# Radiation Defects Manipulation by Ultrasound in Ionic Crystals

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*Abstract*—The radiation-induced optical absorption in ionic crystals is remarkably removed by a room-temperature ultrasonic treatment of the crystals. It is shown that the effect can be explained by defect migration processes occurring in ultrasonic fields. The experimental results display features consistent with a recent theory of the quantum diffusion. This new method of a cold annealing of radiation defects in solids can be termed as ultrasonic defect manipulation.

*Index Terms*—Acoustic applications, radiation effects, crystals.

## I. Introduction

EXPERIMENTAL and theoretical investigations of the properties of materials exposed to ionizing and nuclear radiation remains to be an active area of research and development. Among numerous studies done, one can mention radiation-related changes in optical properties of ionic crystals [1]–[3] and II–VI compounds [4], and radiation-induced generation of point defects in semiconductors [5]–[7] which can substantially affect the electrical characteristics of silicon-based detectors [8]. These substances are widely used as scintillator materials in detector and calorimeter systems for the purposes of high-energy physics and medicine. In these applications, they are exposed to nuclear radiation of different origin, resulting in an increased concentration of point defects and their agglomerates. The defect generation leads to the degradation of material properties and device performance [9].

In ionic crystals, different types of color centers are generated by the high-energy radiation. In particular, they cause the deterioration of transmission characteristics of scintillators due to the appearance of absorption bands and the overall decrease in optical transmission. Therefore, the developments of the techniques able to remove the induced absorption are of significant importance. Meanwhile, there are no too many possibilities to do that. Usually, the induced absorption is removed by a thermal annealing at temperatures ranged from 100 to 900 °C, depending on the crystal used.

An attractive alternative route can offer a defect manipulation approach, sometimes named as "defect engineering" [10]–[13]. For example, this respectively new method was successfully applied in order to quench an excessive concentration of dopants, mainly oxygen in silicon [10].

This work aims to demonstrate that the optical absorption in ionic crystals induced by X-rays and $\gamma$-irradiation can be removed by the ultrasonic defect manipulation (UDM). The idea of the UDM approach consists in the ultrasound-mediated activation of the defect diffusion, altering the concentration and the space location of point defects in a given crystal volume including the near surface region of the crystal.

It is therefore the purpose of this work to achieve cold annealing of radiation defects without the sample heating. This would have particular relevance to detector technologies and performance of calorimetric systems. In many cases, when the device is a rather complicated system consisting of a crystal on a chip, bounding wiring, etc., the high temperature can destroy some elements of the structure. Then the UDM is thought to be applicable as the ultrasound source can be integrated into the multicomponent detectors or detector array systems. However, the construction of the real integrated detector would be a subject of a separate research and development work.

## II. UDM Experiment

Single crystals of CsI(Tl), CsI(Na), and KBr are used in experiments. Acoustic waves in the megahertz frequency range are employed to cure the samples, which were preliminary exposed to X-rays or $\gamma$- irradiation. All experiments are done at room temperature. Nevertheless, during the ultrasonic load, the sample temperature is slightly elevated. Thus, in these studies, the temperature rise as high as $\approx 30$ K is detected by a Cu-Constantan thermocouple directly attached to the sample surface. However, it should be noted that, in this particular case, such a partial conversion of the ultrasonic energy to the heat can be useful effect able to activate the point defect migration process. It is also worth noting that the temperatures below $\approx 550$ K can be defined as low temperatures for defect annealing.

