High-temperature and ionization-induced effects in lithium-doped MgO single crystals*

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Thermal quenching of Li-doped MgO crystals from temperatures in excess of 1300 K results in the formation of $[Li]^0$ defects, which are substitutional lithium ions each with a bound hole at a neighboring oxygen site. Unlike $[Li]^0$ defects generated at low temperatures by a low dose of ionizing radiation, those formed by quenching are stable against hole release well above room temperature. This stability is much higher than for all previously reported hole defects in alkaline-earth oxides. These stable $[Li]^0$ defects have also been produced by high-dose electron irradiations at both 290 and 85 K, with formation cross sections of $\sim 10^5$ and 10^3 barns, respectively. The magnitude and temperature dependence of the cross section suggests a radiation-induced diffusion, rather than an elastic collision, process as the production mechanism. These defects anneal at 450 K in the electron-irradiated crystals and 830 K in the quenched ones. A model involving localized regions of high lithium concentration is proposed in order to explain the stability of the bound hole at the $[Li]^0$ defect.

I. INTRODUCTION

The effects of radiation on the electronic properties of alkaline-earth-oxide crystals have been the subject of extensive investigation for a number of years.^{1,2} Both electron paramagnetic resonance (EPR) and electron-nuclear double resonance (ENDOR) techniques have played central roles in these investigations and, in correlation with optical data, have resulted in the establishment of definitive models for many of the defect's formed in these materials. The electronic properties of the particular class of radiation-induced centers known as "trapped-hole" or V-type centers have received considerable attention, and the ensuing results have provided valuable information concerning the nature of both intrinsic and impurityrelated defects in the alkaline-earth oxides.³ The intrinsic trapped-hole centers that have been identified in MgO include the V^- and V^0 centers, which are alkaline-earth vacancies with one and two trapped holes, respectively.⁴⁻⁹ The earliest investigations of impurity-related trapped-hole centers were naturally directed toward those defects associated with impurities which either commonly occurred in the oxides or which were inadvertently introduced as a result of some particular crystalgrowth technique. Examples of these defects are: the $V_{\rm F}$ and $V_{\rm OH}$ centers¹⁰⁻¹² in which an alkalineearth vacancy is charge-compensated by a fluorine or hydroxyl ion respectively, substituting for a neighboring oxygen ion, and the V_{A1} center⁵ in which an alkaline-earth vacancy is compensated by a nearby trivalent aluminum ion. A third category of trapped-hole centers is that which can be formed after the deliberate addition of an alkali ion by diffusion into undoped crystals or by doping the melt during crystal growth using the arc-fusion

16

technique.¹³ This type of center is composed of an alkali-metal ion located at a substitutional cation site with a hole trapped on one of the six nearest-neighbor $ions^{9,14-20}$ (linear configuration: O^{2} -Li⁺- O^{-}). In general, it is necessary to subject the sample to ionizing radiation in order that the de-fect traps a hole and becomes optically and paramagnetically active.

A common characteristic of trapped-hole defects in the alkaline-earth oxides is that at some temperatures, they become unstable. The centers formed for the alkali ions, $[Li]^{\circ}$, $[Na]^{\circ}$, and $[K]^{\circ}$, by low-temperature irradiation are not stable at room temperature.¹⁴⁻²⁰ Past investigations have indicated that the thermal decomposition of trapped-hole defects occurs as a result of loss of the trapped holes rather than by the capture of electrons.³ Therefore, it is expected that the rate at which a trapped hole leaks off will increase with the distance of the O⁻ ion (which represents a hole trapped at an O^{2-} ion) to the net positive charge of the aliovalent impurity. Figure 1 depicts the linear configurations of five typical trappedhole centers in MgO. We would expect the order of increasing stability to be $[Li]^0$, V_{OH} , V_F , V_{A1} , and V^- centers (top to bottom), since the O⁻ to aliovalent-impurity distances are ~2.1, 3.2, 4.2, and 6.3 Å and infinity, respectively.⁹ Indeed, this appeared to be the case. At 295 K, both the [Li]^o and $[Na]^0$ centers were found to be unstable.^{14,15,17} The V_{OH} center has a half life of hours, and the V_{A1} of days, while the V⁻ center is the most stable with a half life of years.³ The activation energy E_A for the release of the trapped hole appears to bear out the expected stability. For the V_{OH} center, $E_A = 0.84 \text{ eV.}^2$ An activation energy of 1.13 ± 0.5 eV was obtained by Searle and Glass²¹ for the 2.3-eV band, which we speculate to consist pri-



