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Neutron irradiations in oxidized lithium-doped MgO crystals

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The effect of neutron irradiation on lithium-doped MgO crystals is investigated using optical-absorption and EPR measurements. Unlike electron irradiation, neutron irradiation does not produce stable $[\mathrm{Li}]^0$ centers in as-grown MgO:Li crystals. In crystals containing stable $[\mathrm{Li}]^0$ centers produced by oxidation at elevated temperatures, neutron irradiation results in a decrease of $[\mathrm{Li}]^0$ concentration and the formation of stable V^0, V^-, V_{OH}, and V_F centers. The loss of $[\mathrm{Li}]^0$ centers is attributed to the recombination of holes from these centers with electrons at multivacancy defects created by neutrons.

I. INTRODUCTION

During growth of lithium-doped MgO crystals by the arc-fusion technique, the crystals were subjected to a reducing environment caused by the graphite electrodes. The result is that most of the lithium impurities are present in a Li_2O second phase.[1] A subsequent heat treatment in oxygen causes the Li^+ ions from the Li_2O precipitates to disperse into the MgO matrix, resulting in the formation of two centers: the paramagnetic $[\mathrm{Li}]^0$ center (substitutional Li^+ ion with a trapped hole at an adjacent oxygen ion), which absorbs optically at 1.8 eV (680 nm), and an unknown diamagnetic center whose absorption centers at 5.3 eV (235 nm).[2–5] Most of the $[\mathrm{Li}]^0$ centers concentrate in the vicinity of dislocations.[6,7] This method of producing $[\mathrm{Li}]^0$ centers at high temperatures is very efficient in that it takes only a few minutes to achieve a saturation level. Another way of dispersing Li^+ ions from the Li_2O precipitate into the MgO solute involves a radiation-induced diffusion process,[4] similar to that for hydrogen in MgO.[9] This process requires high-dose electron irradiations of $> 10^{17}$ e/cm^2. It is natural, therefore, to inquire whether neutron irradiations would have similar effects. The purpose of the present study is to investigate the differences between the effects of electron and neutron irradiations and how they relate to the dissolution of Li_2O precipitates and to the interaction between defects.

II. EXPERIMENTAL DESCRIPTIONS

Single crystals of MgO:Li were grown by the arc-fusion technique[8] using a mixture of 5 wt.% Li_2CO_3 and high-purity MgO powder from the Kanto Chemical Company. The concentration of lithium impurities in the resulting crystals was analyzed by atomic emission spectrophotometry to be approximately 0.04 at.%. Heat treatment of the samples in flowing oxygen was performed inside a quartz tube inserted in the horizontal axial hole of a Sentry furnance model 7 AV. Optical-absorption measurements were performed with a Cary model 17D and a Perkin-Elmer infrared spectrophotometer. A Sulfrian optical cryostat was used for low-temperature measurement. Electron paramagnetic resonance (EPR) spectra at X band were measured at 87 ± 1 K in a Varian 4531 cavity. The absolute concentrations of the different defects were determined by comparing the resonance line of the defects with that of the Cr^{3+} signal from another MgO crystal. The Cr^{3+} concentration of this latter sample had been standardized by several techniques.[9] The values of the concentrations, as determined by EPR, are believed to be accurate within ±25%. Electron nuclear double resonance (ENDOR) measurements were made at ~1.2 K using X-band (~9.5 GHz) superheterodyne systems.

Neutron irradiations were performed in the CP-15 facility of the Oak Ridge Bulk Shielding Reactor, where the neutron flux with energy exceeding 1 MeV was 4.4×10^{12} $n/\mathrm{cm}^2\,\mathrm{sec}$. The thermal-neutron flux was $\sim 2.5 \times 10^{13}$ $n/\mathrm{cm}^2\,\mathrm{sec}$. The sample temperature during irradiation was about 350 K. A 2-MeV Van de Graaff generator provided capabilities for electron irradiations. The beam intensities were typically 10–15 $\mu\mathrm{A/cm}^2$. Samples were cooled during electron irradiation by flowing water. ^{137}Cs served as a source of γ rays (1×10^6 rad/h, average energy = 0.66 MeV).

