Desorption kinetics of Li-atom delayed emission from LiF under low-energy-electron bombardment

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Electron-stimulated desorption of lithium atoms from LiF crystals has been investigated with quadrupole mass spectroscopy at temperatures in the range 350 to 440 °C. Following the cessation of electron bombardment, a delayed maximum in the emission of lithium atoms is observed. In a previous publication, this surprising result was explained in terms of a homogeneous nucleation of large clusters (colloids). The diffusion of F centers was neglected, because the diffusion time was considered to be much shorter than the lifetime of the colloids. In this paper we report on studies that use undoped LiF and LiF doped with divalent impurities in order to test the homogeneous-nucleation model and to investigate the details of the behavior of the cluster centers responsible for the occurrence of the delayed maximum. Our results suggest that the formation of small F-center agglomerates (not large Li colloids) during bombardment along with F-center diffusion are the important factors which account for the delayed emission of Li atoms. Based on our results, we suggest a modified model that incorporates diffusion, and a heterogeneous-nucleation picture in which small F-center clusters with widely varying thermal stabilities form and decay at impurity sites.

INTRODUCTION

It is well established that ionizing radiation produces F and H centers in alkali halides.[1,2] At temperatures high enough for migration (typically above 300 K in the case of F centers and above 60 K for the H centers), these centers form clusters.[3-6] F-center clusters with sizes ranging from two F centers to many hundreds (colloids) have been investigated by means of optical absorption spectroscopy, Raman spectroscopy, ESR, electron microscopy, and x-ray diffraction.[4,6-9] In most cases the defects were generated far in the bulk of the investigated crystals (by x rays, γ rays, or MeV electrons) such that surface influences were neglected. The best characterized F-center clusters are the small clusters involving only a few F centers. In rather pure crystals (less than 1 ppm impurities) these small F-center clusters, which are not associated with impurities, are *homogeneously* distributed in the crystal [6], whereas in doped or deformed crystals they seem preferentially to occupy lattice sites near impurities or dislocation loops and are therefore said to be *heterogeneously* distributed.[4,10-13]

For high irradiation doses (~ 100 Mrad) metallic particles (colloids) with radii up to several hundred nm are formed.[6,14,15] A striking difference between the metallic colloids and the small F-center clusters is the fact that the former are *always* distributed *heterogeneously* in the crystal.[6] Electron microscopy studies show that they are mainly formed at or near dislocations, or charge compensating defects.[4,6] One suspects that the reason for this fact is that one or more of the small F-center clusters is

not sufficiently stable.[6] Such unstable clusters inhibit a further nucleation of F centers to these small and homogeneously distributed F-center clusters. Near dislocations these intermediate clusters are thought to be stabilized such that they can agglomerate a large amount of F centers. The very early stages of growth of colloids are not well understood.

It is well established that F centers formed under ionizing radiation neutralize Li ions on the surface, after they have diffused to the surface. If the temperature is high enough, the Li atoms desorb from the surface thermally.[16]

Whereas defect formation in alkali halides caused by ion, and neutron impact, or x-ray irradiation had been studied excessively over the past twenty to thirty years,[6] low-energy electron bombardment measurements have only been performed over the past few years.[16-18] For low-energy measurements, the penetration depth of electrons is quite small, and the defects and defect clusters are therefore confined to a volume consisting of only a few monolayers under the surface. The near-surface volume is rather difficult to study since conventional methods for the study of F centers and F-center agglomerates such as NMR, or optical absorption spectroscopy, primarily are bulk-sensitive tools. It is possible, however, to study desorption kinetics of alkali atoms and draw conclusions about what is happening on or under the surface of the crystals during and after electron bombardment.[16-18] Previously, Betz and co-workers[17,18] have bombarded LiF crystals with low-energy electrons at about 400 °C. They monitored the yield of ground-state

