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## LASER-INDUCED DAMAGE OF CALCIUM FLUORIDE

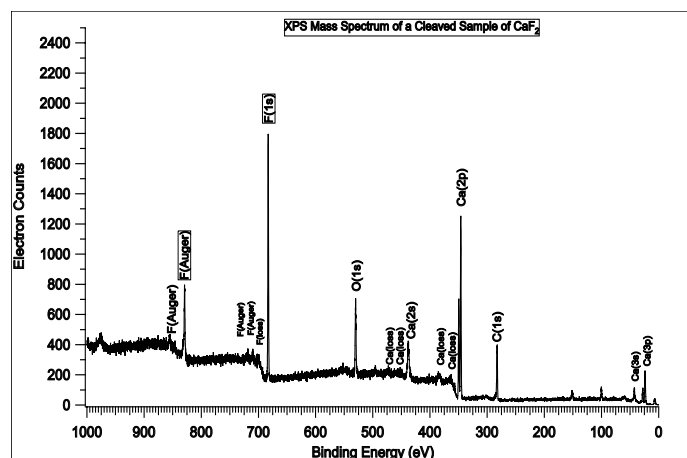
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### ABSTRACT

As advances continue to be made in laser technology there is an increasing demand for materials that have high thresholds for laser-induced damage. Laser damage occurs when light is absorbed, creating defects in the crystal lattice. These defects can lead to the emission of atoms, ions and molecules from the sample. One specific field where laser damage is of serious concern is semiconductor lithography, which is beginning to use light at a wavelength of 157 nm. CaF<sub>2</sub> is a candidate material for use in this new generation of lithography. In order to prevent unnecessary damage of optical components, it is necessary to understand the mechanisms for laser damage and the factors that serve to enhance it. In this research, we study various aspects of laser interactions with CaF<sub>2</sub>, including impurity absorbance and various forms of damage caused by incident laser light. Ultraviolet (UV) laser light at 266 nm with both femtosecond (fs) and nanosecond (ns) pulse widths is used to induce ion and neutral particle emission from cleaved samples of CaF<sub>2</sub>. The resulting mass spectra show significant differences suggesting that different mechanisms for desorption occur following excitation using the different pulse durations. Following irradiation by ns pulses at 266 nm, multiple single-photon absorption from defect states is likely responsible for ion emission whereas the fs case is driven by a multi-photon absorption process. This idea is further supported by the measurements made of the transmission and reflection of fs laser pulses at 266 nm, the results of which reveal a non-linear absorption process in effect at high incident intensities. In addition, the kinetic energy profiles of desorbed Ca and K contaminant atoms are different indicating that a different mechanism is responsible for their emission as well. Overall, these results show that purity plays a key role in the desorption of atoms from CaF<sub>2</sub> when using ns pulses. On the other hand, once the irradiance reaches high levels, like that of the fs case, significant desorption is possible due to multi-photon absorption by the intrinsic material.

## INTRODUCTION

Radiation damage of materials has long been of fundamental interest, especially since the advent of laser technology. One such source of damage comes from UV laser light. Laser systems continue to move into shorter wavelength ranges, but unfortunately are limited by the damage threshold of their optical components. For example, semiconductor lithography is beginning to utilize laser light at 157 nm and requires a material that can not only transmit this light, but also withstand



**Figure 1:** Results from XPS measurements on a cleaved sample of  $\text{CaF}_2$

the highly energetic photons present at this wavelength.  $\text{CaF}_2$ , an alkaline earth halide, is the chosen material for vacuum UV 157 nm excimer radiation. It can transmit light down to 120 nm and is relatively inexpensive. Although it is readily available through natural and synthetic sources, it is often difficult to find in high purity. Impurities in the crystal can result in occupied states in the band gap that induce photon absorption [2] and ultimately lead to the degradation of the material. In order to predict how well  $\text{CaF}_2$  will perform under irradiation of short wavelength laser light, one must understand the mechanisms for laser-induced damage.