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Crystal characterization

Impurities play an important role in radiation damage. This is especially true of hydrogen whose presence in MgO is responsible for the formation of cation vacancies.[10] With the exception of lithium and hydrogen, the impurity contents in these crystals did not depart drastically from analyses published previously.[8]

It is possible to remove hydrogen in nominally pure MgO during crystal growth using the arc-fusion technique.[8] The same procedure which has been successful in completely removing hydrogen from undoped MgO crystals is not as effective in lithium-doped MgO. Infrared-absorption measurements [Fig. 1(a)] have shown that there is a broad absorption band, centering at 3430 cm^{-1}, which is probably due to O-H configurations in the MgO:Li crystals. The sharp lines at 3296 and 3700 cm^{-1}, which represent the most common frequencies observed in lithium-free hydrogen-containing crystals, were not detected. These two resonances are due to V_{OH}^{-} centers (linear configuration: OH^--[Mg vacancy]-O^{2-}) and Mg(OH)_2 precipitates, respectively.[11] Furthermore, a short ionizing γ irradiation did not produce the V_{OH} center (OH^--[Mg vacancy]-O^-), whose associated optical absorption would have been observable at a frequency of 3323 cm^{-1}. After the heat treatment in oxygen to produce stable $[\text{Li}]^0$ centers, the intensity of the broad spectrum at 3430 cm^{-1} diminished slightly, and there appeared to be no significant change in the band shape or the emergence of any discrete frequencies [Fig. 1(b)].

There appears to be a strong affinity of lithium-doped MgO crystals for hydrogen. Aside from the unsuccessful efforts to remove hydrogen completely from these crystals during crystal growth, it was found that hydrogen diffuses readily in these crystals. After the specimens were heated in flowing hydrogen at 1450 K for 5 min, the band at 3430 cm^{-1} increased in intensity. When they were heated in flowing deuterium under similar conditions, a broad band peaking at 2553 cm^{-1} was observed. The ratio of the frequencies is 1.34, which corresponds well to the theroretical expectation of $[\mu(\text{OD})/\mu(\text{OH})]^{1/2} = 1.37$ for the two isotopes, where μ is the reduced mass of the radicals. These results leave little doubt that the infrared absorption at 3430 cm^{-1} is due to O-H stretching frequencies.

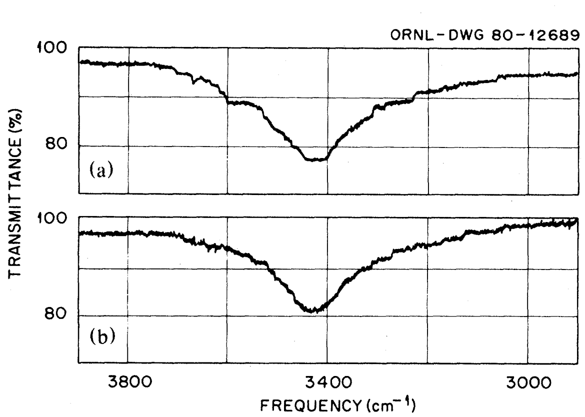

FIG. 1. Infrared-transmission spectra for a MgO:Li crystal (a) before and (b) after heat treatment in oxygen at 1250 K for 10 min.

Magnetic-resonance techniques were used to verify the infrared results. EPR measurements were made on virgin and subsequently oxidized crystals, both before and after γ irradiations. Paramagnetic V_{OH} centers[12] were not detected in either the virgin or the oxidized crystals following γ irradiation. ENDOR measurements on the $[\text{Li}]^0$ centers, however, revealed that distant protons were present. These protons may well be responsible for the broad infrared absorption centered at 3430 cm^{-1}.