Li desorption from the surface of the crystal by means of quadrupole mass spectroscopy. Under certain experimental circumstances an unexpected delayed maximum in the rate of desorbing Li atoms from the surface was observed after the electron gun had been turned off. The authors interpreted this surprising result to be due to the disintegration of *F*-center clusters, which were formed during the electron bombardment. The model used to explain this effect was a homogeneous nucleation picture, which mathematically described the phenomena by a series of rate equations, where they assumed that (a) effects due to diffusion could be neglected (i.e., diffusion time is much shorter than lifetime of clusters) and (b) larger centers are thermally more stable than smaller ones (based on the results of the Thompson equation).[19] The authors did not specify if the observed behavior is due to the disintegration of very large clusters (colloids) or due to small *F*-center clusters. In this paper we report on additional experiments, which utilize LiF crystals with known impurity concentrations, in order to test the homogeneous nucleation model and to investigate the nature of the centers which are responsible for the occurrence of the delayed maximum. Our results indicate that (a) the principal contribution to the delayed maximum phenomena is from small stable *F*-center clusters rather than from larger colloids as suggested by the Thompson equation and (b) it is absolutely essential that the effects of defect migration and nucleation at impurity sites should be taken into account. Consequently, the previous model should be modified to include these findings.

EXPERIMENTAL SETUP

In our experiment, LiF⟨100⟩ surfaces were bombarded with electrons primarily at 300 eV at a range of target temperatures (350–440 °C). By changing the filament current the spot size remained invariant. Under these conditions, the electron current was varied between 10 and 100 μA. The corresponding current density varied between 1 and 10 μA/mm^2. The angle of incidence of the electron beam was 45° with respect to the target normal. Ground-state Li atoms were detected with a quadrupole mass analyzer following electron beam post ionization. The UHV system maintained a base pressure of less than 10^{-9} Torr (1.3×10^{-7} Pa). For our experiments we used Harshaw LiF crystals (with unknown impurity concentration), and LiF crystals containing 10^{-3} mol % MgF$_2$, supplied by F. Luty at the University of Utah. The crystals have been cleaved in air and cleaned in the vacuum system by prolonged heating at 400 °C. Temperatures were measured with a thermocouple, which was thermally connected to the surface of the crystal and to the target holder. The desorbing Li atoms were detected by a quadrupole mass analyzer viewing the LiF surface along the normal.

RESULTS

After the electron gun is turned off at temperatures in the range above 250 °C, one can distinguish a prompt de-

cay as displayed in Fig. 1, which is faster than the detector time resolution of 70 ms, and a delayed decay, whose time constant (in the range of seconds to minutes) is found to depend on several parameters, such as temperature, dose rate, and integrated dose. Under certain conditions, within the temperature range 380–440 °C, the signal following the prompt decay is observed to increase again long (seconds) after the electron gun is turned off. This is referred to as a "delayed maximum." The integral over the delayed emission (all emission following the turn off of the electron beam, which we define as the "delayed yield") we take as a measure of the number of *F*-center defects stored in the crystal during bombardment whether they contribute to the delayed maximum or not.

For electrons over a wide range of energies and current densities, longer irradiation times result in a shift in the position of the delayed maximum toward longer times. The delayed maximum disappears completely for irradiation times on the order of a few seconds, as shown in Fig. 2, for the case of irradiation with 60-eV electrons.

As shown in Fig. 3 for 300-eV electrons, at 400 °C after an irradiation time of about 40 s the delayed yield approaches saturation. It is very interesting to note that the time required to reach saturation for a given temperature is found to be independent of the current density and the concentration of impurities in the crystals. The delayed yield in the case of the doped crystals is about two to three times larger than in the case of the relatively pure Harshaw LiF crystals. For higher temperatures the lithium delayed yield at saturation is lower, and the time to reach saturation is smaller as shown in the inset in Fig. 3 (the inset has the same arbitrary units).

Figure 4 shows the Li desorption rate as a function of time during and after the electron bombardment for two different absolute currents into the same current spot size which corresponds to two different current densities (8 and 4 μA/mm^2). One clearly sees that the shape of the delayed decay depends only on the bombarding time and
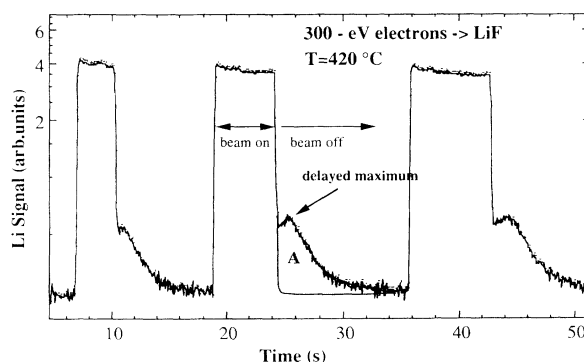

