laser pulses was also reported [7].

The very weak linear absorption of visible radiation in CsI encourages the use of extreme ultraviolet (XUV) and soft X-ray lasers to ablate the ionic crystal effectively [8, 9]. The absorption of short-wavelength radiation occurs due to an atomic photoeffect. The absorption is strong because CsI is composed of heavy elements and has a relatively high density, i.e., 4.51 g/cm^3.

In this study, the source of short-wavelength radiation was a desktop capillary-discharge laser (CDL) providing nanosecond pulses of 46.9-nm radiation at a repetition rate up to 12 Hz [10]. The XUV-CDL--induced ablation of the ionic crystal was studied both experimentally and by numerical modelling, and the etch rates and ablation thresholds were determined.

The XUV-CDL-produced ablation plume was diagnosed by a Langmuir double probe [11, 12] to reveal the fundamental parameters of the plasma, i.e., its electron temperature and electron density. To test the possibility of using the plume for PLD purposes, the attempt to create a thin CsI layer on the MgO substrate was undertaken.

Experimental

The arrangement of the XUV laser–matter interaction experiment is shown in Fig. 1. The angle of incidence between the surface normal and the focused laser beam was 0°, i.e., irradiation was performed under normal incidence conditions. The parameters were the focus–target distance (z scan with Dz = 0.2 mm) and the number of pulses (10, 20, 30, 50, 100, and 111 pulses). The target was a single crystal of CsI placed on a motorized *xyz* positioning stage. The pressure in the vacuum interaction chamber was 10^{-5} mbar. The source of XUV radiation was a compact Ne-like collisionally pumped Ar CDL [10] with a 21-cm-long Al_2O_3 argon-filled capillary emitting at a wavelength of 46.9 nm (i.e., photon energy of 26.4 eV).

XUV pulses provided by the CDL device exhibited a duration and energy of 1.5 ns (full width at half maximum (FWHM)) and up to 10 mJ, respectively. The CDL beam was focused using a 50-cm focal length Sc/Si multilayer mirror with a reflectivity of 30% [13]. The CDL repetition rate was 2 Hz and 3 Hz for the ablation investigation and the PLD test, respectively. Pulse energy of 2 mJ was registered at the target surface after beam guiding and focusing in the vacuum interaction chamber. This value was considered during an estimation of the XUV laser fluence. From the mathematical model of the beam caustics [13] and the known energy delivered within the pulse, the maximum power density on the sample surface was found to be as high as approximately 250 MW/cm^2.

After irradiation, the target surface was analysed by an optical surface profiler based on white light interferometry (WLI; Zygo NewView 7300). The WLI data were analysed using Gwyddion software [14]. The optical profiler provided information on the crater shapes and ablation depths. An analysis of the craters produced at various fluences enabled determination of the ablation threshold. The experimentally determined value was compared to the value calculated using the XUV-ABLATOR computer code [15].

In a previous work [12], we measured both the electron temperature and the density of the plasma plume produced by the XUV-CDL beam focused on a bismuth (Bi) target with a Langmuir probe in both the single and double versions. In the present study, the double-probe technique was applied because the target was an insulator. The measurement was conducted using two parallel tungsten wires with lengths of 1 mm and diameters of 0.1 mm. The distance between the wires was 0.7 mm, and the plane of the wires was parallel to the surface of the target. The probe was located 0.6 mm from the target. To minimize the increased noise level of the electrically insulating target (CsI), we used batteries to bias the probe instead of the standard direct current (DC) power supply utilized in our previous work [12], in which the target was a good conductor (Bi). The target was held on a rotating stage.

A CsI thin layer deposited on the MgO substrate by PLD using the experimental arrangement shown in Fig. 1 was characterized *ex situ* by means of X-ray photoelectron spectroscopy (XPS). The XPS measurements were performed in a ultra-high vacuum (UHV) chamber with a base pressure of about 5 × 10^{-10} mbar. The system was equipped with a Mg/Al dual-anode X-ray source, an Omicron EA 125 hemispherical electron analyzer with a total resolution of about 1 eV [16]. Al K_α radiation, with primary energy = 1486.6 eV, was used in this particular experiment. The binding energies (E_B) of the obtained photoelectron spectra were calibrated using C1s line at E_B = 284.5 eV.

Results and discussion

The typical shape of a crater produced in the single--crystalline CsI by the focused XUV-CDL beam is shown in Fig. 2.