Laser damage is often a two-step process: initial photons create new defects in the lattice and subsequent photons excite these defects. When laser light is absorbed by a solid surface there is an initial production of electron-hole ( $e-h$ ) pairs, a heating of free electrons and a generation of local heating around optically absorbing centers [3]. Once this initial excitation converts to the driving energy for nuclear motion, the result is an ejection of atoms, ions and molecules from the surface, known as desorption or ablation [3]. Secondary processes further driving desorption are photoabsorption, successive excitations of self-trapped excitons (STE's) and defects, and ionization of neutrals by incident laser light [3]. The combination of laser-induced desorption and the alterations to the electronic and geometrical structure of the lattice result in defect formation. In  $\text{CaF}_2$  some of these defects take the form of F-centers, an electron trapped at a halogen vacancy [4], and H-centers, a  $\text{F}_2^-$  molecular ion at a single lattice site [5]. While the F-cen-

ters are stable, the H-centers are mobile and can combine into aggregates that are stable. There are many different configurations the defects can take based on the relative position of F and H centers in the lattice and this is extensively discussed in the literature [1,4,5]. Once these defects form they may further absorb light which ultimately may induce particle emission and the production of even more defects.

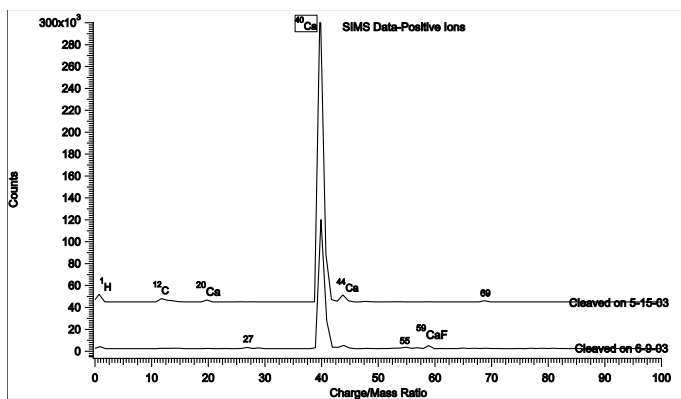
Various forms of laser-induced damage of  $\text{CaF}_2$  have been studied. For example, the mechanism for photon-stimulated desorption (PSD) of  $\text{F}^+$  from  $\text{CaF}_2$  (111) is discussed in ref. 6 and the energy threshold, distribution and kinetics governing electron-stimulated desorption (ESD) is investigated in ref. 7. Desorption of neutral Ca and F atoms has also been explored [1]. In this paper we focus on the emission of ions and neutrals from  $\text{CaF}_2$  under the irradiation of pulsed laser light at 266 nm. The large band gap of  $\text{CaF}_2$  greatly exceeds the photon energies of both 266 and 157 nm light such that the fundamental absorption processes of mid-gap defect states and multiphoton excitation processes of the intrinsic material are similar. A brief study of the crystal's purity and transmittance has also been performed.

## METHODS AND MATERIALS

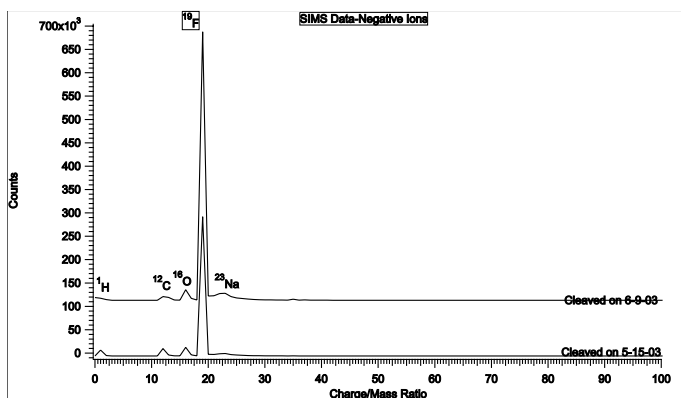
For all experiments synthetically grown  $\text{CaF}_2$  was obtained from International Crystal Labs. The purity of freshly cleaved samples (in air) of the  $\text{CaF}_2$  is checked using X-ray Photoelectron Spectroscopy (XPS) and Secondary Ion Mass Spectroscopy (SIMS).