FIG. 1. Schematic depiction of five trapped-hole defects for MgO in order of increasing distance of the aliovalent impurity from the trapped hole: $[Li]^0$, V_{OH} , V_F , V_{A1} and V^- centers. The O⁻ ion represents a hole localized on an O²⁻ ion site.

marily of the V_{A1} defects.⁵ In proton-irradiated MgO powder, Tench and Duck²² obtained $E_A = 1.6 \pm 0.2$ eV for a "stable V⁻ center," which now appears to be the real intrinsic V⁻ center. This center should be distinguished from the less stable "V⁻ center," which was probably the V_{A1} center.⁵

Recently it was discovered that quenching Lidoped MgO crystals from high temperatures, or performing a heavy electron irradiation, produces [Li]^o centers in MgO which are indefinitely stable at room temperature.²³ Investigations using EPR and ENDOR techniques at 4.2 K established that both stable [Li]⁰ centers have the same local electronic configuration as the unstable centers created at low temperature. Furthermore, a motionally averaged spectrum was observed at higher temperatures for the stable [Li]⁰ system, verifying that a centrosymmetric system (i.e., the hole can reside on any one of the six equivalent surrounding oxygen ions) is indeed involved, and no axial impurity charge compensation is present. Both the unstable and stable [Li]⁰ centers show the same motional averaging with rising temperature in the EPR spectrum. However, the unstable center decays before a single isotropic averaged line can be observed. The difference in stability for the same defect configuration is *unexpected*. and contrary to our present understanding of the production and stability of trapped-hole centers.

The present study has a threefold purpose:

First, we establish the dependence of the production of $[Li]^0$ centers on the quenching temperature, or electron dose. Secondly, the stabilities of the $[Li]^0$ centers produced by the three different methods (low-temperature γ irradiation, heavy electron irradiation and quenching from high temperatures) are determined. Thirdly, we explore factors contributing to the stabilization of holes at $[Li]^0$ centers.

II. EXPERIMENTAL PROCEDURE

The single crystals of MgO used in these experiments were grown by an arc-fusion technique¹³ using MgO powder from the Kanto Chemical Co., Tokyo. The starting MgO powder was mixed with Li_2CO_3 to a concentration of about 5% by weight, but the actual concentration of lithium in the resulting single crystals was determined by chemical analysis to be approximately 0.03–0.05 at.%.

Optical-absorption measurements were taken on a Cary model 14R spectrophotometer at room temperature. For EPR measurements, an X-band homodyne spectrometer with 100-kHz field modulation was used. EPR spectra were observed at 90 K by means of a Varian variable temperature Dewar system and isochronal annealing studies below 295 K were carried out *in situ*.

A Sentry Model 7 AV furnace was used for heat treatments at temperatures up to 1600 K in air. Samples were heated at the desired temperature for 10 min and quenched into a bath of liquid nitrogen. Liquid nitrogen serves as a coolant fast enough for quenching purpose and slow enough as to minimize sample cracking. Nevertheless, for lithium-doped MgO, an unusually high rate of sample cracking was observed for temperatures in excess of ~1300 K. The irradiating sources were: a ¹³⁷Cs γ source (1.1 × 10⁶ R/h, average energy of 0.66 MeV) and a 2.0-MeV Van de Graaff electron accelerator. γ irradiations were performed at both 77 and 300 K. Electron irradiations were carried out either in a specially designed irradiation chamber using water as a coolant, at intensities typically of 10 μ A/cm², or at 5 μ A/cm² in a custom-made liquid-nitrogen cryostat.

III. RESULTS

A broad optical absorption band at 1.83 eV was found to exist in all MgO crystals in which the $[Li]^{\circ}$ center exists. The parallel behavior of the band with the EPR $[Li]^{\circ}$ signal under various situations of production and thermal annealing, and a recent study using a MCD-EPR double-resonance technique,²⁴ unequivocally identified the optical transition at 1.83 eV as due to the $[Li]^{\circ}$ center. Hence the behavior of the $[Li]^{\circ}$ center can be monitored with either EPR or the 1.83-eV optical band.

