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Interaction of wide-band-gap single crystals with 248-nm excimer laser irradiation. X. Laser-induced near-surface absorption in single-crystal NaCl

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Ultraviolet laser-induced desorption of neutral atoms and molecules from nominally transparent, ionic materials can yield particle velocities consistent with surface temperatures of a few thousand kelvin even in the absence of visible surface damage. The origin of the laser absorption required for this surface heating has been often overlooked. In this work, we report simultaneous neutral emission and laser transmission measurements on single-crystal NaCl exposed to 248-nm excimer laser radiation. As much as 20% of the incident radiation at 248 nm must be absorbed in the near-surface region to account for the observed particle velocities. We show that the laser absorption grows from low values over several pulses and saturates at values sufficient to account for the surface temperatures required to explain the observed particle velocity distributions. The growth of absorption in these early pulses is accompanied by a corresponding increase in the emission intensities. The diffuse reflectance spectra acquired after exposure suggest that near-surface *V*-type centers are responsible for most of the absorption at 248 nm in single-crystal NaCl. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1847696]

I. INTRODUCTION

Laser interactions with nominally transparent materials have important implications in laser ablation deposition, laser surface modification, laser-based chemical analysis, and laser damage. For a number of years we and others have investigated the desorption of particles (ions and neutral atoms and molecules) due to exposure to photons with energies well below the band gap and at fluences well below the threshold for plasma formation.[1–23] In many of these systems, surface and near-surface defects play critical roles in particle desorption. Similar defects may also play an important role in desorption from nanoparticles.[24,25]

In many cases, the time-of-flight (TOF) distributions of neutral particles desorbed from nominally transparent materials are consistent with Maxwell–Boltzmann velocity distributions with temperatures of 1000–2500 K.[22,23,26–28] The origin of the surface heating required to produce these high effective temperatures has often been overlooked. In this work, we show that the absorption of single-crystal NaCl during 248-nm irradiation grows from low values over several laser pulses to high values consistent with the measured, effective surface temperatures. In this case, the high surface temperatures are associated with the end state of a rapid incubation or induction process, where the density of near-surface defects grows with exposure. The neutral emission intensities also grow from low values over this same time scale. We show that defects critical to the desorption process and surface heating are generated by the laser itself via two-photon absorption. At higher fluences, these near-surface defects are expected to play an important role in optical breakdown and surface damage.

II. EXPERIMENT

The results described below employed cleavable single-crystal NaCl obtained from Almaz Optics (West Berlin, New Jersey). The experimental arrangement has been recently described in Ref. 23. Pulsed laser radiation at 248 nm was provided by a Lambda Physik Lextra 200 excimer laser (KrF excimer, 5-eV photons, 30-ns pulse width). Irradiation was carried out at a pressure of about 10^{-7} Pa. Desorbed neutral particles were detected with a UTI 100C quadrupole mass spectrometer (QMS) after electron-impact ionization. The output of the mass spectrometer was amplified, discriminated, and pulse counted by fast electronics.

An estimate of the peak surface temperature T during the laser pulse can be derived from the time of flight of thermally desorbed particles. At particle removal rates well less than a monolayer per pulse, the time of flight can often be modeled by a delta function source of effusing particles with a Maxwell–Boltzmann velocity distribution. If N particles are emitted from the surface, the signal detected with the quadrupole ionizer mounted a distance d from the surface, with d much greater than the dimensions of the ionizer, is given by

$$I(t) = \left(\frac{m}{2\pi kT}\right)^{3/2} \frac{\alpha NVd}{(t-t')^4} \exp\left(\frac{-md^2}{2kT(t-t')^2}\right), \qquad (1)$$

where α is a constant that accounts for the ionization and transmission efficiencies, assumed to be uniform over the volume V of the ionizer. The constant t' accounts for the

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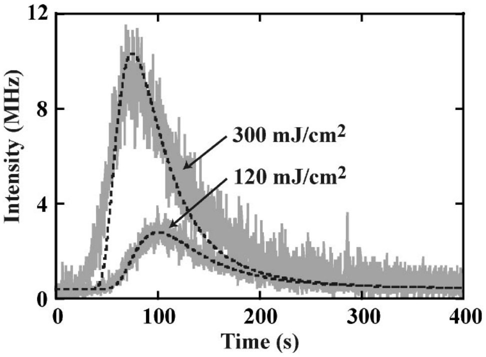

FIG. 1. Time-of-flight curves for neutral emissions at 35 amu/e (Cl^0) during exposure of single-crystal NaCl to a 248-nm laser radiation at fluences of 120 and 300 mJ/cm^2. The sample mount was heated to maintain a background sample temperature (between laser pulses) of 800 K. The dotted lines are least-squares fits of Eq. (1) to the data.

time between particle ionization and detection, and k is the Boltzmann constant. It should be emphasized that all work presented here is at fluences well below those required for the formation of any plasma or plume.

