## Molecular Ion Desorption from LiF(110) Surfaces by Positron Annihilation

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We have studied the desorption of positive ions from a LiF(110) crystal surface using positron and electron irradiation at 500 eV to examine the interaction between positrons and ionic crystals. Only monatomic ions, such as  $H^+$ ,  $Li^+$ , and  $F^+$ , are detected under electron irradiation. However, positron irradiation leads to the significant desorption of ionic molecules, specifically,  $FH^+$  and  $F_2^+$ . Molecular ion yields are more sensitive to temperature than atomic ion yields. Based on the findings, we propose a desorption model in which positronic compounds are initially produced at the surface and subsequently desorbed as molecular ions via Auger decay following positron annihilation.

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Positrons have been employed in various experiments to investigate their interactions with matter, thereby providing valuable insights into the fields of atomic physics [1], material engineering [2–4], medical diagnostics [5], and astrophysics [6,7]. The knowledge gained from these studies has also led to the development of useful positron probes for determining the physical and chemical properties of thin films, bulk solids, and crystal surfaces [8–10].

Further intriguing investigations into the interactions between positrons and matter concern the bound states of positrons with ordinary species prior to their annihilation. Although the positronium (Ps), a composite system of an electron and a positron, is well known, theoretical predictions suggest the existence of positron-bound states with various atoms, molecules, and ions [11–18]. Experimentally, positron attachment to molecules has been studied by vibrational Feshbach resonance [12,19]. Moreover, these positronic compounds, which are typically discussed as isolated systems, can be produced in condensed matter. These stability and binding properties provide unique perspectives on the interaction of antiparticles with ordinary substances, paving the way for novel investigations in the field of quantum chemistry. Furthermore, understanding the behavior of positronic compounds in solids will provide a more accurate interpretation of the information obtained from positron probes, which are highly sensitive to the electronic and geometric structures of the impacted material. However, the experimental production of these compounds has proven challenging. Thus, establishing techniques to experimentally elucidate the positron-matter interactions involved in the formation of positronic compounds is essential to further development in this field.

When a low-energy beam of positrons impinges on the surface of a crystal, positively charged ions can desorb into a vacuum [20–22]. This positron-stimulated desorption  $(e^+SD)$  results from the interaction between positrons

and the atoms, molecules, and ions that comprise the crystal surface. Hence, the study of the desorption process can provide valuable information on the behavior of compounds that have interacted with positrons. In this Letter, we describe the  $e^+$ SD from the surface of an ionic LiF(110) crystal, which comprises Li<sup>+</sup> cations and F<sup>-</sup> anions in a cubic NaCl-type structure. This solid sample serves as a suitable medium for gaining a deeper understanding of the interactions between ionic atoms and positrons. Our results demonstrate for the first time that molecular ion desorption occurs via positron bombardment, whereas only atomic ion desorption occurs via electron-stimulated desorption (ESD). We propose that the interaction between positrons and the ionic lattice results in the formation of positronic compounds with lattice anions, which are subsequently desorbed as positively charged ions via positron-annihilation-induced Auger decay.

Our experiments were conducted in an ultrahigh vacuum (UHV) chamber with a base pressure below  $1.0 \times 10^{-8}$  Pa. A schematic diagram of the measurement system is shown in Fig. 1. The LiF sample was cleaved in air and immediately mounted on a sample holder in the chamber, where it was repeatedly heated to approximately 300 °C

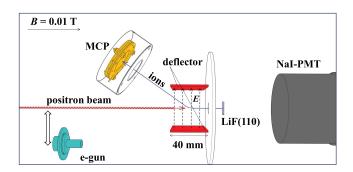


FIG. 1. Schematic diagram of the measurement system.