For the experiments involving ion and neutral particle emission, samples of  $\text{CaF}_2$  are cleaved in air and mounted in an ultrahigh vacuum (UHV) at a base pressure of  $5 \times 10^{-10}$  torr. Two different laser systems are used in order to create transient defect centers in the sample, subsequently leading to particle emission. In the first experiment a sample is irradiated with femtosecond UV light pulses. Femtosecond UV pulses are produced in an amplified Ti:sapphire-based laser system operating at a wavelength of 798 nm. A Ti:sapphire oscillator (80 fs pulses at an 82 MHz repetition rate) is used to seed the Ti:sapphire amplifier pumped at 20 Hz by the frequency-doubled output of an Nd:Yttrium-aluminum-garnet (YAG) laser. The amplified output is frequency-doubled and then summed in KDP crystals, producing  $1.5 \text{ mJ pulse}^{-1}$  at 266nm [8]. A Wiley-McLaren time-of-flight mass spectrometer (TOF-MS) is used to extract and accelerate the positive ions. The power dependence of  $\text{Ca}^+$ ,  $\text{CaF}^+$  and  $\text{Ca}_2^+$  emission is determined by measuring the area in the corresponding ion peak as a function of laser fluence. The energy of the incident light is varied from low to high power and vice versa while recording the area.

A second experiment involved measuring the ion yield due to irradiation from nanosecond pulses. Nanosecond pulses are produced by a Nd:YAG flashlamp-pumped laser system operating at a 20 Hz repetition rate. The second harmonic output (532nm) is frequency-doubled, yielding pulse energies from 0.25 to 5.0  $\text{mJ pulse}^{-1}$  at 266nm [8]. Positive ions are again extracted using TOF-MS.



**Figure 2a:** SIMS measurements of the positive ions ejected from the surface of two samples of  $\text{CaF}_2$



**Figure 2b:** SIMS measurements of the negative ions ejected from the surface of two samples of  $\text{CaF}_2$

This same nanosecond system is used to induce neutral particle emission from the cleaved sample of  $\text{CaF}_2$ . In order to detect the neutrals a third laser is required. Tunable light from a Nd:YAG pumped frequency-doubled dye-laser, operating at 20 Hz, is used to ionize ground state  $\text{Ca}(4s^2 \ ^1S_0)$  atoms in a (2+2) resonance-enhanced multi-photon ionization scheme (probe pulse). The specific two-photon transition used is  $\text{Ca}(4s^2 \ ^1S_0) \rightarrow \text{Ca}(4s5s \ ^1S_0)$  at a wavelength of 600.15 nm. The focused probe-pulse intersects the desorbed atoms 3.8 mm above, and oriented parallel to, the sample surface [9]. Velocity profiles are taken of the Ca and K neutrals ejected from the sample. These are converted into kinetic energy profiles using a Jacobian matrix transformation.

In order to measure the transmittance of  $\text{CaF}_2$  a cleaved sample is mounted in air and irradiated with 266 nm femtosecond pulses. A 10 cm lens is used to focus the laser beam onto the crystal face. Measurements of the amount of light transmitted through and reflected by the crystal are taken at an incident spot size of roughly  $50 \mu\text{m}$ . To vary the pulse intensity, the lens is removed and the transmittance and reflectance measurement repeated. The spot size in this case is  $\sim 1 \text{ mm}$ .