Multiple pulse irradiations were performed using different numbers of shots, and the resulting series of craters was analysed. As shown in Fig. 3, the maximum depth of a particular crater increases with the number of pulses. The width of a crater is almost independent of the number of laser pulses. For a few pulses, the ablation rate (i.e., the thickness of the material layer removed by one pulse, Fig. 4) decreases, reaching a plateau at 100 pulses. This decrease was previously found for other ionic crystals [15]. This effect could be caused by (a) radiation--induced hardening of the irradiated material and/or (b) the development of the crater shape, which obstructs the exit of the material from the crater, enhancing its redeposition on the crater wall.

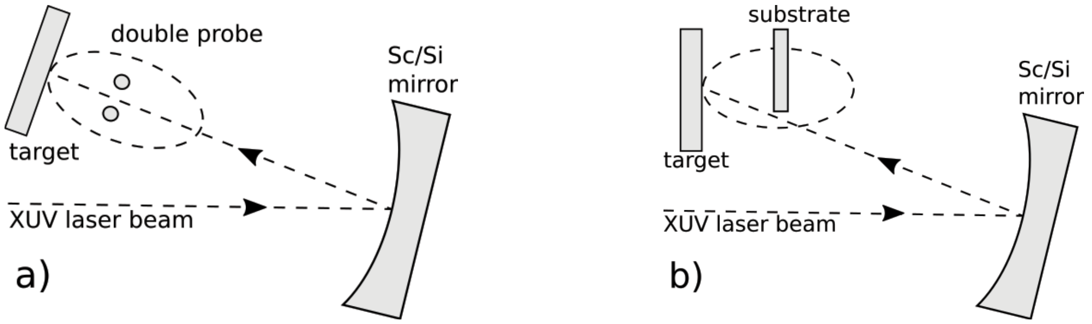

Fig. 1. Experimental arrangement. (a) The plasma plume is measured by a pair of probes; (b) for the deposition of the thin film, an MgO (001) substrate was placed in the probe position at a distance of 2 mm from the target. Not to scale.

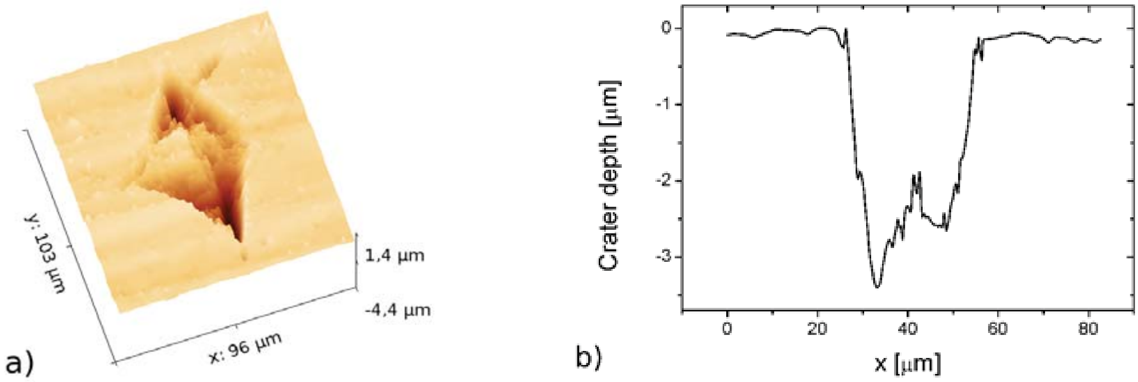

Fig. 2. (a) WLI image of the CsI surface after XUV-CDL-induced ablation at a fluence value slightly above the ablation threshold. The image was taken by a Zygo optical profiler after 100 laser pulses. (b) The maximum depth of the crater is 3.5 mm. The ablation processes dominate the material removal. WLI = white light interferometry.

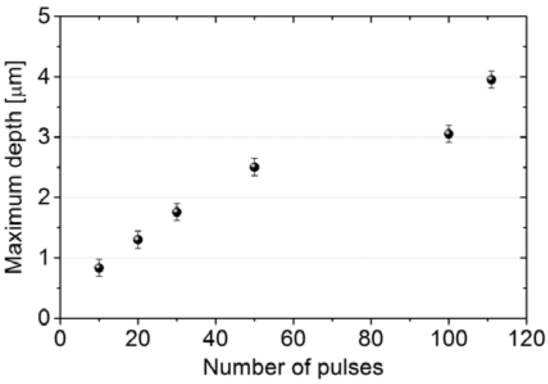

Fig. 3. Dependence of the maximum crater depth on the number of laser pulses.