B. Effect of neutron irradiation on $[\text{Li}]^0$ population

Whereas electron irradiations produce stable $[\text{Li}]^0$ centers in virgin crystals, neutron irradiations do not. Even after doses of $\sim 10^{18}$ $n\,\text{cm}^{-2}\,\text{sec}^{-1}$, $[\text{Li}]^0$ centers were not observed. Therefore, we proceeded to examine the effects of neutron irradiations on crystals containing stable $[\text{Li}]^0$ centers produced by oxidation. The optical absorption between 1–6 eV for a MgO:Li crystal heat-treated for 10 min at 1250 K in oxygen is shown as curve (a) in Fig. 2. The most pronounced absorption was the $[\text{Li}]^0$ band, absorbing at 1.8 eV (680 nm). The weak absorption near 4.8 eV is characteristic of MgO:Li after oxidation at 1250 K. Optical-absorption curves following several neutron irradiations are shown as curves (b)–(f) in Fig. 2. Several observations are noteworthy. (1) The composite F^+ and F bands (corresponding to one- and two-electron anion vacancies, respectively), both of which absorb at ~ 5.0 eV (~ 250 nm),[13] increased in intensity with irradiation dose, but at a slower rate than that in undoped MgO crystals[14]; in neutron-irradiated crystals, anion vacancies are primarily in the F^+ state.[15] (2) The absorption coefficient of the 1.8-eV band decreased with neutron dose. (3) At doses near 1×10^{17} n/cm^2 the band peak shifted to about 2.3 eV. (4) At still higher doses the 2.3-eV band began to vanish, and a narrower absorption band emerged at 2.2 eV.

The decrease of the optical absorption at 1.8 eV is plotted as a function of neutron dose in the upper curve of Fig. 3. Since the 1.8-eV absorption includes contributions from other defects at doses $> 1 \times 10^{17}$ n/cm^2 such as defects responsible for the 2.3-eV band, the upper curve is denoted as due primarily to $[\text{Li}]^0$ centers. EPR measurements were also made at different doses to determine the various paramagnetic defects which could contribute to the optical absorption in the 1- to 3-eV region. It was confirmed that $[\text{Li}]^0$ centers were the only trapped-hole defects prior to irradiations. With increasing neutron doses, the $[\text{Li}]^0$ concentration diminished, and the decrease was

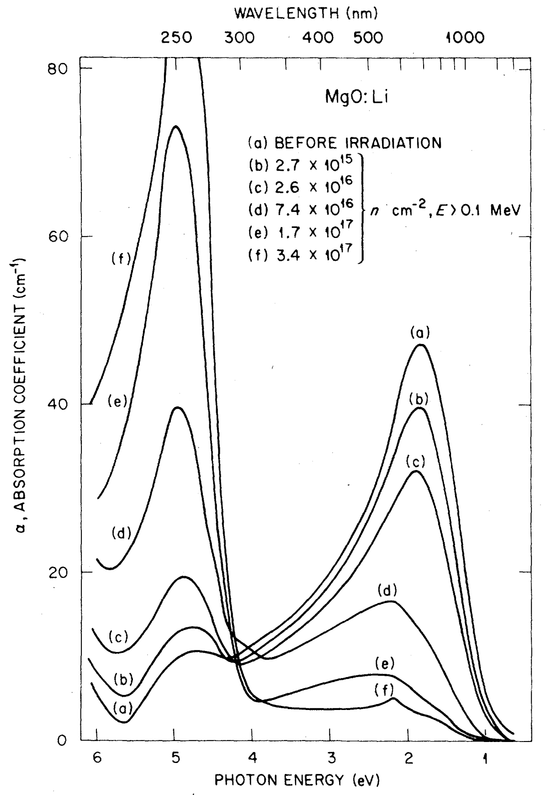

FIG. 2. Optical-absorption spectra for a MgO:Li crystal oxidized at 1250 K for 10 min, and subsequently irradiated with several doses of energetic neutrons.

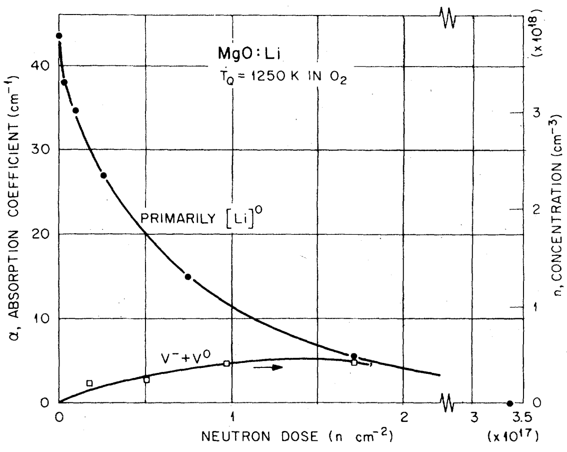

FIG. 3. Optical-absorption coefficient and concentration vs neutron dose for a MgO:Li crystal initially oxidized at 1250 K for 10 min. The optical absorption was monitored at 1.8 eV (upper curve), representing $[\mathrm{Li}]^0$, V^-, and V^0 centers. The lower curve represents ($V^- + V^0$) concentrations measured by EPR. The concentration of the $[\mathrm{Li}]^0$ center was determined from the optical-absorption coefficient using Smakula's formula: $n = 6 \times 10^{15} f^{-1} W \alpha$ where the oscillator strength $f = 0.1$ and the half-width $W = 1.44$ eV.