FIG. 1. The emission of ground-state Li atoms from LiF crystals containing 10^{-3} mol % MgF$_2$ under pulsed electron irradiation is shown in a logarithmic plot. After the electron bombardment is stopped, the Li signal exhibits a prompt decrease followed by a delayed emission in the order of seconds (area *A*). The position of the delayed maximum shifts for longer irradiation times.
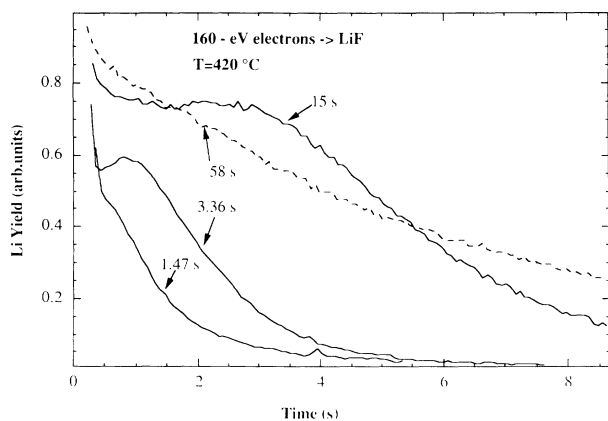
FIG. 2. The emission of ground-state Li atoms from LiF crystals containing 10^{-3} mol % MgF_2 as a function of time after the electron gun has been turned off is shown in a linear plot. The crystal was bombarded with 160-eV electrons, with a current density of 5 $\mu A/mm^2$ at 430 °C. The bombardment time was varied between 1.5 and 60 s.

FIG. 4. The desorption rate of ground-state Li atoms as a function of time is plotted for two different currents and irradiation times (the values are noted directly on each data plot) on a semilogarithmic plot. One clearly sees that the shape of the decay does not change if the current is changed, but depends strongly on the irradiation time.

not on the dose rate. In agreement with the data shown in Fig. 2, the delayed maximum becomes less pronounced and appears later in time for longer electron irradiation times.

The differences in lithium emission between undoped and doped LiF crystals can be seen in Fig. 5. For a given electron current, the Li desorption rate and total yield *during irradiation* are found to be markedly higher in the case of the undoped crystal as compared to the doped crystal. In striking contrast the delayed yield is higher and the delayed maximum is much more pronounced in the case of the doped crystals. Further experiments performed at lower temperatures (20–30 °C lower) show that delayed maxima also appear in the case of undoped crystals, but the delayed maxima for the doped case was found to be even more prominent than in the undoped case. At all temperatures studied, an increase in the total

current and current density did not change significantly the shape of the decay curve.

Over the entire electron current range used in these experiments, the delayed yield, which we assume reflects the stored amount of F centers, is proportional to the total current and since the spot size remained the same, also to the current density in the case of doped crystals for the same irradiation time of 3 s, as shown in Fig. 6.