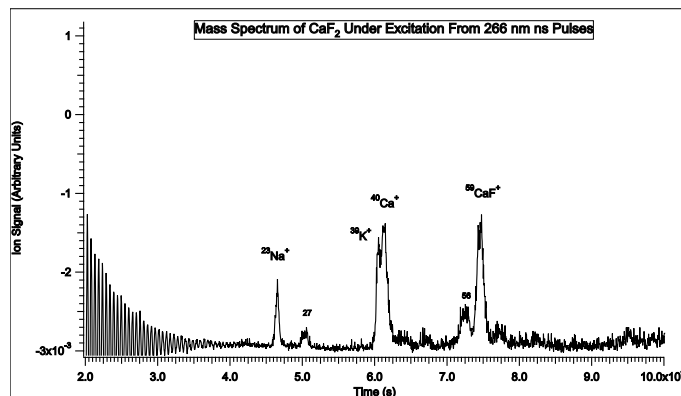
## RESULTS

Figure 1 shows the results from the XPS scan on the cleaved sample of  $\text{CaF}_2$ . The peaks have been labeled with their corresponding element and electron orbital. The larg-

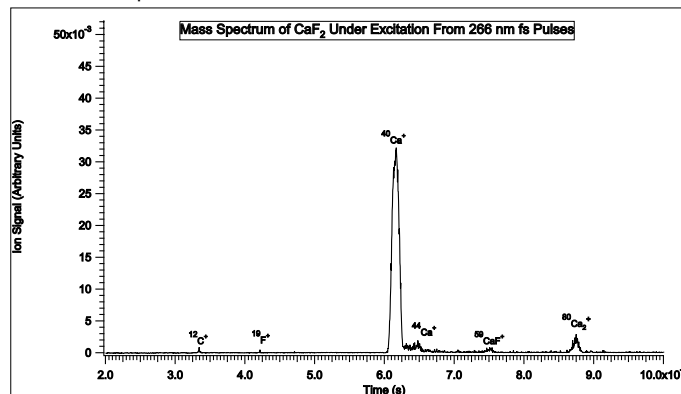
est peaks correspond to F(1s) and Ca(2p). Figures 2a and 2b show SIMS scans run on freshly cleaved samples of  $\text{CaF}_2$  in positive (a) and negative (b) ion mode. In both cases the dominant peaks correspond to  $\text{F}^-$  and  $\text{Ca}^+$  ions ejected from the surface. It is evident however that both samples contain H, C, O and Na surface contaminants.

Figure 3a is a TOF mass spectrum of  $\text{CaF}_2$  following 30 minutes of irradiation from ns 266 nm laser light. In this case the beam was focused down to a diameter of 2.8 mm and had an irradiance of  $6.5 \text{ MW/cm}^2$ . The large peak is a combination of  $^{40}\text{Ca}^+$  and  $^{39}\text{K}^+$ . Also visible are  $\text{CaF}^+$  ions, corresponding to charge/mass ratio of 59, whose peak is equivalent in size to that of  $\text{Ca}^+$ . The contaminant Na is also present in this mass spectrum. Pictured in Figure 3b is a TOF mass spectrum corresponding to fs 266nm laser radiation. The beam was focused down to a diameter of  $200 \mu\text{m}$  and had an irradiance of approximately  $31.8 \text{ TW/cm}^2$ . The most prominent peak is due to  $\text{Ca}^+$  ions, corresponding to a charge/mass ratio of 40. Calcium has an isotope at mass 44 which is also present in the spectrum. Other peaks of interest are  $\text{CaF}^+$ ,  $\text{Ca}_2^+$ , and a small peak due to  $\text{F}^+$  ions. A small yield of  $\text{C}^+$  makes up the only contaminant present in this mass spectrum.

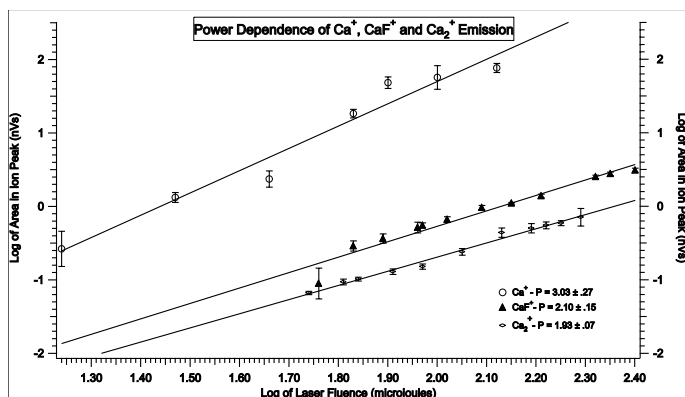
Tables 1, 2 and 3 show the results of the transmission and reflection measurements. Table 1 shows the percentage of light transmitted with the 10 cm lens in place. It was found that only 17%-20% of the light is transmitted. Table 2 shows that less than 1% of the focused light is reflected from the sample of