attended by the emergence of stable V^-(O^{2-}-[Mg vacancy]-O^-), V^0(O^--[Mg vacancy]-O^-), V_F(F^--[Mg vacancy]-O^{2-}), and V_{OH} centers. All these trapped-hole centers are known to absorb optically at about 2.3 eV.[16,17] Magnetic-resonance experiments showed that the concentrations of V_F and V_{OH} centers were negligible compared with those of the V^- and V^0 centers. (The V^- center observed here is unusual in one respect: an isotropic EPR signal could not be observed at 255 K or above, even though it had the proper axial g values at low temperature and did not possess an aluminum ENDOR signal.[18,19] We shall assume that this EPR signal is due to the V^- center by virtue of the presence of V^0 centers.[10]) The concentrations of the V^- and V^0 centers were plotted against neutron dose in the lower curve of Fig. 3. Unlike trapped-hole centers in undoped crystals,[17] these four centers were indefinitely stable at room temperature; EPR measurements indicated that the concentration of these centers remained unchanged over a four week period. The stability of the V^0 and V^- centers need not be surprising, since the conditions that made the $[\mathrm{Li}]^0$ centers electronically stable would also make other trapped-hole centers electronically stable. If these trapped-hole centers are present within lithium-rich regions (referred to as microgalaxies[3]), in which charge neutrality must be maintained, they would appear to be thermally stable by virtue of the holes hopping from site to site.

C. Mechanism for $[\mathrm{Li}]^0$ loss

The rate of decrease of the $[\mathrm{Li}]^0$ concentration was unexpectedly large. If one assumes that the loss of the $[\mathrm{Li}]^0$ centers was due entirely to displacements of the Li^+ ions, resulting in the formation of V^- and V^0 centers, then the displacement cross section σ is estimated to be $\sim 10^7$ b, using the relationship $\sigma = -(\Delta N/\Delta\phi)N^{-1}$ where N is the initial concentration of the $[\mathrm{Li}]^0$ centers and $-\Delta N/\Delta\phi$ corresponds to the initial decrease of the $[\mathrm{Li}]^0$ concentration per unit dose, as deduced from Fig. 3.

The more massive neutrons produce single vacancies, multiple vacancies, and extended defects by elastic collisions, while the Compton electrons produced by the concomitant γ rays during neutron irradiation can also produce vacancies by ionization effects or elastic collisions. In order to decipher whether it was the neutrons or the Compton electrons that were responsible for the loss of $[\mathrm{Li}]^0$ centers, electron irradiations at 300 K were carried out with a Van de Graaff generator on an oxidized crystal. After an irradiation dose of 2×10^{17} e cm^{-2} was accumulated, an *increase* in $[\mathrm{Li}]^0$ concentration of $\sim 10\%$ was observed by both EPR and optical absorption. Furthermore, no V^-, V^0, V_{OH}, or V_F centers were observed by EPR. Therefore, while the magnitude of the cross section could be compatible with the ionizationinduced displacement of Li^+ ions, the loss of $[Li]^0$ centers was clearly not due to the Compton electrons during neutron irradiations.

The effect of transmutation of lithium was also considered. The nuclear reaction for the 7% abundant ^6Li is $^6Li(n, \alpha)T^3$ with a cross section of 953 b, and that for the 93% abundant ^7Li is $^7Li + n \rightarrow {}^8Li \rightarrow 2\,{}^4He + \beta^-$ with a cross section of 0.04 b. Both cross sections appear to be too small to account for the loss of $[Li]^0$ centers due to the large thermal-neutron fluxes. For experimental verification that transmutation is not involved, a simple experiment to prevent thermal neutrons from penetrating the samples was employed. A crystal, previously oxidized at 1250 K in oxygen, was split into two pieces. One piece was wrapped in several layers of cadmium foil, which serves to capture thermal neutrons, and the other piece was not wrapped. Both pieces were irradiated to a dose of 3.4×10^{17} $n\,cm^{-2}$, and both were observed to have experienced almost total loss of $[Li]^0$ centers. Therefore, we conclude that the loss of $[Li]^0$ centers in neutron-irradiated crystals was not due to transmutation effects by thermal neutrons.