We have numerically integrated the diffusion equation (Fig. 7) for F-center diffusion under the assumptions that their are no sinks for F centers, and that the density of F centers at the surface equals zero.[25,26] The latter assumption is reasonable for high temperatures, since the formation and the evaporation of neutral alkali atoms on the surface is very fast at temperatures as high as 400 °C, and therefore cannot be the rate limiting step. The former as-
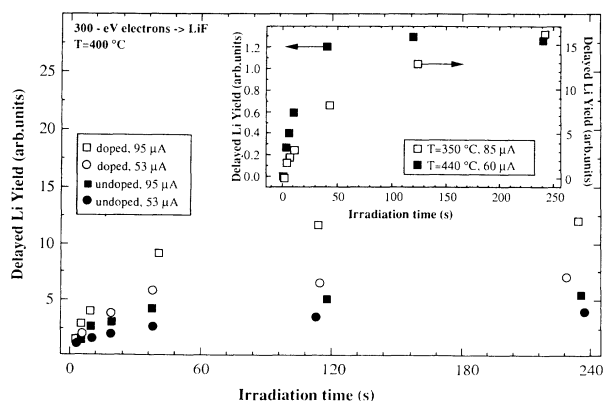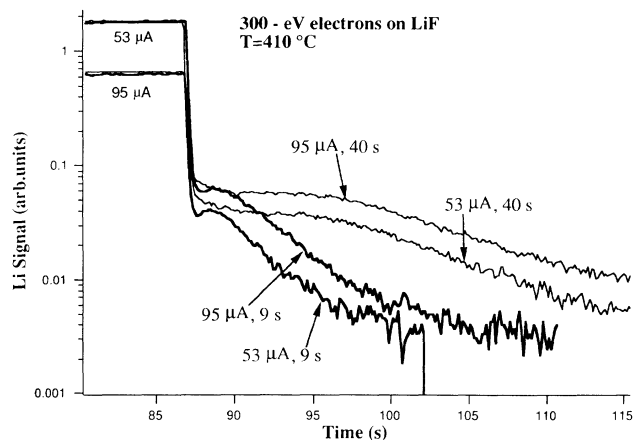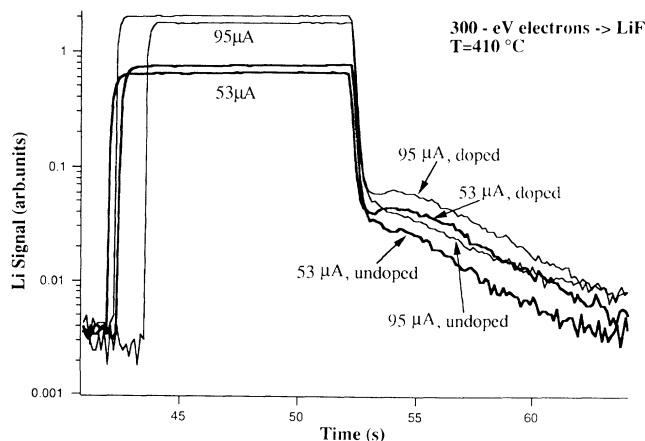

FIG. 3. Experimental data of the delayed yield of Li atoms as function of irradiation time are presented for different currents for undoped and doped LiF crystals. One clearly sees that the saturation level depends on the doping level and the total current of the electron beam. The inset shows that saturation is reached later for lower temperatures.

FIG. 5. The delayed maximum ("delayed maximum") is clearly much more pronounced in the case of the with 10^{-3} mol % MgF_2 doped crystals, whereas the desorption rate during bombardment is smaller in the case of the doped crystals. The irradiation time was in each case around 10 s.
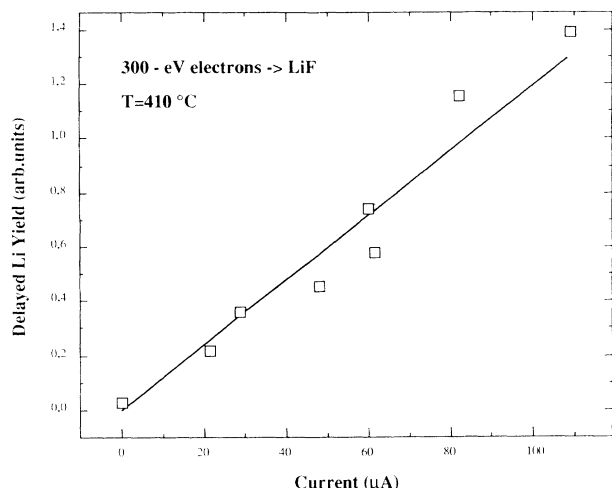
FIG. 6. The amount of stored defects during irradiation (chosen irradiation time was 3.0 s) increases more or less linearly in current in the case of irradiation of doped crystals at 410 °C.