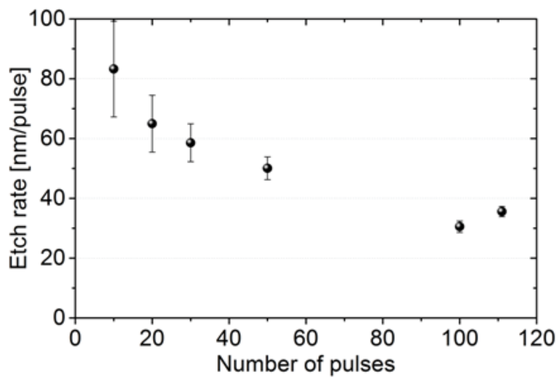

Fig. 4. Dependence of the etch rate on the number of pulses.

Ablation threshold evaluation

Ablation is a threshold process. For the estimation of a fluence threshold F_{th} (i.e., the maximum fluence when the etch rate is equal to zero), we use the Beer–Lambert law:

(1) $$d = a \ln(F/F_{th})$$

where d is the maximum crater depth, a is the attenuation length of the 46.9-nm radiation in CsI (38 nm as calculated using Henke's tables [17]), and F is the focused laser beam fluence. In Fig. 5, the data are shown after being processed using Eq. (1). F_{th} is computed from constant c in the linear fit, as follows:

(2) $$y = x + c$$

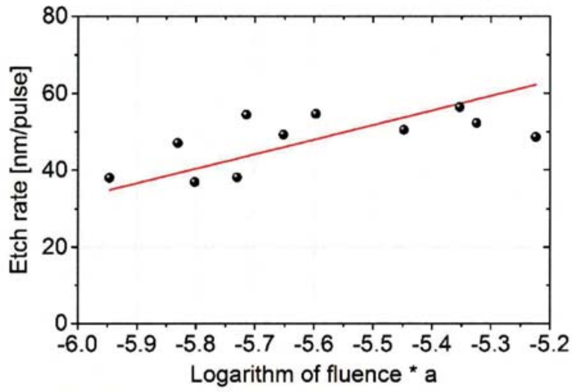

Fig. 5. Dependence of the etch rate on the fluence. Etch rates were calculated from the 30-shot z scan ('a' is the attenuation length).

(the red line in Fig. 5), where y is d/a, x is the estimation of $\ln(F)$ as $\ln((E \cdot y)/S)$, and c is equal to $-\ln(F_{th})$. Further, S is the experimentally measured area of the focused beam on the irradiated surface from the 30-shot ablation patterns.

For a similar number of pulses, the maximum etch rate in CsI (60 nm) is approximately three times higher than the value obtained previously in LiF (20 nm [18]). The difference can be explained by the attenuation lengths of 46.9-nm radiation in CsI and LiF, which are 38 nm and 13.6 nm, respectively.

The threshold fluence for the XUV-CDL-induced ablation of CsI is F_{th} = 105 mJ/cm^2 (the data used for this estimation are shown in Fig. 5). This estimation is the upper level of the ablation threshold for a single-shot process because of the declining etch rate. Numerical modelling of the XUV-induced CsI removal, performed with the 1D finite-difference Lagrangian code (XUV-ABLATOR [15]), provides a prediction of the maximum CsI etch rate, d 62 ± 11 nm per pulse, at a peak fluence of 0.375 J/cm^2 achieved in a tight focus. Computer simulations revealing the etch rate dependence on the fluence (Fig. 6) indicate that the ablation threshold is approximately 0.15 J/cm^2. This value is in good agreement with the threshold obtained experimentally.

The ablation threshold obtained here in CsI is very close to the values of 0.06 J/cm^2 [18] and 0.1 J/cm^2 [19] found previously for LiF, which is an expected result because in CsI and LiF, the attenuation length of the 46.9-nm radiation is 38 nm and 13.6 nm, respectively, and the lattice energy is 5.7 eV and 10.56 eV, respectively [20]. In contrast to LiF, the longer attenuation length in CsI (38 nm) results in a lower energy density in the near-surface region at the same surface fluence; however, a lower amount of energy is required to decompose the CsI lattice (5.7 eV). Therefore, the XUV-CDL energy densities needed to trigger the ablation of CsI and LiF are similar.