A. Production of stable [Li]⁰ centers

1. Electron irradiation. For the radiation-induced [Li]^o center, an as-grown Li-doped MgO crystal was used. Electron irradiations were performed at two temperatures at a current of 4.4 μ A/cm². Flowing water at *T* = 283 K was used as a coolant for the high temperature irradiation, and it is estimated that the radiation heating would raise the temperature of the crystal to no more than 290 K. Low-temperature irradiations were performed in a chamber with liquid nitrogen in contact with the copper sample mount. The sample temperature was at 90 K or below during irradiation. Initially irradiated at T = 290 K, the sample gave rise to the growth curve shown in Fig. 2. which illustrates the increase in the absorption coefficient of the 1.83-eV band with electron dose. After annealing out the [Li]^o centers at $T \sim 1000$ K and slow-cooling to room temperature, the 90 K irradiations yield a growth rate of the [Li]⁰ center about two orders of magnitude less than that of the higher temperature irradiations. Using known half widths and oscillator strengths of trapped-hole centers, $W \sim 1.3$ eV and $f \sim 0.05$, and assuming that the total lithium content in the crystal was 400 ppm, the cross section for the production at room temperature irradiation was estimated to be $\sim 10^5$ b, and that for the low temperature $\sim 10^3$ b. This temperature dependence of the cross sections resemble that for the displacement of substitutional hydrogen in MgO.²⁵

Another Li-doped crystal was irradiated to high doses and at beam intensities varying between $15-20 \ \mu A/cm^2$. The sample was taped to a graphite



FIG. 2. Dose-dependent production of $[Li]^0$ centers by electron irradiation at T = 90 and 290 K.

block containing liquid nitrogen. At such a beam intensity the temperature of the sample was uncertain and probably exceeded 290 K, the irradiating temperature for the sample, whose data are shown in Fig. 2. Initially the sample was irradiated with γ rays at ~300 K, and the resulting spectrum is shown in Fig. 3(a). (Irradiation at 77 K would have produced the band at 1.83 eV, which would vanish upon warming to room temperature.) After electron irradiations to high doses, $\sim 10^{18} e/$ cm², the optical spectra exhibit a strong absorption band at 1.83 eV, shown in Figs. 3(b)-3(d). If the data of Fig. 3 were plotted as absorption coefficient vs electron dose, one would obtain a growth curve which is steeper than that shown in Fig. 2. Whether the increase is due to sample dependence, higher irradiating temperature, or the increased beam intensity has not been investigated.

In neither of the two Li-doped MgO crystals, for which data are shown in Figs. 2 and 3, were oxygen vacancies detected after electron irradiations, as is usually found in crystals not doped with lithium: EPR measurements failed to reveal the presence of the F^* center (an oxygen vacancy with one electron), and optical absorption measurements were not able to detect the presence of a band at ~5.0 eV, where both the F^* and F (oxygen vacancy with two electrons) centers absorb. However, subtraction of the optical spectra before and after irradiations (shown in Fig. 3) revealed a band at ~5.2 eV. No new EPR signals were observed that could be



FIG. 3. Optical-absorption spectra after (a) γ irradiation and electron irradiation with (b) $0.4 \times 10^{18} e/\text{cm}^2$, (c) $3 \times 10^{18} e/\text{cm}^2$, and (d) $5 \times 10^{18} e/\text{cm}^2$.



FIG. 4. Production of $[Li]^0$ defects by thermal quenching: α vs quenching temperature. No subsequent ionizing irradiations were necessary to produce the defects.

attributed to this optical band. Furthermore, a MCD experiment on the 5.2-eV band could not correlate any signal with this band.²⁴ It may be that the MCD at this energy is not sufficiently sensitive to produce a correlation. Notwithstanding, we tentatively conclude that the 5.2-eV band is due to a diamagnetic center.

2. Thermal production. An as-grown Li-doped crystal was also used to study the thermal generation of $[Li]^0$ centers. The sample was heated isochronally for 10 min in air at an elevated temperature, followed by a quench into a liquid nitrogen bath. This procedure was repeated several



FIG. 5. Optical absorption spectra of an MgO:Li quenched at T = 1306, 1430 and 1500 K. No subsequent ionizing irradiations were performed after the thermal quenching.

times at increasing temperatures. The absorption coefficient of the 1.83-eV band after each heat treatment is plotted as a function of the temperature (Fig. 4). $[Li]^0$ centers began to appear at 1300 K and their concentration rapidly increased until 1500 K. The optical spectra after the heat treatments at three different temperatures are illustrated in Fig. 5. These spectra represent the difference in absorption before (as grown) and after each heat treatment. The negative absorption at the high-energy portion merely indicates that the absorption after annealing had decreased. It should be noted that the 1.83-eV band was generated thermally in air, without irradiations of any form.