sumption that there are no sinks for F centers is doubtful since F-center clusters are formed under our experimental conditions.[20] However, the purpose of our calculations is to show that the F-center density is quite high even far in the bulk with respect to the penetration depth of 300-eV electrons [~ 100 Å, (Ref. 19)]. Consequently,
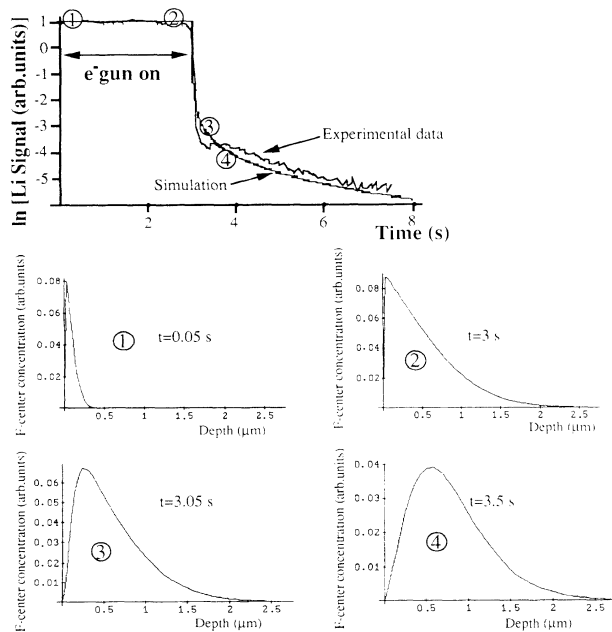

FIG. 7. The desorption rate of Li atoms from LiF during and after 300-eV electron bombardment, and the distribution of F centers in the crystal for certain times (circles 1, 2, 3, and 4) have been calculated under the assumptions explained in the text. One clearly sees that the F-center diffusion tends to smooth out concentration differences along the diffusion depth. The simulation fits the experimental data, except the delayed maximum (which the authors explain due to disintegration of F-center clusters).
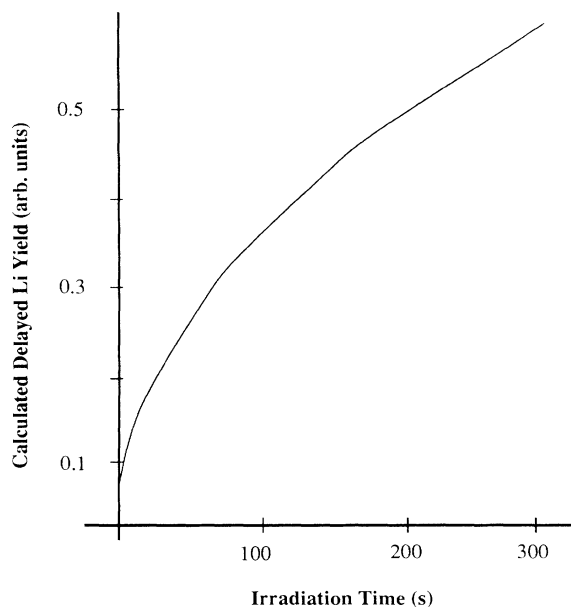

FIG. 8. The delayed yield has been calculated as a function of the bombarding time under the assumption that diffusion is the dominant process. The delayed yield increases monotonically with dose.

F-center agglomerates are formed much deeper in the crystal than the penetration depth, and the time for the F centers after disintegration of the agglomerates to reach the surface must therefore be considered. Figure 7 shows that under our assumption the F-center concentration at 800 Å is half of the maximum at 60 Å for an irradiation time of about 3 s. For this calculation we used diffusion constants derived by Hughes and Lidiard from experimental fits for F centers in NaCl.[14]

The calculated delayed yield increases monotonically as a function of the irradiation time (Fig. 8). It is important to note that recombination processes and clustering processes have been neglected in these calculations. Consideration of these processes would lead to a saturation of the delayed yield for large irradiation times.