**Figure 3a:** TOF mass spectrum of  $\text{CaF}_2$  under irradiation from nanosecond laser pulses at 266 nm.



**Figure 3b:** TOF Mass Spectrum of  $\text{CaF}_2$  under irradiation from femtosecond laser pulses at 266 nm.



**Figure 4:** Power dependence measurements for  $\text{Ca}^+$ ,  $\text{CaF}^+$  and  $\text{Ca}_2^+$  emission from a cleaved sample of  $\text{CaF}_2$  under irradiation from femto-second laser pulses at 266 nm.

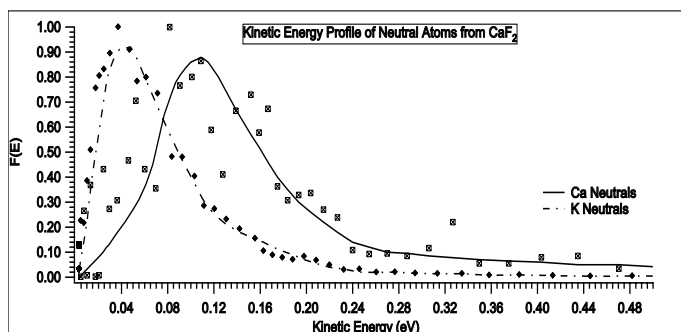
$\text{CaF}_2$ . Upon removal of the lens, the transmittance went up to ~80% (Table 3).

The dependence of ion yield on laser fluence from 266 nm fs pulses vs. ion yield for  $\text{Ca}^+$ ,  $\text{CaF}^+$  and  $\text{Ca}_2^+$  are plotted on a log-log scale in Figure 4. The slopes of these lines yield the power dependence for these particular sets. Nine data sets were taken for  $\text{Ca}^+$ , five for  $\text{Ca}_2^+$ , and four for  $\text{CaF}^+$ . Each data set was plotted in the same manner as Figure 4 and their power dependences determined. These dependences were averaged together to yield final values of  $2.9 \pm 0.9$  for  $\text{Ca}^+$ ,  $1.9 \pm 0.5$  for  $\text{Ca}_2^+$  and  $1.8 \pm 0.4$  for  $\text{CaF}^+$ .

Figure 5 is a kinetic energy profile of the Ca and K neutrals emitted from the cleaved sample of  $\text{CaF}_2$  following irradiation by ns 266 nm pulses. Both peaks appear to follow a Gaussian distribution. It is also evident that the peak energy of the Ca neutrals is centered at a higher kinetic energy than that of the K neutrals.

## DISCUSSION AND CONCLUSION

The results of the XPS (Figure 1) and SIMS (Figure 2) studies give useful information about the purity of the  $\text{CaF}_2$  samples. The XPS shows that there is oxygen and carbon present on the freshly cleaved sample. The height of these peaks indicates that at least  $\frac{1}{2}$  to one monolayer of these contaminants is present on the sample. The SIMS results indicate that both of the cleaved samples of  $\text{CaF}_2$  have significant surface



**Figure 5:** Kinetic energy profiles of Ca and K neutrals emitted from a cleaved sample of  $\text{CaF}_2$  under irradiation from nanosecond laser pulses at 266 nm. Lines are to guide the eye only.

contamination. The most dominant peak in the positive ions corresponds to  $\text{Ca}^+$ , with smaller peaks indicating the presence of H, C,  $\text{Ca}^{2+}$  and  $\text{CaF}^+$  (Figure 2a). There were also some unknowns at mass/charge of 27, 55, and 69. As for the negative ions,  $\text{F}^-$  is the dominant feature in both samples. The contaminants present are H, C, O and Na (Figure 2b). Attempts made