The experimental setup is shown in Fig. 1. Piezoceramic (PZT) ultrasonic transducers 1 and 3 are bounded to the sample 2. A MHz-frequency $(f)$ rf-voltage with the amplitude value of $V_{RF}$ is applied to the input transducer 1, which converts this voltage into the ultrasonic wave. By measuring an output rf-voltage $(V_{OUT})$, one can find an optimum frequency for maximum acoustic amplitude inside the sample 2; see Fig. 2. The data shown below are taken at $f \approx 2$ MHz corresponding to fundamental resonant frequency of the transducer along its thickness. The wavelength of the ultrasonic wave ($\approx 1.2$ mm in CsI and $\approx 1.8$ mm in KBr) is significantly shorter than the sample length ($\geq 12$ mm). In addition, the opposite faces of the samples are not free since the ultrasonic transducers are attached. Consequently, in our experiments, the wave transmission regime rather than the standing wave regime is
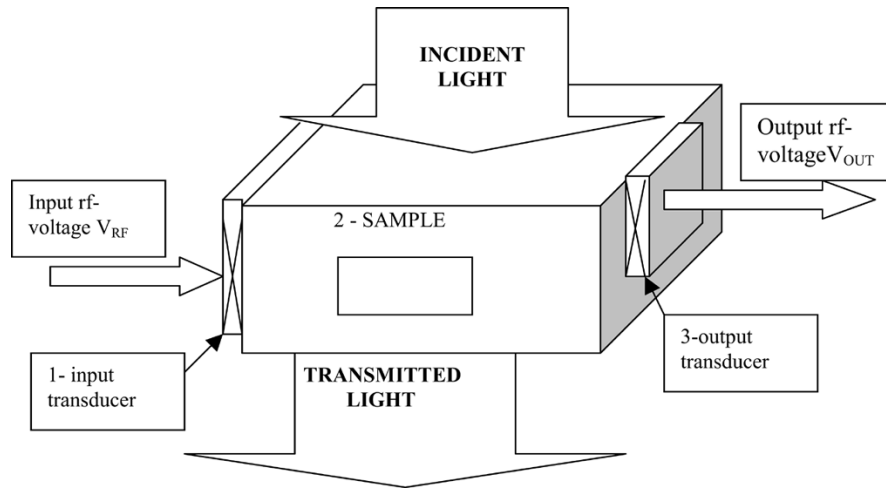
Fig. 1. Experimental setup of the UDM and optical measurements: 1- input piezo-ceramic transducer exciting acoustic waves in a sample 2, 3—output piezo-ceramic transducer.
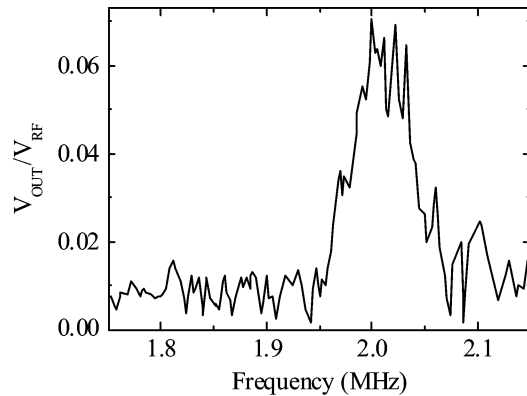


Fig. 2. Output voltage $V_{OUT}$ versus frequency at constant input voltage $V_{RF}$. Acoustic amplitude is maximum at frequency 2 MHz.
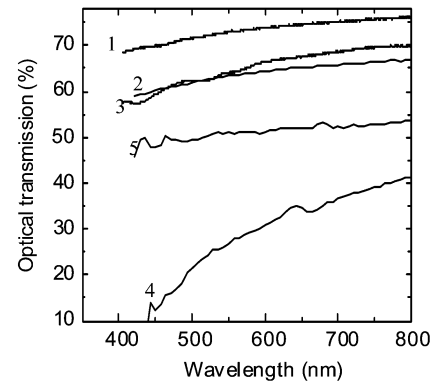


Fig. 3. Influence of X-ray irradiation and UDM on optical transmittance in CsI(Tl) (1, 3) and CsI(Na) (2, 4, 5): 1, 2—before X-ray irradiation; 3, 4—after X-ray irradiation at a dose of $\approx 4$ krad (no UDM); 5—after UDM of irradiated sample ($f = 2.04$ MHz, $V_{RF} = 14.8$ Volts). Time of UDM is 2.5 hrs. Irradiation: Ag-$K_\alpha$, 25 KeV.

implemented. It has been found that there is a clear correlation between the frequency dependence of the effect discussed in this work and that of the transducer vibration amplitude displayed in Fig. 2.