B. Stability of [Li]⁰ centers

As we have shown, the [Li]^o center can be produced by three methods: (a) low-dose ionizing radiation at low temperatures, (b) high-dose electron irradiation, and (c) quenching from elevated temperatures. The second and thir methods yield [Li]^o centers which are indefiniely stable at room temperature. In order to deter line the relative stability of these centers, we performed isochronal annealing of 10 min each on samples in which [Li]⁰ centers were produced by these three ways. The results are shown in Fig. 6. Curve (a) illustrates the annealing behavior of the [Li]^o center in a crystal which was γ -irradiated at 77 K. These measurements were made by EPR. Curve (b) represents the annealing data of a crystal which was electron irradiated to a dose of $5 \times 10^{18} \ e/cm^2$. The optical absorption of the 1.83-eV band was used to determine the normalized ratio α/α_0 . This crystal was the same one whose absorption spectra were illustrated in Fig. 3. The sample was heated at the desired temperature in air and



FIG. 6. Annealing of [Li]⁰ centers, which were produced by (a) γ irradiated at 77 K, (b) electron irradiated at $5 \times 10^{18} e/\text{cm}^2$ at 290 K, and (c) thermally quenched from 1450 K (without subsequent irradiations).

quenched into a bath of liquid nitrogen. After the [Li]⁰ centers were annealed out $(\alpha/\alpha_0 \simeq 0)$, a short ionizing radiation with either γ rays or electrons at room temperature did not rejuvenate the [Li]^o centers. Curve (c) in Fig. 6 illustrates the stability of [Li]^o centers generated by thermal treatment. A sample, heated in air at 1420 K and quenched into a liquid-nitrogen bath, had an initial absorption coefficient of 40 cm⁻¹ at 1.83 eV. Annealing was performed in a horizontal quartz tube inserted into the furnace and exposed to air. After 10 min at the desired temperature the tube was removed. The heat capacity of the tube resulted in a slower cooling than if the crystal was quenched directly. The result was a monotonic decrease in absorption coefficient as a function of the annealing temperature [Fig. 6(c)]. The [Li]^o centers lost by annealing could not be regained by a short ionizing radiation at room temperature.

It is evident from Fig. 6 that the $[Li]^0$ centers, produced by the three different ways, have their own characteristic stabilities. The temperatures at which half of the defects have been annealed out for low-temperature irradiation, high-dose electron irradiation, and thermal quenching were ~210, 450, and 830 K, respectively.

Figure 7 shows the EPR spectra of an MgO:Li crystal before and after thermal treatments. In the as-grown state (a), the crystal contained no detectable $[\text{Li}]^0$ centers, but exhibited Cr^{3+} and Fe^{3+} signals. After quenching from 1500 K (b), a large concentration of $[\text{Li}]^0$ centers appeared, attended by "Li pair" signals. The Cr^{3+} vanished and the Fe^{3+} diminished. (The relative concentration of the Fe^{3+} is best gauged by the 57 Fe signal.) After an anneal at 850 K (c), the $[\text{Li}]^0$ center diminished in concentration and the original Cr^{3+} and Fe^{3+} signals recovered partially.

IV. DISCUSSION

We shall consider the various possibilities responsible for the stability of the $[Li]^0$ defect.