**Table 1:** Transmitted light through  $\text{CaF}_2$  crystal with 10cm lens in place

$I_0$ ( $\mu\text{J}$ )	$I_T$ ( $\mu\text{J}$ )	% Transmitted
125	10.2	8.2
205	9.6	4.7
31	6.5	21.0
63	10.7	17.0
89	15.1	17.0

**Table 2:** Reflected light from  $\text{CaF}_2$  crystal with 10 cm lens in place

$I_0$ ( $\mu\text{J}$ )	$I_T$ ( $\mu\text{J}$ )	% Reflected
46	0.1275	0.3
71.5	0.3075	0.4
100.5	0.642	0.4

**Table 3:** Transmitted light through  $\text{CaF}_2$  crystal without lens in place

$I_0$ ( $\mu\text{J}$ )	$I_T$ ( $\mu\text{J}$ )	% Transmitted
18.4	15.25	82.9
37.5	30	80
62	49.5	79.8

on both samples to ion drill into the surface (using SIMS) in order to determine the purity of the bulk were unsuccessful due to severe charging effects

The ns mass spectrum (Figure 3a) gives important insights into the mechanisms of laser damage and the effect purity has on laser interaction with the material. The most noticeable features in the ns mass spectrum are the peaks associated with  $\text{Ca}^+$  and  $\text{CaF}^+$ . These two peaks are virtually equivalent in size. In addition the contaminant yields are relatively high in comparison to the intrinsic peak of  $\text{Ca}^+$ . In fact,  $\text{Ca}^+$  is hardly distinguishable from the  $\text{K}^+$  peak. One factor contributing to the high ion yield of desorbed contaminants is the longer duration of ns pulses. The ns pulse that drives the initial interaction may still be present after the atoms have been ejected. Therefore the same pulse that induced the emission can then ionize the desorbed neutral atoms. This suggests that much of what is seen in the Figure 3a could possibly be the result of desorbed neutrals. The duration of the ns pulse also has an effect on the defects in the lattice. The ns pulses can actually serve to enhance the excitation of defects created from initial laser interaction with the crystal. It is important to try to pinpoint how the light interacts with  $\text{CaF}_2$ . If the laser pulses directly interact with the intrinsic material, a significantly higher yield of  $\text{Ca}^+$  desorbed from the material should result. Instead desorption of both the intrinsic atoms and contaminants is observed. This suggests that the light is not directly interacting with the pure material. Non-simultaneous single photons may be absorbed and it is their cumulative effect together that drive the desorp-

tion process. So although the pulses do not excite the pure material, through single photon absorption there is a definite excitement of both intrinsic atoms and impurities. As a result, the purity of the crystal is of extreme importance when using ns pulse irradiation.

The fs mass spectrum on the other hand yields different results. The most noticeable feature in Figure 3b is that the Ca<sup>+</sup> peak dwarfs all other peaks. In fact, the only other observed contaminant corresponds to Carbon. This indicates that there is a completely different mechanism for ion emission taking place. The fs pulses are now interacting with the pure material. There is no longer a cumulative effect of single photon absorption. Instead simultaneous multi-photon absorption is responsible for ion emission. In addition, it is unlikely that neutral ionization is occurring since the fs pulse is no longer present when the atoms are ejected from the lattice. From this it is possible to conclude that when laser intensity is high, as is the case with fs irradiation, the purity of the material may not matter as much because the light now strongly interacts directly with the material.