Having ruled out the effects due to Compton electrons and nuclear transmutations, we now propose that the mechanism responsible for the loss of $[Li]^0$ centers is caused by elastic collisions with energetic neutrons and is associated with multiple-vacancy defects produced by these neutrons. The probability for producing cation vacancies by displacements of substitutional $[Li]^+$ ions is small and therefore inadequate to account for a cross section of 10^7 b. A one-to-one correspondence between $[Li]^0$ centers destroyed and Mg vacancies created failed to exist, since the $(V^0 + V^-)$ concentration produced by the neutron irradiation constituted only a small fraction of the $[Li]^0$ centers annihilated. Furthermore, 2-MeV electrons, which are energetic enough to produce primarily single vacancies in MgO, do not result in loss of $[Li]^0$ centers, as we noted. Therefore, we attribute the mechanism to higher-order defects. We propose the following:

The optically detectable defects which energetic neutrons produce in MgO crystals are anion vacancies (primarily F^+ centers), anion divacancies which absorb optically at 1.3 and 3.5 eV (975 and 355 nm, respectively), and an unidentified multiple defect which absorbs at 2.2 eV (573 nm). These defects are known to be electron centers.[16] When these centers are produced in the $[Li]^0$-rich microgalaxy, the holes which are normally hopping from $[Li]^0$ to $[Li]^0$ sites are captured by these electron centers. The net result is that the $[Li]^0$ band and the absorption bands responsible for the electron centers are mutually annihilated. The F^+ center cannot capture a hole, since the F^{2+} center (the "nude" anion vacancy) is energetically unfavorable and occupies an energy level in the valence band[20]; indeed, it does not appear from Fig. 2 that the F^+ center and the $[Li]^0$ annihilate one another. The divacancy bands[16,21] at 1.3 and 3.5 eV, representing four-electron centers, are in a position to accommodate holes from $[Li]^0$ centers, thereby creating a situation in which both the $[Li]^0$ and the divacancy bands are mutually annihilated. Presumably, the same may be true for the defects responsible for the 2.2-eV band. If this scheme is correct, one would expect that holes normally available to $[Li]^0$ centers would diminish with neutron dose and, when the holes are depleted, the electron centers would experience a monotonic growth with increasing dose. It can be seen that the 2.2- and the 1.3-eV bands became readily discernible after the $[Li]^0$ centers disappeared [Fig. 2(f)]. If the decay of the $[Li]^0$ band is caused by displacements of indigenous lattice ions rather than Li^+ ions, then the cross section would be $\sim 10^3$ rather than $\sim 10^7$ b, since N would be 5×10^{22} rather than 4×10^{18} cm^{-3}. This value of 10^3 b is compatible with the magnitude estimated for the production of multiple-vacancy defects.[22]

A further test for this model can be made. If the loss of $[Li]^0$ centers is due to the annihilation of holes by electrons from multiple-vacancy defects, it should be possible to prevent the electron-hole recombination by "freezing-in" the holes at their respective $[Li]^0$ sites by maintaining the sample at low temperature following an ionizing irradiation. Both the $[Li]^0$ and the multiple-vacancy bands would then increase in intensity. Figure 4 illustrates the low-temperature versions of the optical spectra shown in Fig. 2(f) before and after a short electron irradiation at 77 K. (The crystal had been oxidized at 1250 K and subsequently neutron irradiated to a dose of 3.4×10^{17} $n\,cm^{-2}$.) It is evident that, after the ionizing irradiation, the $[Li]^0$ band and the divacancy bands, which are represented by the attending sharp zero-phonon transitions[16,21,23] at 1.87 and 3.430 eV (1044.5 and 361.5 nm, respectively), increased in intensity. In undoped MgO, an ionizing radiation at low temperatures has little effect on the intensity of these bands.[24]