To detect the changes in the concentration of point defects, the evolution of the optical transmission spectrum is studied. This is done with Perkin Elmer UV/VIS Lambda 18 spectrometer (CsI crystals), and a single beam DMR-4 monochromator in case of KBr samples.

Optical transmission spectra are taken before radiation treatments and after irradiation of the samples, during ultrasound load, and finally after acoustic wave are removed from the samples. The samples of KBr and CsI(Na) are from the same vendor (Eastern Europe), and CsI(Tl) samples are made from the original material used in SLAC (Stanford Linear Accelerator) for the crystal detectors of nuclear radiation. All the samples have the form of a parallelepiped and are cut out from a massive piece. The dimensions of KBr and CsI(Na) are about 5 by 5 mm$^2$ for end faces, and their length is about 10–12 mm. CsI(Tl) samples are bit larger, 25 mm in diameter and 38 mm long. The crystals have been exposed to X- or $\gamma$- irradiation with the doses ranged from 0.1 to 10 krad, which allows to visibly detecting

the presence of color centers. Before UDM, the samples are well colored, slightly brown in case of CsI and dark-blue in case of KBr.

## III. EXPERIMENTAL RESULTS

The optical transmission spectra taken in CsI(Tl) and CsI(Na) samples are shown in Fig. 3. Spectra 1 and 2 display initial transmittance, whereas spectra 3 and 4 illustrate the deterioration of the transmittance after X-ray irradiation. By numerous investigations, it has been found that the optical transmittance drops, and only $1/3$ to $1/2$ of the initial transmission of light is observed with the irradiation doses used. Another effect is the transmission band-edge shift which is most evident by comparing spectra 2 and 4 in Fig. 3. Both the reduction of the light transmission and the low-energy shift are typical of ionizing radiation which is known to produce color centers with wide absorption bands [14], [15]. It is worth noting that the efficiency of the radiation-induced coloring strongly depends on the alkali halide crystal used as well as on the nature of the impurity complexes present in the crystal. In particular, an enhanced

dipole-aggregation rate and decreased concentration of impurity-vacancy dipoles have been reported to occur in doped alkali halides owing to room-temperature X-ray irradiation [3]. The oxygen contamination has been observed to influence the radiation damage in CsI crystals [16]. Taken together, these facts may explain the differences in the radiation effects on the light transmission in CsI(Tl) and CsI(Na) crystals seen in spectra 3 and 4 of Fig. 3, respectively. These would furthermore explain the absence of well resolved spectral bands in plot 4 of Fig. 3, which can be related to the light absorption by color centers.

Indeed, the transmission coefficient is

$$T = [(1 - R)^2 \exp(-\alpha d)]/[1 - R^2 \exp(-2\alpha d)] \quad (1)$$

where $\alpha$ is the absorption coefficient, $d$ is the sample thickness, and $R$ is the reflection coefficient given by

$$R = [(n - 1)/(n + 1)]^2 \quad (2)$$

where $n$ is the index of refraction. Taking $n \approx 1.8$ in the wavelength range from 500 to 800 nm one gets $R = 0.08$. Implicitly assuming $R$ remains unchanged by the radiation; we find $\alpha \approx 0.12$ and 1.45 cm$^{-1}$ at ~500 nm for spectra 3 and 4 in Fig. 3, respectively. It is therefore likely to expect the appearance of resolved bands in the transmission spectra of the irradiated samples since such a value of $\alpha$ is known to produce a well-resolved absorption band in alkali halides [17].