III. RESULTS AND DISCUSSION

A. Time-of-flight signals and effective surface temperatures

At relatively low laser fluences, 248-nm irradiation of NaCl produces a variety of neutral products, including neutral Na^0, Cl^0, and small amounts of Cl_2^0 and $NaCl^0$. Typical TOF signals for neutral Cl^0 (mass-to-charge ratio of 35 amu/e) at two laser fluences appear in Fig. 1. In recent work, we have ruled out molecular NaCl as a major source for the two signals.[23] Further, the data indicate that the velocity distributions are not strongly affected by collisions after emission.[23,29,30]

Least-squares fits of Eq. (1) to the data are shown by the dotted lines in Fig. 1, verifying that these emissions are described by the appropriate Maxwell–Boltzmann distributions. These curve fits to the two signals yield temperatures of 2150 K at 120 mJ/cm^2 and 3800 K at 300 mJ/cm^2. The background sample temperature during these measurements was maintained at 800 K with a resistive sample heater. Taken at face value, these time-of-flight curves would therefore indicate laser-induced temperature changes of 1350 and 3000 K, respectively. A similar analysis of the Na^0 time-of-flight data acquired at these two fluences yields very similar temperatures.[23] (The Na^0 fits show slightly higher temperatures, which we have shown to be due to the contribution of Cl_2 fragments to the signal at 35 amu. This would lower the apparent temperature of the Cl^0 signal.) The fits to the data suggest that the great majority of both species are emitted in quasithermal equilibrium from a laser-heated surface at temperatures above the melting point of NaCl (1074 K). Thermal relaxation following nanosecond laser-induced heating is relatively fast (<1 μs). The high effective temperature of these emitted particles indicates that the great majority of emission occurs in this submicrosecond period of high surface temperature.

When the emission intensities are plotted in Arrhenius form against the apparent surface temperature derived from the time-of-flight signals, both emissions show Arrhenius-like behavior with an activation energy of approximately 0.30 eV.[23] This suggests that both atomic emissions are rate limited by the same, thermally activated process. Höche *et al.* have observed a similar activation energy in the recovery of helium atom scattering signals from UV-irradiated NaCl surfaces when the UV source is turned off.[31] We have recently argued that emission during 248-nm irradiation is a thermally-assisted, photoelectronic process similar to that observed by Höche *et al.* near room temperature.[23]

B. Absorption and surface heating

Although similar temperatures have been observed in a number of our earlier studies involving several wide-band-gap ionic materials, we have had difficulty in accounting for the associated laser absorption in transparent materials.[21,27,32–36] Physically plausible mechanisms require that absorption be concentrated within a thin surface layer. A simple estimate of the temperature change produced when an energy per unit area E is deposited at the surface of a material with a constant thermal conductivity k and thermal diffusivity a in a time $\tau=30$ ns (the duration of the laser pulse) is given by[37]

$$\Delta T = \frac{2E}{k}\sqrt{\frac{a}{\pi\tau}}. \tag{2}$$

For convenience, we use thermal constants for NaCl at 600 K: $k\sim 0.04\ \mathrm{cm^2\,s^{-1}}$ and $a\sim 0.03\ \mathrm{W\,cm^{-1}\,K^{-1}}$. So the temperature change of about 1000 K observed at a fluence of 100 mJ/cm^2 requires the absorption of about 20 mJ/cm^2 along the surface. Thus about 20% of the incident radiation must be absorbed within the first few hundred nanometers along the surface. This is a remarkably strong absorption for a nominally transparent material like NaCl.