using a heater built into the sample holder for surface cleaning. Moderated positrons from a <sup>22</sup>Na source were accelerated to 800 eV and guided toward the sample using an axial magnetic field of 0.01 T. Before entering the sample, the beam trajectory was slightly deflected by an ion deflector comprising parallel plates with a potential difference of 460 V. The intensity of the positron beam at the sample location was approximately  $1 \times 10^4$  s<sup>-1</sup>. A NaI(Tl) scintillator combined with a photomultiplier tube was placed near the sample to monitor 511 keV annihilation  $\gamma$  rays. Positive ions desorbed from the sample surface following positron beam irradiation were detected using a time-of-flight (TOF) technique. A voltage of 300 V was applied to the sample holder to accelerate the ions. Thus, the positrons were decelerated to 500 eV when they entered the sample. The accelerated ions were deflected by the electric field created by the ion deflector and directed to the chevron microchannel plate (MCP), which was placed off the axis normal to the sample surface and to which -2.0 kVwas applied at the front. Typically, the ions will undergo a slight cyclotron motion because of the axial magnetic field, but the effect is negligible. This fact was confirmed by ion trajectory simulation using SIMION [23]. Conversely, scattered and reemitted positrons that are accelerated from the sample surface become thinly wrapped around the magnetic field, because they have a much smaller mass than ions. These positrons do not move toward the MCP even if an electric field is present within the ion deflector. Parenthetically, detecting secondary or Auger electrons is impossible in this configuration for the same reason, even when the bias polarity of the electrodes is reversed. The TOF spectra of  $e^+$ SD ions were obtained by analyzing the time difference between the  $\gamma$ -ray signals from the photomultiplier and the ion signals from the MCP detector. For ESD measurements, the sample surface was irradiated with a pulsed electron beam from an electron gun mounted on a manipulator for controlled movement within and outside the beam path. The energy of the electron beam from the *e*gun was set to 200 eV with a duration of approximately 20 ns at a repetition rate of 1 kHz. The electrons were accelerated by the sample bias of 300 V and attained the same incident energy as that of the positron beam (500 eV). ESD TOF spectra were obtained by capturing the ESD ion signals from the MCP detector using a start signal synchronized with the pulse frequency output from the *e*-gun controller.

Figure 2 shows (a) ESD and (b)  $e^+$ SD TOF spectra of positive ions desorbed from the LiF(110) surface. The black curves represent the data obtained at room temperature; the red and blue curves were acquired at 300 °C. The higher temperature enhances ionic mobility, which reduces sample charging and facilitates surface reconstruction. The MCP detector also responds to the annihilation  $\gamma$  rays emitted from the sample, resulting in a peak appearing only in the  $e^+$ SD spectrum at 0 µs. Remarkably, even

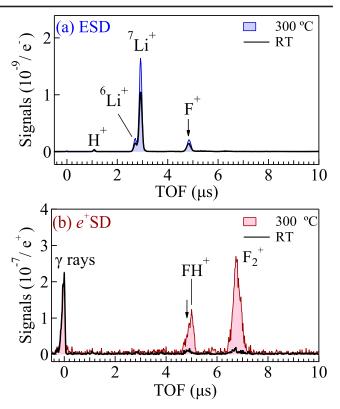


FIG. 2. TOF spectra of ions desorbed from a LiF(110) surface by (a) electron and (b) positron bombardment at an incident energy of 500 eV. The red and blue curves were acquired at a sample temperature of 300 °C. The black curves were obtained after cooling to room temperature. The arrows indicate the expected peak position of  $F^+$  ions.

disregarding this difference owing to its antiparticle nature, the  $e^+SD$  TOF distribution differs significantly from that of ESD. This finding indicates that the ion species of  $e^+SD$  do not match those of ESD despite irradiation with beams of the same incident energy.

The primary desorbing species identified in the ESD TOF spectrum [Fig. 2(a)] are the monatomic  $H^+$ ,  ${}^{6}Li^+$ ,  ${}^{7}Li^+$ , and  $F^+$  ions. These findings are consistent with previous studies on ESD and photon-stimulated desorption (PSD) from a well-prepared clean LiF surface [24-29]. Parks, Shirley, and Loubriel [24] and Yasue et al. [25] employed monochromatic synchrotron radiation to measure the incident energy dependence of PSD yields. These authors observed that the yields of F<sup>+</sup> and Li<sup>+</sup> ions increased rapidly at photon energies corresponding to the  $Li^+(1s)$ core excitation energy. Based on their results, the desorption process of the atomic ions was interpreted as follows. Initially, a lithium 1s hole is filled with a valence electron from a neighboring  $F^-$  anion. Then, the  $F^-$  anion is converted to a  $F^+$  cation via Auger electron emission by interatomic Auger decay. Finally, either F<sup>+</sup> or Li<sup>+</sup> is desorbed into the vacuum by Coulomb repulsion between F<sup>+</sup> and its surrounding Li<sup>+</sup> cations. Another threshold for  $F^+$  ion desorption was reported at the energy corresponding