It appears, therefore, that multiple vacancies are responsible for the annihilation of the $[Li]^0$ band. The problem of determining how the anion vacancies, V^- and V^0 centers, in MgO:Li crystals are formed during neutron irradiation, on the other hand, is more complex. They are probably formed by the displacement of either substitutional H^+ or Li^+ ions. The uncertainty in determining the ion is attributed to the fact that although we can normally produce hydrogen-free MgO crystals we have been unsuccessful in producing hydrogen-free MgO:Li crystals. Past investigations using infrared absorption, EPR, and ENDOR techniques have shown conclusively that these intrinsic vacancies are formed by the ejection of substitutional hydrogen (in V_{OH}^- and

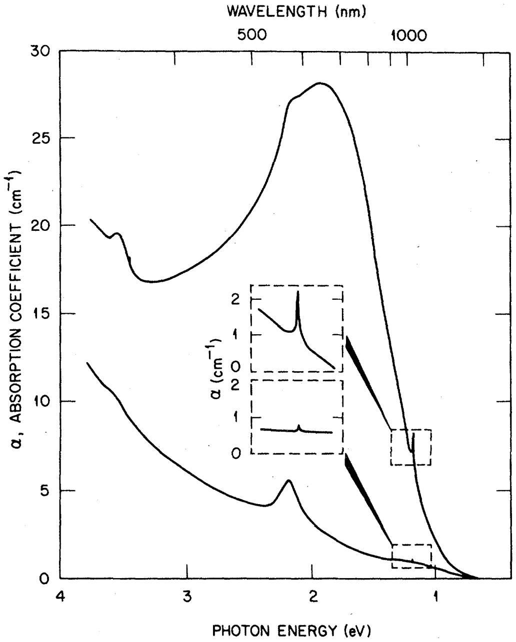

FIG. 4. Optical-absorption spectra at 77 K before (bottom curve) and after (top curve) a short electron irradiation of a MgO:Li crystal which had been oxidized at 1250 K for 10 min and subsequently neutron irradiated to a dose of 3.4×10^{17} n/cm^2. The two insets illustrate the intensities of the zero-phonon transition at 1.187 eV (1044.5 nm) which correspond to the anion-divacancy concentration.

$V_{\rm OH}$ centers) via an efficient process involving radiation-induced displacement[25]; irradiations of hydrogen-free crystals either by neutrons or electrons do not produce these centers.[24] Even though the $V_{\rm OH}^-$ and $V_{\rm OH}$ centers were not observed in the virgin and oxidized MgO:Li crystals, it is incorrect to assume that they cannot be formed during neutron irradiations; in fact, stable $V_{\rm OH}$, as well as V^- and V^0 centers, were observed after irradiating oxidized crystals with neutrons. So far, we were able to form V^- and V^0 centers only for those crystals which had been oxidized to form stable [Li]^0 centers and subsequently neutron irradiated. Electron irradiations of oxidized crystals do not produce these vacancies, nor do neutron irradiations of virgin MgO:Li crystals. At present we have no basis to determine unequivocally whether the V^- and V^0 centers were formed at the expense of substitutional H$^+$ or Li$^+$ ions.

IV. CONCLUSIONS AND SUMMARY

Two contrasting effects are noted when comparing results of the electron irradiations with those of neutron irradiations in lithium-doped MgO. In virgin MgO:Li crystals electron irradiations produce stable [Li]^0 centers,[2] whereas neutron irradiations do not. On the other hand, in crystals containing stable [Li]^0 centers produced by oxidation at elevated temperatures, neutron irradiations result in a decrease of the [Li]^0 concentration, while electron irradiations produce an increase.

In virgin crystals the formation of stable [Li]^0 centers by bombarding electrons is caused by their ionizing property.[3] Neutrons do not induce ionization. In a fission reactor there are attending Compton electrons, but the flux is much less than that available from a Van de Graaff generator.

In oxidized crystals the ionizing property of the electrons increases the concentration of the stable [Li]^0 centers. Neutron irradiations, however, result in loss of [Li]^0 centers. The most probable mechanism involves a recombination of holes from the [Li]^0 centers and electrons at multivacancies created by neutron irradiations. The rejuvenation of the [Li]^0 and divacancy bands by a short ionizing irradiation at low temperature (caused by the immobilization of holes trapped at [Li]^0 centers) lends credence to such a mechanism. The decoloration of the [Li]^0 band in this investigation appears to be similar to that obtained in plastically deformed MgO:Li crystals in which the loss of [Li]^0 coloration was selectively displayed along slip planes.[26]

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