Since our time-of-flight signals represent averages over several tens of laser pulses, our data allow the possibility that the required absorption and emission develop gradually over several laser pulses (a so-called induction or incubation process). Measurements of the neutral emission intensities (Na^0 and Cl^0) for each pulse over the first 20 laser pulses at a fluence of 100 mJ/cm^2 are shown in Fig. 2 for different samples. Consistent with the induction hypothesis, emission grows in a few pulses from low values to the much larger emissions represented in Fig. 1. The more intense emissions observed after induction would tend to dominate the total emission intensities, and thus yield time-of-flight signals consistent with the high temperatures expected after induction. Unfortunately, the low signal intensities observed during the first few laser pulses do not permit temperature estimates on a pulse-by-pulse basis during induction.

The growth of absorption at 248 nm during pulsed irradiation can be observed by measuring the intensity of the transmitted laser light with a conventional power meter. Figure 3 displays the transmitted energy on a pulse-by-pulse basis during the first 20 laser pulses at pulse energies from

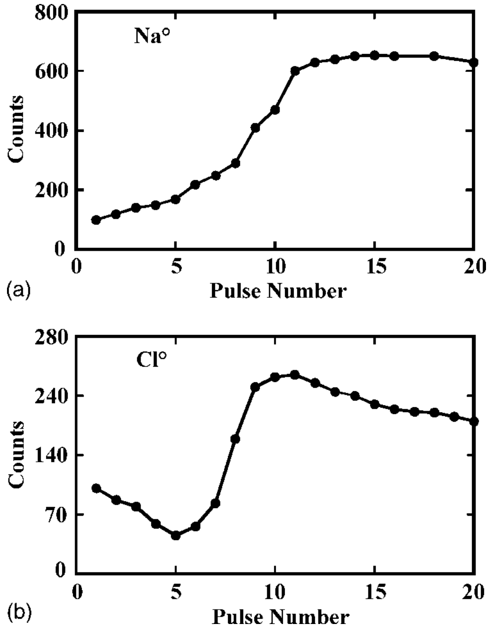

FIG. 2. (a) Total Na^0 and (b) total Cl^0 counts per pulse detected during the first 20–248-nm laser pulses incident on two different cleaved NaCl samples at a laser fluence of 100 mJ/cm^2. The background substrate temperature was 300 K.

50 to 300 mJ/cm^2. In each case, the energy transmitted in the absence of the sample has been assigned a value of 1.0.

At each fluence, the transmission signal drops markedly during the first several pulses and saturates in the limit of long exposure. This saturation is consistent with a relatively constant amount of near-surface laser absorption after the first several laser pulses. Thus the peak surface temperature during the remainder of a 100-pulse time-of-flight signal acquisition should be reliable. The dark lines in Fig. 3 represent least-squares fits to a model described below.

C. Diffuse reflectance spectra

Some indication of the nature of the relevant defects appear in the diffuse reflectance spectra of cleaved NaCl exposed to 50 pulses of 248-nm laser radiation at 300 mJ/cm^2. Laser irradiation produces strong composite absorption peaks in the 220–280-nm region, attributed to *V*-type centers, and in the 420–520-nm region, attributed to *F*-type centers. A weaker absorption centered at 720 nm coincides with the absorption of the F_2 center (also called the *M* center).

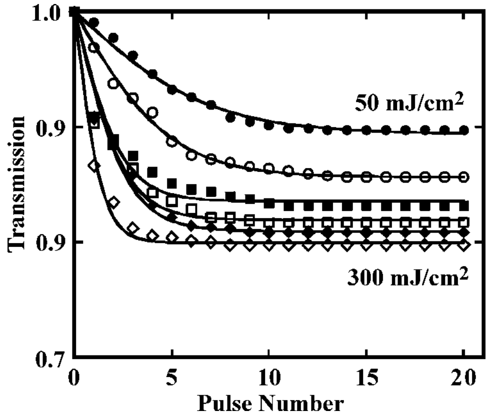

FIG. 3. Laser transmission through cleaved, single-crystal NaCl at 248 nm measured on a pulse-to-pulse basis for the first 20 pulses at fluences of 50, 100, 150, 200, 250, and 300 mJ/cm^2. The solid curves represent fits to Eq. (3).