to the  $F^-(2s)$  inner-shell level [24–26]. In this case,  $F^+$  is believed to form from lattice  $F^-$  anions via *intra-atomic* Auger decay. The driving force behind  $F^+$  desorption is still interpreted as the Coulomb repulsion between cations, but no rapid increase in the yield of Li<sup>+</sup> ions is noted around the  $F^-(2s)$  threshold.

In the  $e^+$ SD TOF spectrum at 300 °C [Fig. 2(b)], the peak intensity of the desorbed Li<sup>+</sup> ions is much less than that of the background signal, although Li<sup>+</sup> has the highest ESD yield. Instead, two large peaks appear, which are assigned to the  $FH^+$  and  $F_2^+$  ions. These ions maintain elevated yields for a duration of at least one day. Previous investigations provide evidence that collisions between positrons and molecules in the gas phase generate fragment ions through ionization processes that include Ps formation, annihilation, and impact ionization [30-34]. However, the  $FH^+$  and  $F_2^+$  ions observed in the present Letter are not dissociated from large molecules or clusters adsorbed on the crystal surface. Otherwise, the absence of these ions in the ESD spectrum cannot be explained. For the same reason, we postulate that the detected  $e^+SD$  ions do not contain multiply charged molecules. Even doubly charged ions of diatomic molecules are unstable due to Coulomb repulsion, which results in the generation of fragment ions [35]. The broadening on the left side of the  $FH^+$  peak is likely due to the desorption of F<sup>+</sup> ions. In the desorption model via interatomic Auger decay accepted in ESD and PSD studies [24,25], desorption of Li<sup>+</sup> ions should be observed simultaneously. Therefore, the absence of a Li<sup>+</sup> signal in Fig. 2(b) suggests that the  $e^+SD$  of  $F^+$  ions is primarily due to intra-atomic Auger decay following corehole formation in the inner-shell orbital of the F<sup>-</sup> anions. We also should consider the possible presence of an additional peak superimposed within the  $F_2^+$  response. However, the detection of this species is impossible given the resolution limitations of our experimental setup.

The distinction between ESD and  $e^+$ SD processes from the LiF surface is attributed to ionization resulting from the pair annihilation of  $e^+$ SD. Like ion desorption from the LiF sample, the ESD and PSD of O<sup>+</sup> ions from the TiO<sub>2</sub> surface are attributed to the decay of core-hole states of the surface atoms created by electron bombardment and photon absorption, respectively [36,37]. Conversely,  $e^+SD$  of O<sup>+</sup> ions occurs mainly via the creation of holes owing to the pair annihilation of positrons with core electrons [22]. Furthermore, the behavior of positrons before their annihilation in the TiO<sub>2</sub> crystal is strongly reflected in the desorption phenomena, resulting in features different from those of ESD and PSD. For instance, the  $e^+$ SD yield of O<sup>+</sup> ions is one order of magnitude larger than the ESD yield, whereas the yields of other  $e^+SD$  species are comparable to their ESD yields.

In our previous study, we found that the selectivity of  $e^+$ SD ions for the TiO<sub>2</sub> surface is due to positron trapping at the surface and their annihilation sites [38]. Upon