1. Hole release. It may be argued that holes leak off from the $[Li]^{\circ}$ centers, but that the holes are unable to find suitable trapping sites. In Fig. 7, we note that the production of the stable centers by quenching and irradiation was attended by the decrease of the Fe³⁺ and Cr³⁺ concentrations, and presumably the corresponding increase of Fe²⁺ and Cr²⁺ (and/or Fe⁺ and Cr⁺) impurities. A counter argument is that the Fe²⁺ and Cr²⁺ would be suitable trapping sites for the holes released from the $[Li]^{\circ}$ defects. Indeed, this is the situation for holes released from unstable $[Li]^{\circ}$ centers. We have observed the simultaneous decrease of the $[Li]^{\circ}$ signal and the increase of Fe³⁺ and Cr³⁺ con-



FIG. 7. EPR spectra of an MgO:Li crystal (a) as grown (b) thermally quenched from 1500 K, without subsequent irradiations, and (c) annealed at 850 K for 10 min.

centrations upon warming a γ -irradiated sample above 200 K. Additionally, the failure of a short ionizing radiation to rejuvenate the stable [Li]⁰ defects, after they have been annealed out, casts serious doubt that the annihilation of the stable [Li]⁰ defects is due to an electronic process. If the annihilation of the stable center is due to the thermal release of the hole, then a short ionizing irradiation at room temperature should rejuvenate the stable [Li]⁰ defect. Therefore, it appears highly improbable that the stability of the defect is due to the lack of suitable capture sites for the hole.

2. Electron capture. The notion that the trapped-hole defects may decay by electron capture has received no support in the past.³ First, it is unlikely that electrons from divalent impuri-

ties, such as Fe^{2+} and Cr^{2+} , could be thermally excited into the conduction band and proceed to recombine with holes at trapped-hole sites. Secondly, the monotonic dependence of thermal stability of the entire c

combine with holes at trapped-hole sites. Secondly, the monotonic dependence of thermal stability on the O⁻ to aliovalent-impurity distances in the neutral defects, as noted in the introduction section and Fig. 1, implies hole transport as the primary mechanism. Thirdly, as noted previously, the failure of a gamma irradiation to rejuvenate the stable defects after annealing tends to rule out an electronic transfer mechanism. Therefore, thermal decay of trapped holes by electron capture also cannot be considered seriously.

3. Ionic mobility. One is led to deduce, therefore, that the production and decay of the stable [Li]^o centers are the result of ionic mobility. A cross section of $\sim 10^5$ for [Li]⁰ production by electron irradiation is several orders of magnitude too small to be associated with the process of populating existing [Li]⁻ sites with holes. On the other hand, this value is far too large to be associated with a knock-on displacement of O^{2-} , Mg^{2+} , or Li⁺ ions. (The experimental cross section for oxygen displacement is 1.7 b,²⁶ while the theoretical value for oxygen and magnesium displacements²⁷ is about 10 b for a displacement threshold energy²⁸ of T_d = 60 eV. The theoretical cross section may be regarded as corresponding to an idealized situation, in which no interstitial-vacancy recombination occur.) Furthermore, the strong temperature dependence of the cross section for the stable [Li]^o formation, being larger at higher temperature (Fig. 2), appears to be incompatible with a knockon mechanism, since the displacement process is normally less efficient at higher temperatures due to the increased probability of interstitial-vacancy recombination. Indeed, the magnitude and temperature dependence of the cross section suggest a radiation-induced displacement, and/or diffusion (RID) process, analogous to that of hydrogen and deuterium in MgO.25

The strongest evidence that the stable [Li]^o center involves the diffusion of lithium ions lies in the experimental observation that similar thermal quenching or heavy electron irradiation does not generate stable impurity-compensated trappedhole centers other than the $[Li]^{\circ}$. Attempts to produce a stable V_{OH} , V_F , V_{A1} or $[Na]^0$ center were unsuccessful, indicating that the stability is characteristic of the lithium ion only. Also, another interesting note is that the stable [Li]⁰ centers. produced thermally or by electron irradiation are even more stable than the intrinsic V^- centers. In principle, the V^- center, being an uncompensated vacancy, should possess the highest hole-binding energy of all trapped-hole defects! This is evident from the illustration in Fig. 1.

Therefore the stable $[Li]^{\circ}$ defect in MgO is an anomaly and appears to be inconsistent with previously held concepts of production and stability of the entire class of hole defects in the alkalineearth oxides. The formation and the stability of the thermally generated and high-dose ionizationinducéd $[Li]^{\circ}$ defects cannot be simply explained in terms of our present understanding of trappedhole defects.