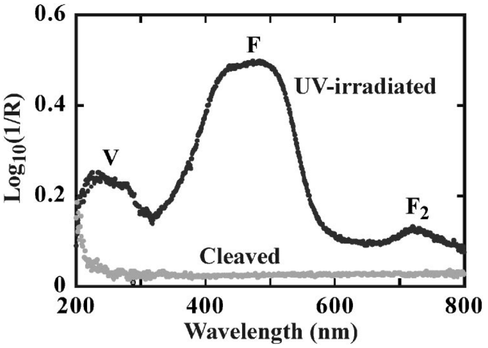

FIG. 4. Diffuse reflectance spectra of single-crystal NaCl exposed to 50 pulses of 248-nm laser radiation at 300 mJ/cm^2. The peaks near 250 and 470 nm can be assigned to composite *V*- and *F*-like defects, respectively. The peak at 720 nm coincides with the absorption of the F_2 center.

V-type centers would account for the strong absorption at 248 nm. At room temperature, stable *V* centers in NaCl involve Cl_2^- or Cl_3^- units associated with other defects, such as cation vacancies or impurities. Casler *et al.*[38] observed an absorption at 220 nm that would account for the room-temperature absorption on the UV end of the broad peak in Fig. 4. Bazhin *et al.*[39] assigned this absorption to a Cl_3^- unit associated with a NaCl divacancy. Divacancies can be produced in great numbers by dislocation jog dragging during deformation[40,41] and cleavage.[42–44] Thermodynamic arguments suggest that divalent impurities are also highly concentrated in a layer immediately below the surface,[45] which would also trap Cl_2^- units that absorb in this wavelength range.

The broad peak in the 420–520-nm region is composite in nature, involving the overlapping *F*-center and F^--center bands. The *F* center is a halogen vacancy with a trapped electron, which absorbs near 460 nm. The F^- center is a halogen vacancy with two trapped electrons. The F^- absorption typically peaks near 440 nm,[46] but is much broader than the *F*-center absorption and displays a long tail on the long-wavelength side. Both transmission spectra (not shown) and the diffuse reflection spectrum shown here are well described by combining these defect center bands.

Diffuse reflection spectroscopy is especially sensitive to defects in the near-surface region. Assuming that the thickness of the surface layer probed by the reflected beam is on the order of a wavelength, the observed *V*-center absorption change of at least 0.03 units corresponds to an absorbance of 600 cm^{-1}. The defect density in the near-surface region associated with this strong absorption is therefore quite high.

D. Model of near-surface transmission change

Defect production in the alkali halides by electronic excitations has been extensively studied[47] and will be only schematically treated here. In NaCl, we recently showed that at 248-nm (30-ns pulse duration), detectable two-photon absorption occurred at fluences above ~80 mJ/cm^2.[23] The results presented here are totally consistent with two-photon absorption. Electron-hole pairs generated by two-photon absorption form self-trapped excitons which decay into *F*-*H* center pairs, where *H* centers consist of a molecular halogen ion (here Cl_2^-) on a halogen site. At room temperature, *H* centers are mobile and can recombine with *F* centers or become trapped at other lattice defects to form the *V* centers which we observe here (Fig. 4).

In a recent work, we have modeled the growth of absorption in the NaCl *F*-center region due to femtosecond laser radiation at 800 nm by an equation of the form[48]

$$\rho(N) = \alpha \tanh(\beta N^n), \tag{3}$$

where $\rho(N)$ is the density of *F* centers versus *N*, the number of laser pulses and α and β are adjustable constants. Nonintegral values of the exponent *n* are often observed when defect motion involves multiple trapping and retrapping events. Least-squares fits of Eq. (3) applied to the transmission data in Fig. 3 are shown by the dark lines; the best fits were determined with a value of $n=0.6\pm0.1$. Similar exponents are observed in charge transport through amorphous semiconductors.[49] The excellent fit of Eq. (3) to the transmission data in the *V*-center region of the spectrum in this work is consistent with *H*-center transport to the surface, where reactions with dislocation- or impurity-related defects form high concentrations of near-surface *V* centers.