impacting the crystals, low-energy positrons promptly decelerate through inelastic collisions to attain thermal equilibrium with some individuals diffusing back to the surface [8]. In many metals, diffusing positrons that reach the surface can be trapped in potential wells and eventually are annihilated by electrons present in the topmost layers. Although TiO<sub>2</sub> crystals are transition-metal oxides with relatively wide band gaps, theoretical evidence suggests that positrons can be trapped in surface potential wells [38]. ESD and PSD occur because of primary beam interactions at the surface layers. Once the incident electrons and photons penetrate the bulk, they are rarely involved in desorption. Conversely, in  $e^+SD$ , positrons that enter the solid interior can contribute to ion desorption after diffusing back to the surface. Annihilation of these positrons is more effective in removing inner-shell electrons from the surface layer than electron impact in ESD and photon absorption in PSD [20]. In addition, theoretical predictions suggest that positrons trapped on the surface tend to be attracted to surface oxygen sites in TiO<sub>2</sub> crystals, which comprise O<sup>2-</sup> anions and Ti<sup>4+</sup> cations [38]. Thus, the  $e^+$ SD of O<sup>+</sup> ions exhibits higher yields than the ESD yields of O<sup>+</sup> and other  $e^+$ SD ions.

In the present Letter, ESD was performed with an incident electron energy of 500 eV, which exceeds the ESD thresholds of F<sup>+</sup> and Li<sup>+</sup> ions, thus confirming the generation of both ions. However, surface-trapped positrons exhibit annihilation-site selectivity on the LiF crystal and preferentially annihilate close to F<sup>-</sup> anions rather than  $Li^+$  cations. This preference leads to the desorption of  $F^+$ ions through intra-atomic Auger decay and suppresses the desorption of Li<sup>+</sup> ions. Consequently, F<sup>+</sup> desorption, but not Li<sup>+</sup> desorption, is observed in  $e^+$ SD.  $e^+$ SD also involves desorption resulting from positron-impact ionization on the surface, similar to ESD. However, this process is less efficient than positron-annihilation-induced desorption, as mentioned previously. Furthermore, because the intensity of the positron beam is much less than that of the electron beam, assessing the contribution of positron impact to ion desorption is difficult.

The site selectivity of positrons on the LiF surface may be related to the desorption of  $F^+$  ions and formation of molecular ions. Each  $F^-$  anion in the LiF crystal is surrounded by six Li<sup>+</sup> cations. We assume that the presence of  $F_2^+$  ions in  $e^+SD$  induces lattice distortions. Creating valence holes and conduction electrons through electron impact and photon adsorption is known to cause significant changes in the charge distribution within highly ionic alkali halide crystals [39]. Excess holes (electrons) repel (attract) near alkali metal cations and attract (repel) halide anions. This distortion follows the carriers as they move through the crystal, thereby increasing their effective mass. Although the electrons are free to move, the holes in the conduction band become self-trapped because of lattice distortion. Local lattice distortion shortens the interhalogen

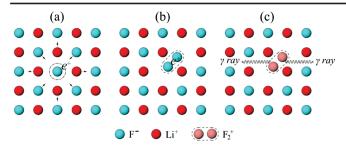


FIG. 3. Schematic diagram of the  $e^+$ SD process of molecular ions. (a) Initially, a positron is localized around an F<sup>-</sup> anion, which attracts (repels) the nearby halide anion (alkali metal cation). (b) Subsequently, a negatively charged positronic compound is produced owing to local lattice distortions. (c) Finally, a positively charged molecular ion is generated and expelled into the vacuum through Coulomb repulsion with the surrounding cations via a charge-exchange process that involves positronannihilation-induced Auger decay.

distance, causing the hole to be shared between two adjacent halogen species, essentially forming a dimer ion  $(X_2^-)$ , where X is a halogen atom). Electron capture by  $X_2^$ immediately forms a self-trapped exciton  $(X_2^- + e^-)$ , which then decomposes into colored centers, i.e., an H center (interlattice  $X_2^-$ ) and an F center (electron captured in a halogen vacancy). The H center is a highly mobile defect that can diffuse to the surface, leading to the emission of neutral halogen atoms. We propose that the formation of molecular ions at the LiF crystal surface in  $e^+$ SD arises from local lattice distortions associated with the localization and self-trapped states of the diffusing positrons.