DISCUSSION

The previous homogeneous nucleation model expressed as a series of rate equations[17,18] governing F-center adsorption and desorption for clusters ranging from two to six F centers accounted qualitatively for the occurrence of a delayed maximum and its disappearance (without any shift in the position in time of the maxima) for long irradiation times. However, Fig. 2 shows that the experimentally determined delayed maxima not only broaden for longer bombardment times, but also shift. Clearly, the previous model does not reproduce successfully the pronounced shift of the maximum as a function of bombarding time as it is observed experimentally (Fig. 2).[18] One possible explanation for this discrepancy is that the set of rate constants used previously in the rate equations are not consistent with the correct picture of cluster sta-

DESORPTION KINETICS OF Li-ATOM DELAYED EMISSION . . .

bility. The earlier work utilized a set of rate equations to describe the generation of F-center clusters caused by the aggregation of F centers. In this work, it was assumed that larger clusters have a higher formation rate (due to their larger geometrical size) and a higher thermal stability (in accordance with the Thompson equation,[19] where larger clusters have correspondingly lower vapor pressure). The latter condition clearly is valid for colloids,[6] but in general is not true for small F-center clusters consisting of only a few F centers. Optical absorption spectroscopy in the case of additively colored KCl has proven that the F_2 center (i.e., M center) is the most stable center of the small aggregates.[9] F_n denotes F-center clusters consisting of n F centers. It is responsible to assume that in the case of LiF the F_2 center is more stable than the F_3 center (R center).[21,21] Lord and Gallon[20] bombarded LiF at 310 °C under similar experimental conditions (900-eV electrons, similar bombarding times, but smaller currents) and investigated the optical absorption *in situ*. The authors observed that after 3 min of bombardment, there was almost no F band (probably neutralized Li ions, which desorbed thermally from the crystal surface) and no R bands, but the M band and also the N bands, due to F_4 centers, at 520 and 550 nm, were quite pronounced. This is a strong evidence that some of the small F-center clusters like the F_2 centers are very stable in LiF at high temperatures while others are not. As mentioned in the Introduction, the fact that the small F-center clusters are homogeneously distributed in pure crystals, whereas colloids are not, hints that one or more of the small F-center clusters (consisting of more than five F centers [6]) are thermally rather unstable. The rate constants chosen under the assumption that the Thompson equation is valid throughout the range of cluster sizes would therefore not be valid for small F-center clusters.

Using the assumption that the Thompson equation governs the stability of the cluster distribution, the rate equations predict that the density of the biggest clusters formed during electron bombardment is an order of magnitude larger than the density of F centers.[18] If we identify the biggest cluster with colloids, this would mean that we have during bombardment a larger density of colloids than of F centers. This is a not a physically reasonable result, since a typical saturation colloid density is known to be approximately $10^{16}/cm^3$,[6] whereas under our experimental conditions the F-center density is readily calculated to be about $10^{19}/cm^3$ as will be shown. Therefore the F-center density is much higher than the density of colloids under our experimental conditions, which is contrary to the predictions of the rate equations. The rate equations and the chosen set of rate constants therefore do not describe formation and decay dynamics of colloids. The following discussion will show that under our experimental conditions large clusters (colloids) are not formed in significant quantities.

Following Hughes and Lidiard[14] we assume that 100 eV are needed to create an (F,H)-center pair, which leads to approximately $10^{15}F$ centers per second formed under our experimental conditions. The penetration depth characteristic of 300-eV electrons is about 100 Å.[22] Since the F centers are very mobile at 400 °C, most of the

F centers form Li atoms at the surface of the crystal and evaporate very quickly. Therefore only a relatively small number of F centers remains in the crystal at these temperatures. An analysis of our data show that the ratio between the amount of Li desorbing during irradiation and desorbing after irradiation is about 50 or larger. Furthermore, we can say that the F-center diffusion tends to smooth out concentration differences along the diffusion depth (see Fig. 7). The mean diffusion length L of F centers at 400 °C equals approximately

$$L = \sqrt{Dt} \ ,$$

where D denotes the diffusion constant and t the diffusion time. D is approximately 10^{-9} cm^2/s at 400 °C.[14] The diffusion length L obtained using the simple Einstein diffusion relation shown above equals approximately 3000 Å in 1 s. In the diffusion calculations (Fig. 7), the F-center concentration in the crystals after 1 s of bombardment equals 10^{19} F centers per cm^3 based on an average diffusion length of 1000 Å (Fig. 7) rather than the penetration depth of the electrons (60 Å). Note that $10^{19}F$ centers per cm^3 corresponds to an effective dose of about 10 Mrad, under the assumption that 100 eV is required to produce one (F,H)-center pair. Under the assumption that colloids in LiF are as stable as colloids in NaCl, the dose is much too low to form large colloids in LiF. In NaCl colloids are produced typically at a dose of more than 100 Mrad, under conditions of lower temperatures (250 °C) (Refs. 6 and 14) where the colloids are more stable. As mentioned earlier, Lord and Gallon[20] observed, under experimental conditions almost identical to our own, that the M band (i.e., F_2) is the dominant band in LiF with no indication of significant concentrations of larger clusters. In summary, we can say that the formation of colloids under our experimental conditions is very improbable.