Examination of the transmittance and reflectance data from Tables 1, 2 and 3 provides further evidence of the non-linear absorption in CaF<sub>2</sub> under irradiation from 266 nm fs pulses. The first interesting result is that barely any light is transmitted when the laser beam is focused onto the crystal. At high incident energies around 100 to 200 μJ/pulse, a faint white plume can be seen, indicating that a high percentage of the incident light results in emitted particles. At intensities below 100 μJ/pulse, at which point a blue luminescence can be seen, only 17%-20% of the light is transmitted. Ideally, CaF<sub>2</sub> should be transparent to light at a wavelength of 266 nm. One possible explanation is that a large amount of light is actually being reflected from the surface. Measurements were made in order to determine if this was indeed the case. As seen in Table 2, the measured reflectance was less than 1%. This proved to be a difficult measurement to make and the actual value is most certainly higher than this. Fresnel's equations can be used to predict the light reflected when incident on a surface at angle θ<sub>i</sub> with a refracted angle of θ<sub>t</sub>.

$$R_{\text{perp}} = \frac{\sin^2(\theta_i - \theta_t)}{\sin^2(\theta_i + \theta_t)} \quad (\text{for perpendicular polarization})$$

$$R_{\text{parallel}} = \frac{\tan^2(\theta_i - \theta_t)}{\tan^2(\theta_i + \theta_t)} \quad (\text{for parallel polarization})$$

At small angles of incidence, the reflected light can be simplified to the following:

$$R_{\text{perp}} \approx R_{\text{parallel}} \approx \frac{(n-1)^2}{(n+1)^2}$$

where n is the index of refraction. For CaF<sub>2</sub> this value is 1.47 resulting in the reflection of only 3.6% of light incident at small angles. At an angle of incidence equal to 45°, this value rises to 8.5%. Based on our measurements and theoretical calculations, we are able to rule out reflection as the explanation for the small percentage of transmitted light. Another possible explanation for the low percentage of transmitted

light is that the crystal is actually absorbing the light. Due to the high intensity of the focused beam it is quite possible that a multi-photon absorption process is taking place. In order to check this theory, the lens was removed and the transmittance measurements repeated.

Table 3 reveals that ~80% of the light is transmitted at this lower intensity. Based on this we can once again conclude that when the incident intensity is high enough, a multi-photon absorption process is possible in CaF<sub>2</sub>. This is consistent with the observations from the fs TOF-MS. These results also serve to explain the low ion yields present in the ns mass spectrum. Even the unfocused fs beam could not drive significant absorption of light judging from the 80% that is transmitted. Therefore we cannot expect that the ns pulses, which are at a significantly lower level of irradiance, to be able to induce multi-photon absorption.

Power dependences of the ion emission following 266 nm fs irradiation are shown in figure 4. As mentioned in the results section, the average value of each of the power dependences were P<sub>Ca<sup>+</sup></sub> = 2.9 ± 0.9, P<sub>CaF<sup>+</sup></sub> = 1.8 ± 0.4 and P<sub>Ca<sup>2+</sup></sub> = 1.9 ± 0.5. In all three cases highly non-linear ion yield occurs following irradiation by ultrafast pulses. This further supports the arguments that multi-photon absorption is the mechanism for ion emission at high incident intensities.

When examining the kinetic energy profile of Ca and K neutrals emitted from CaF<sub>2</sub>, it is immediately evident that the Ca peak is centered at a higher kinetic energy than that of the K peak. Since the masses are virtually the same for K and Ca, this indicates that their velocities must be different. In fact, from the profile we can conclude that the Ca neutrals are ejected with ~3 times greater kinetic energy than the K neutrals. This indicates the mechanism for emission of the neutral K contaminant is different in comparison to that governing the emission of intrinsic Ca neutrals. Determining this mechanism is the focus of future research on CaF<sub>2</sub>.

The results of each of the experiments making up this research combine to form a general picture for laser-induced damage of CaF<sub>2</sub>. We have shown that impurity levels in the lattice have a strong effect on the crystals behavior under excitation from ns 266 nm laser pulses. However these impurities become less important when using fs irradiation. High intensity fs pulses interact with the pure material causing desorption of intrinsic atoms or the onset of laser damage. So although CaF<sub>2</sub> is the chosen material for the next generation of semiconductor lithography, it is still subject to damage if attention is not paid to the impurity level or the incident irradiation levels.

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1830). AE would like to thank the Department of Energy and the SULI program.

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