Figure 3 shows a schematic of the process proposed for the  $e^+$ SD of molecular ions from a LiF crystal. As with other carriers, positrons in LiF crystals are postulated to undergo strong interactions with the ionic lattice. Our results indicate that the positrons trapped on the LiF crystal surface tend to accumulate at the site of F- anions [Fig. 3(a)]. In addition to the desorption of  $F^+$  ions, the site selectivity of positrons may result in local lattice distortions. Recent theoretical calculations predict that two halide anions and one positron can combine to form  $e^{+}[X^{-}X^{-}]$ , where the positron density is concentrated in the internuclear region [16]. Such positronic compounds may also form in LiF crystals. Specifically, as illustrated in Fig. 3(b), the positron can attract another anion while repelling Li<sup>+</sup> cations to form  $e^{+}[F^{-}F^{-}]$ . This process appears to be the initial stage of molecular ion desorption, which cannot be caused by positron impact.

If desorption of  $F_2^+$  ions occurs via the formation of  $e^+[F^-F^-]$  compounds, the transfer of at least three electrons is required. This charge exchange may be facilitated by the emission of two electrons, specifically, Auger electrons from positron-annihilation-induced Auger decay [40].  $F_2^+$  cations formed at the surface may be ejected into the vacuum owing to Coulomb repulsion with the surrounding

Li<sup>+</sup> cations. However, because double Auger decay is less frequent than single Auger decay, an additional chargetransfer process preceding the Auger transition must also be considered. For example, as is the case with self-trapped excitons,  $e^+[F^-F^-]$  might decompose into  $e^+[F^-F^0]$  and H centers. In this scenario,  $F_2^+$  cations are formed by single Auger decay in the  $e^+[F^-F^0]$  compound. The surface adsorption of molecular hydrogen, which is a prominent residual gas in the UHV chamber, is believed to be implicated in the desorption of FH<sup>+</sup> ions. Instead, H<sup>-</sup> anions dissociated from hydrogen molecules fill the vacancies of the F<sup>-</sup> anions [24]. Thus, heteronuclear  $e^+[H^-F^-]$ compounds [16] may subsequently form as a precursor to desorbed FH<sup>+</sup> ions.

A significant difference is observed in the temperature dependence of the  $e^+$ SD and ESD desorption yields. The black curves in Fig. 2 represent the TOF spectra acquired for ESD and  $e^+$ SD following cooling to room temperature. Although both measurements exhibit lower desorption yields at room temperature than at 300 °C, the contrast is more evident for  $e^+$ SD. Given the significantly greater intensity of the electron beam compared with that of the positron beam, the difference is unlikely due to charge-up effects at low temperatures, because ESD should be more susceptible to these effects. Another factor to consider for the decrease in the  $e^+SD$  yield at room temperature is contamination of the sample surface through the gradual adsorption of residual gases. However, the yield drops rapidly upon reduction of the sample temperature. In addition, ESD measurements conducted under similar circumstances [Fig. 2(a)] exhibit no significant increase in signals attributable to surface contamination. Reheating the sample produces  $e^+SD$  yields equivalent to those observed prior to the cooling process. Consequently, temperature-dependent lattice mobility or vibrations of the LiF crystal may contribute to the formation of positronic compounds. The observed difference in temperature dependence also suggests that the  $e^+SD$  process of molecular ion generation differs discernibly from the ESD-like impact process, which is consistent with our interpretation.

In conclusion, a novel aspect of the interaction between matter and positrons in the form of creation and desorption of molecular ions on the surface of LiF crystals is described. To explain these findings, we propose a desorption model, which posits that positronic compounds are generated on the surface and subsequently desorb as molecular ions via Auger decay following positron annihilation. This model will be substantiated through desorbed ion– $\gamma$ -ray coincidence measurements employing a HPGe detector [41] and the analysis of Auger electrons [40]. These measurements should be conducted using a positron beam with energies below the ion desorption thresholds, because this condition allows exclusive observation of phenomena originating from annihilation. The  $e^+$ SD method holds significant promise as an innovative tool for investigating positron properties and for providing comprehensive explanations of intricate processes in condensed matter. Our studies on positron-annihilation-induced desorption have shown the possibility of applying the positron annihilation method, which has been used only for the nondestructive measurements of solids, to the modification of surface properties by altering surface composition and structure. More importantly, this Letter demonstrates the possibility of producing molecular ions comprising several atoms via slow positron injection. The  $e^+$ SD technique may pave the way for future generation of novel molecular ions that cannot be realized by other methods.

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