Our experiments are consistent with the supposition that the stability of successive cluster sizes varies markedly in a nonuniform manner. Indeed, we find no evidence for a uniform distribution of cluster sizes. If, for example, in the course of cluster formation, one cluster size was distinctly unstable, this would inhibit further growth and consequently would severely limit the population of larger clusters largely independent of total dose. This is consistent with the experiments of Serughetti et al.,[21] which showed that F_3 is much less stable than F_2 and F_4 in LiF.

The fact that doping the crystals had a pronounced effect on whether the delayed maxima was observed at all (Fig. 5) and also influenced markedly the magnitude of the delayed yields (Figs. 3 and 5) indicates that the centers responsible for the delayed emission and for the occurrence of the delayed maximum are located in the crystal at impurity sites (*heterogeneously*). The observation that the delayed yield is larger for the doped crystals cannot be explained by an higher effective production rate of F centers caused by the doping of the crystal, since the desorption rate *during* the electron bombardment is lower and the effective production rate (sum of the delayed yield plus the integral over the desorption

rate during bombardment) is actually smaller in the case of the doped crystals. It is well known that the introduction of divalent cation impurities into alkali halides strongly affect the formation efficiency of small F-center agglomerates during irradiation.[10,11,23] High concentrations of divalent impurities suppress the formation of colloids, probably because the F centers are stabilized at impurity sites, which decreases the average radius of the clusters.[23] This is further evidence that colloids do not contribute to the occurrence of the delayed maximum, since under our experimental conditions the delayed maximum was much more pronounced in the case of the *doped* crystals. The influence of divalent impurities on the formation rate of small F-center clusters is quite complicated and depends on the irradiation dose and on the amount of impurities in the crystal. For high doping levels and high irradiation doses, the F_2-center concentration increases even when the concentration of F centers saturates at temperatures around room temperature.[10,11] Schneider[13] observed in additively colored KCl:Na and KCl:Li, that F_{2A} centers (F_2 centers at impurity sites) are more stable than homogeneously distributed F_2 centers (not associated with impurity centers). Those investigations were performed at temperatures equal to and lower than room temperature.

Figure 4 clearly shows that the bombarding time has a much greater influence on the shape of the delayed emission than the total integrated dose. Increasing the production rate (current density) by a factor of 2 has almost no influence on the characteristics of the decay. A four times higher bombarding time (as well as a two times larger one as shown in Fig. 2) causes the delayed maximum to shift significantly, although the total dose was the same. The model of Betz and co-workers does not reproduce this effect. In this model, changing the production rate, or the bombarding time, results in practically no shift of the delayed maximum with the chosen rate constants of Ref. 18. A possible solution to this discrepancy is the consideration of diffusion. According to Fig. 7 the maximum concentration of F centers saturates at a concentration of 0.08 (arbitrary units) after a few hundreds of a second for the penetration depth of about 60 Å, caused by enormous losses of F centers to the surface and into the bulk. After a few seconds of electron bombardment, the F-center concentration at 800 Å equals half the maximum value of the F-center concentration at 60 Å. Consequently F-center clusters are formed relatively deep in the crystal, and it takes about the same time for the F centers after disintegration of the F-center clusters to reach the surface as it took them to diffuse into the bulk. If diffusion is taken into account, it follows that clusters are formed deeper in the crystals for longer bombardment times. The longer the bombardment time, the deeper F-center clusters are formed in the crystal, and the longer it takes them to diffuse back to the surface. Furthermore, the probability that an evaporated F center from an F-center cluster or any F-center sink may be captured again during its diffusion to the surface cannot be routinely neglected. Figure 7 also shows that the F-center concentration in the bulk stays rather high for several tens of a second *after* the electron bombardment.