The ablation threshold at 0.1 J/cm^2 found for 1.5-ns pulses of XUV laser radiation is located lower than the ablation threshold 0.25 J/cm^2 determined in

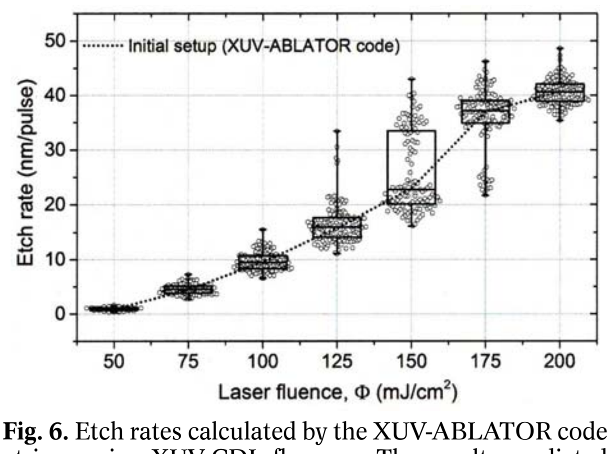

Fig. 6. Etch rates calculated by the XUV-ABLATOR code at increasing XUV-CDL fluences. The results predicted with the initial setup of the input parameters (dotted line with star symbol) are depicted together with the values of the etch rate of the sample (box and whiskers) obtained by a quasi-Monte Carlo approach considering the relevant uncertainties of the modelling input parameters.

CsI illuminated by 50-fs pulses of visible radiation (400 nm; second harmonic of Ti:sapphire laser) [7]). Even taking into account an order of magnitude difference in attenuation length (which could be revealed from the etch rates), the XUV laser ablation begins at much lower irradiances (approximately 70 MW/cm^2) than the ablation induced by ultrashort pulses of visible laser radiation, which requires at least 5000 GW/cm^2. The difference in thresholds can be explained by a difference in the photon energies. Even a single photon of XUV laser radiation carries enough energy to induce a point defect in the CsI lattice, while in the visible spectral range, multiphoton absorption is needed to reach energies able to damage the lattice. The probability of multiphoton processes is significantly lower with respect to single-photon effects. Therefore, the threshold irradiance should be much higher when long-wavelength laser radiation is utilized to ablate an ionic crystal.

The determination of the ablation threshold at ~0.1 J/cm^2 sheds light on a previous soft X-ray laser experiment conducted with CsI [6]. In that experiment, radioluminescence of CsI was induced by a Ne-like quasi-steady-state collisionally pumped Zn soft X-ray laser (wavelength: 21.2 nm, pulse duration: 80 ps). Experiments were conducted with irradiance of 1.0 GW/cm^2 or 6.7 MW/cm^2 at the sample surface. The highest intensity was obtained by adding a Si/Mo multilayer mirror to the laser to form a half-cavity that allows for double-pass amplification [6]. The arrangement with the half--cavity corresponds to lower yield (lower efficiency of the soft X-ray-laser-induced luminescence) in comparison to the lower irradiance case without the half-cavity. Our results indicate that substantial decomposition of the lattice could be responsible for the lack of luminescence efficiency at higher irradiance (i.e., during the irradiation by the laser with the half-cavity) reported earlier [6].

Plasma characterization

A typical signal obtained from the double probe immersed in the plasma plume is shown in Fig. 7. Registered data were processed by techniques described in detail elsewhere [12]. The plasma parameters in the plume are as follows: the electron temperature T_e reaches 7.5 eV and the electron density n_e is 8.7 × 10^{14} m^{-3}. Both values are significantly higher than the parameters of the Bi plasma achieved in our previous study [12].

The difference in density may be due to the ablation characteristic of CsI removal, in contrast to the desorption phenomena (as defined, e.g., in an earlier paper [21]) responsible for the formation of the Bi plasma plume reported previously [12]. A desorption process, even if induced by energetic photons, typically removes a thin layer of material per laser pulse [22]. Ablation blows off much more material than desorption. The dense material ablated by the front of the incoming XUV-CDL pulse is efficiently ionized and heated by the remainder of the pulse, which results in higher electron density n_e. The higher

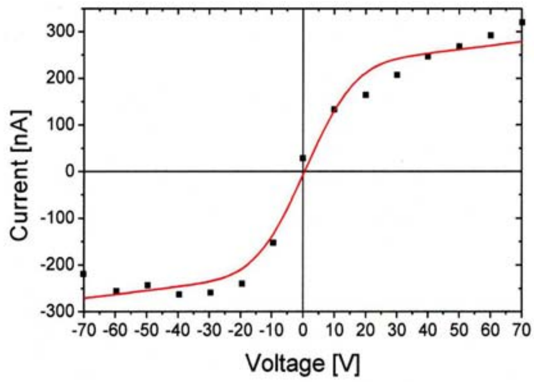

Fig. 7. Double-probe signals registered in the plasma plume normal to the CsI target surface (the target-probe distance is 0.6 mm).