E. Laser-induced heating

Given near-surface *V* centers of sufficient density, heating follows by nonradiative deexcitation following linear absorption of the laser light. From simple loss calculations, we find that the duration of this elevated temperature is quite short, on the order of tens of nanoseconds. Therefore, the duration of thermally activated emission should also be quite short. The highest apparent surface temperatures indicated by the time-of-flight data exceed the boiling point of NaCl (1686 K). Further, the emission mechanism does not appear to change as the effective temperature passes through the melting and boiling points. In a previous work, we have shown that the apparent activation energy remains ~0.30 eV for effective surface temperatures both below and above the melting and boiling points.[23] This activation energy is well below the enthalpy for NaCl sublimation (2.23 eV). Recent atomic force microscopy (AFM) imaging of NaCl surfaces irradiated under similar conditions show flat terraces with roughened atomic steps and monolayer islands of aggregated NaCl (Ref. 23) that are incompatible with melting. This raises two significant questions: (1) Do the apparent temperatures determined from time-of-flight measurements really indicate peak surface temperatures during the laser pulse? and, if so, (2) Why do we not see evidence of surface melting at these high temperatures?

Studies of desorption from metals by nanosecond lasers have occasionally yielded kinetic-energy distributions corresponding to temperatures well above the boiling point. An extreme case is provided by Bennet and Farrelly, who observed Hg atoms desorbed from liquid Hg with Maxwell–Boltzmann energy distributions at apparent temperatures above the critical temperature;[50] although these temperatures are inconsistent with thermal equilibrium, Arrhenius plots analogous to those in our work yield activation energies consistent with the latent heat of vaporization. Even at lower laser fluences and powers, the effective temperatures of emissions from metals can be well above estimates based on known optical properties.[24,25,50,51] We note that, in the case of NaCl, the highest measured effective temperatures are much less than the critical temperature (7500 K).[52] Equally important, we do in fact observe sufficient laser absorption to account for the observed temperatures. We therefore suggest that, in the case of NaCl, the apparent temperatures reflect actual peak surface temperatures.

The absence of a modulation in emission intensity as the effective surface temperature passes through the melting point is also an important issue. During gradual heating, the vaporization rate of NaCl changes dramatically as the material passes through the melting point.[53] Time-resolved reflectively measurements provide unambiguous evidence for melting in some other material systems. In the case of single-crystal silicon, clear evidence for melting is observed following 248-nm laser heating to near the expected melting point.[54] Scanning tunneling microscope observations of laser-melted Si also show an atomic scale order with no evidence of disorder.[55] In contrast, the appearance of disordered roughened step edges and monolayer pits on NaCl cannot be interpreted in terms of melting.[23,56,57] (Meltlike features are produced by exposure to higher fluences yielding optical breakdown.[7])

In laser-heated NaCl, disordering of the surface due to melting may be hindered by the close-packed ionic nature of the lattice and the thinness of the heated layer. Unlike single-crystal metals and silicon, the molecular motions associated with melting in NaCl require considerably more free volume than is present in the solid. The volume change associated with heating NaCl from room temperature through the melting point is very large—over 40%. Constraints provided by the underlying lattice will largely confine this expansion to the direction normal to the surface. Motions are additionally constrained by the need to maintain local charge neutrality. These factors would provide large kinetic barriers to melt nucleation in thin heated layers on NaCl. In this case, steps and kinks continue to provide low-energy, site-density-limited paths for the emission of adsorbed atoms, similar to the reaction paths responsible for emission at lower temperatures.

IV. CONCLUSIONS

In this paper we have shown a complete picture of the sequence of events leading to robust, high-temperature desorption of neutral atoms from a nominally transparent material. During the initial stages of exposure to nanosecond pulses of 248-nm laser radiation, cleaved, single-crystal NaCl develops a strong UV absorption in a thin layer along the surface via two-photon electron-hole production. Diffuse reflectance spectroscopy on the exposed samples show strong, composite absorption bands in the 250-nm region consistent with *V*-type color centers. At fluences between 50 and 300 mJ/cm^2, less than ten pulses are required to produce a strong absorption. Subsequent linear absorption in this defective layer heats the surface. The apparent surface temperatures derived from particle time-of-flight distributions are thousands of kelvin, well above the boiling point of this material. Although significant questions remain, we suggest that meltlike surface disordering is strongly hindered, and that solid-state-like emission processes dominate even at high transient temperatures. The high absorption observed in these samples after several laser pulses strongly supports our contention that the high apparent surface temperatures derived from the time-of-flight data reflect actual surface temperatures with important impact on emission processes. At higher laser fluences, this heating would contribute to laser damage and the onset of optical breakdown.

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