V. POSSIBLE MODEL

A satisfactory model must be capable of describing, as its main features, the stabilization of the bound hole and the thermal creation of stable [Li]^o centers without any ionizing radiation. We propose that in slow-cooled or as-grown crystals, the lithium impurities are concentrated primarily in precipitates, with some soluble Li⁺ ions randomly distributed in the crystal. (The randomly distributed Li⁺ ions are responsible for the unstable [Li]^o centers following ionizing irradiation at low temperatures.) At high temperatures, or upon extensive electron irradiation, each precipitate gives rise to a localized lithium-rich environment (here referred to as a microgalaxy) surrounding the precipitate. The precipitate would, of course, diminish in size as a result. Within the microgalaxy of abundant substitutional Li⁺ ions, charge neutrality requires that the natural state of the impurity be a Li⁺ ion with a trapped hole, which is the neutral [Li]^o center. In a very dilute system, such as found in a slow-cooled MgO:Li crystal, charge neutrality within a given region can be maintained between the randomly distributed Li* ions and other impurities of comparable concentrations, such as Fe^{3+} . However, in a region of high concentration of isolated Li⁺ ions, such as in the microgalaxy, charge compensation with other impurities is impossible, since the concentration of impurities other than lithium is effectively nil. Therefore, the substitutional Li⁺ ion in the microgalaxy must itself maintain charge neutrality. The resulting [Li]^o centers are separated sufficiently far apart from one another that they exhibit identical electronic configuration as the unstable centers but close enough so that charge neutrality is maintained within the microgalaxy. Furthermore, leakage of a few holes from the [Li]⁰ centers will result in a corresponding negative charge in the microgalaxy, which will tend to prevent further leakage and result in stable $[Li]^{\circ}$ centers.

Figure 8 is a schematic illustration of our model. A lithium precipitate in a slow-cooled, or asreceived, crystal is shown on the left (a). The two dots serve to illustrate the soluble, randomly scattered Li^{*} ions in the MgO matrix; these ions,



FIG. 8. Graphical display of the proposed model to explain the highly stable $[Li]^0$ centers. Slow cooling, such as in as grown crystals, results in a large aggregate of lithium impurities (a). At high temperatures, substitutional lithium ions are dispersed around the aggregate, forming a microgalaxy of $[Li]^0$ centers (b).

when irradiated at low temperatures, correspond to the unstable $[Li]^{\circ}$ centers. Figure 8(b) portrays the effect of heat-treatment of the crystal at high temperature, followed by a rapid quench. The precipitate diminishes in size and is now attended by a microgalaxy containing a concentrated environment of isolated Li⁺ ions; these are the stable $[Li]^{\circ}$ centers. A roughly similar scheme may be proposed for the effect of heavy doses of ionizing radiation. Lithium ions from the precipitate would migrate by radiation-induced diffusion²⁵ to replace Mg⁺² sites in the neighborhood.

VI. SUMMARY

A previous study using EPR and ENDOR measurements indicated that stable [Li]⁰ centers can be created by thermal quenching and extensive electron irradiation and that these defects have identical local electronic configuration as the unstable $[Li]^{0}$ center created by ionizing irradiation at low temperatures.²³ Stabilization resulting from an impurity charge compensation by an isotope of zero nuclear spin, which would escape EPR and ENDOR detection, is considered improbable since a motionally averaged spectrum of the g_{\parallel} and g_{\perp} components for the stable $[Li]^{0}$ system was observed near room temperature. Failure of an electronic-transfer mechanism, either by hole release or electron capture, cannot adequately account for the stabilization of the hole at the $[Li]^{0}$ center. The present study demonstrates that migration of lithium ions is involved in the stability of the $[Li]^{0}$ center.

A model is proposed whereby, in the as-grown state, the lithium impurities are considered to be present primarily in precipitates either metallic or in oxide forms, such as Li₂O. Subjected to high temperature or extensive electron irradiation, each precipitate would generate a microgalaxy of substitutional lithium ions surrounding the precipitate. Charge neutrality within the microgalaxy requires that the lithium ions are predominantly in the [Li]⁰ state. Hence the size of each precipitate would diminish under these conditions. A system in which the Li⁺ ions are concentrated in localized regions can induce cooperative phenomena. Therefore a search for such effects and studies by transmission electron microscopy (TEM) on possible lithium precipitates can be useful in determining the validity of this model. Preliminary TEM results in this laboratory revealed that precipitates were found in as-grown, or slowcooled, Li-doped MgO. Upon quenching from high temperatures, the precipitates were found to decrease in size. The result of this study will be reported.29

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