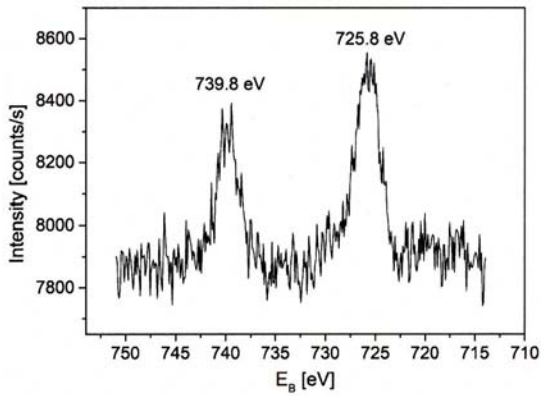

Fig. 8. XPS signal of the submonolayer deposited on the MgO substrate showing Cs peaks of $3d_{3/2}$ and $3d_{5/2}$.

electron temperature registered in the present study is also due to the difference in thermophysical properties of the chosen targets: in the case of CsI, ablation takes place and the ablation plasma plume formed under the given conditions contains a higher amount of energy. This is so mostly due to the fact that CsI is an insulator, in which the rapid release of the energy absorbed by heat conduction from the near-surface region into the bulk of the target is not possible, in contrast to the case of a conductor, such as Bi.

Thin film preparation

For the purpose of thin film preparation, 10 000 laser pulses (corresponding to the lifetime of one capillary) were used. X-ray photoelectron spectroscopy (XPS) spectra have demonstrated that deposition by the PLD method is partially successful. Using the ratio of the substrate signal (Mg 1s peak – not presented here) and the intensity of the deposited Cs 3d doublet, it can be concluded that the deposited amount of CsI is equivalent only to a fraction of the monolayer. The Cs 3d doublet spectrum is presented in Fig. 8. The binding energies E_B = 725.8 eV and 739.8 eV correspond to Cs $3d_{3/2}$ and $3d_{5/2}$ peaks, respectively. Cs is probably in the iodide or oxide form. The E_B of $3d_{5/2}$ electrons in these species should be about 724 eV. The higher E_B measured corresponds to the supposed thin noncontinuous deposited film. No signal of iodide was indicated in the spectra. It is possible that in the early phase of thin layer formation, the crystal CsI was not created, so that iodide atoms tend to sublimate. Note that a thin layer prepared by another capillary laser CAPEX [23] showed similar properties.

Conclusions

Taking into account the uncertainty related to the absolute calibration of the pulse energy measurement and the shot-to-shot fluctuations of the XUV-CDL output energy, it can be concluded that the experimentally determined etch rates and the threshold fluence for CsI are reproducible and can be compared to the values obtained in computer simulations conducted with the XUV-ABLATOR code. The agreement between the experiment and theory is satisfactory – the ablation threshold is located at ~0.1 J/cm^2 in both cases. The erosion of irradiated material is mainly due to ablation, with the desorption process playing a minor role. Ablation dominates the erosion processes because the XUV-CDL-induced rate of material removal quickly exceeds the attenuation length of the XUV laser radiation in the material. This fact is also supported by results of the double-probe measurement. The electron temperature of the plasma plume (~7.5 eV) is noticeably higher than in the case of Bi plasma [12].

The low effectiveness of material transfer during PLD is a result of the relatively low pulse energy delivered by the XUV-CDL device at the 2-Hz repetition rate used in this experiment. The pulse energy of the XUV-CDL is at the microjoule level, whereas standard UV excimer lasers, which are typically used in PLD experiments, deliver hundreds of millijoules in a single pulse. Nevertheless, this study has produced one of the first thin films of an ionic crystal material, which has been prepared by PLD with an XUV laser and, at the same time, it has showed the possibility of further investigation in the early phases of thin film growth.

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