Therefore the observed prompt decay of the Li desorption rate (Fig. 5) of one order of magnitude does not indicate that most F centers in the bulk diffused to the surface in a few ms (which was our time resolution). F centers may be captured by sinks even after the bombardment. This capture, escape, and diffusion process could qualitatively explain the broadening and the shift of the delayed maximum. The fact that 300 and 60-eV electron bombardment gives very similar results, although the penetration depths of the electrons are 26 and 3 monolayers, respectively, is also consistent with an heterogeneous nucleation model which incorporates diffusion, since the average diffusion length is much larger than the electron range under our experimental conditions.

The delayed yield depends approximately linearly on the current, which is shown most vividly in the case of doped crystals (Fig. 6). If we assume that the major contribution to the delayed yield results from the disintegration of small F-center clusters, then our data suggest that the total number of F_2, F_3, etc. centers formed during irradiation is proportional to the production rate of F centers. It is well known that, at least in pure crystals, the F_2-center concentration is quadratic in the F-center concentration.[10,11] Therefore, if we assume that the F-center concentration is proportional to the current density (in Fig. 6 the bombarding times are 3 s, which is much smaller than the time for saturation ~ 40 s), we should rather observe a quadratic relation than a linear one. One possible solution is that F centers are stabilized at impurity sites forming what are commonly referred to as Z centers. Even the formation of F_2 centers at impurity sites would then be rather linear than quadratic in the current density under the condition that the Z centers are stable enough. The lifetime of Z centers have not been investigated so far for temperatures around 400 °C in the case of LiF, but we know that they are very stable at least up to 200 °C.[24] If the major contribution to the delayed yield results from diffusing single F centers rather than from evaporated F centers form clusters or impurities, the linearity is not surprising, since the diffusion equation is linear in the production term. However, the fact that diffusion alone cannot explain the occurrence of the delayed maximum shows that diffusion and clustering processes have to be taken into account.

It is interesting to note that for a given temperature the shape of the curve obtained by plotting the delayed yield as a function of irradiation time does not depend on doping, or current density as shown in Fig. 3. In all cases the delayed yield approaches saturation after an irradiation time of approximately 40 s. This behavior is not easily explained by invoking only cluster disintegration and diffusion. Figure 8 is a plot of delayed yield calculated as a function of bombardment time taking into account diffusion. Clustering has no influence on this effect under the assumption that all the clusters eventually decay into F centers. Note that in this calculation, the delayed yield continues to rise monotonically with no indication of saturation. We interpret this to mean that there are loss terms, perhaps associated with recombination that we have not taken into account.

Our data and calculations indicate that the model of

Betz and co-workers should be modified to take into account heterogeneous nucleation, where F centers form clusters of nonuniform stability near impurity sites. Time- and production-rate-dependent investigations strongly suggest that diffusion of F centers also has to be taken into account. We further showed that no large colloids are formed under our experimental conditions. Further investigations and calculations taking into account diffusion, nonuniform clustering, and recombination are in progress.

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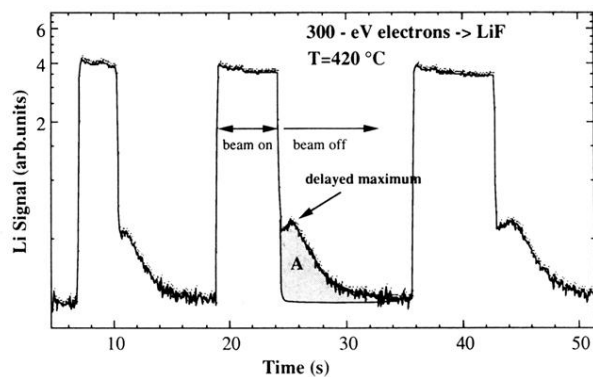[26]Y. Al. Jammal, D. Pooley, and P. D. Townsend, J. Phys. C 6, 247 (1973).

FIG. 1. The emission of ground-state Li atoms from LiF crystals containing 10^{-3} mol % MgF_2 under pulsed electron irradiation is shown in a logarithmic plot. After the electron bombardment is stopped, the Li signal exhibits a prompt decrease followed by a delayed emission in the order of seconds (area A). The position of the delayed maximum shifts for longer irradiation times.