One can suggest that the irradiation may cause an enhanced surface roughness and, to some extent, the observed transmission drop is due to the light scattering at the sample surfaces. However, even though the trend of decreasing transparency with increasing $R$ is clear, several considerations argue strongly against the transmission drop being purely surface in origin. First, a considerable amount of previous experience has been shown that the deterioration of optical transmission of CsI(Tl) and CsI(Na) materials is due to volume coloring (see [1] and references therein). Second, the transmission spectrum of the sample irradiated with X-rays at a lower dose shown in Fig. 4 exhibits well-resolved absorption bands which are marked by arrows. Significantly, all the 430-, 520- and 560-nm bands have been reported to be a family of F-center absorption bands [1], [18], [19]. Most important is the fact that the 840-nm absorption band clearly resolved in Fig. 4 has been attributed to the F-aggregate centers which absorb at the long-wavelength side of an F-center band [18]. That supports the conclusion that the volume coloring is indeed observed in CsI crystals irradiated with X-rays. Furthermore, defect aggregation likely to occur in our samples. Third, previous works illustrated the acoustically-affected crystal properties, including those revealed on the surface, mainly originated from the interaction of ultrasound with the lattice defects that are located deep inside the sample [20], [21]. Therefore, a guess that presented in this work effect of ultrasound on radiation defects is purely a surface phenomenon seems unlikely.

Applying UDM, we are able to remarkably remove the radiation-induced absorption. The level of the effect depends on the UDM parameters, as will be discussed below. In CsI, the
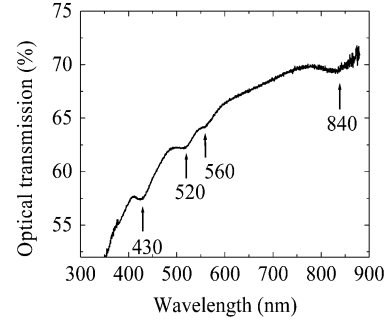


Fig. 4. Enlarged transmission of X-ray irradiated CsI(Tl) in the transparency range llustrating the occurrence of F-centres absorption bands (details are in the text). Radiation dose is of the same order of magnitude as in Fig. 3.

maximum effect is illustrated by spectrum 5 in Fig. 3. It is seen that about 60–70% of the radiation-induced absorption can be removed by UDM. The rest of the lost transmission which amounts to 30–40% in Fig. 3 cannot be removed by UDM. It is likely that this is due in part to defect aggregates that appear to be unaffected by ultrasound in the frequency and intensity range employed in this work, and probably in part to some theoretically possible enhanced surface nano-roughness imposed by X-rays that we can not detect through optical microscope.

To perform a quantitative analysis, one should take into account the fact that the effects of ionizing radiation in doped alkali halides depend sensitively on the state of the impurity ions at the start of the irradiation process. Moreover, this has been found to be of particular importance when studying the kinetics of radiation-induced coloring in alkali halides (see, for example, [3] and references therein). Given that special emphasis should be made on the processes of impurity precipitation due to irradiation and their interplay with the color center production, it is furthermore expected that these would influence the defect annealing during UDM. Therefore, it appears that more correct account for the influence of UDM on the concentration of color centers in an initially pure crystal lattice can much better be done in undoped alkali halides. In this work, we used $\gamma$–irradiated KBr crystals.

Absorption spectrum (1 in Fig. 5) exhibits a well-known F band in KBr peaked at about 630 nm [17]. The band is quenched by ultrasound (spectrum 2 in Fig. 5). Using Smakula's equation [17] applicable to an approximately Gaussian absorption band, the F-center concentration is

$$N_F(cm^{-3}) = 8.7 \cdot 10^{16} \cdot n \cdot \alpha_m(cm^{-1}) \cdot H(eV)/(n^2 + 2)^2 \cdot f_0 \quad (3)$$

where $f_0$ is the oscillator strength of the transition with the F center, $\alpha_m$ is the peak absorption coefficient, and $H$ is the absorption band half-width. Taking $f_0 = 0.75$ [17], we found the amplitude and time dependencies of $N_F$ at UDM, which are shown in Fig. 6 by plots (a) and (b), respectively. It is seen in panel (a) that small changes in the F-center concentration are only observed below $V_{RF} \approx 24$ Volts whereas the crystal coloration bleaches out very rapidly above this threshold value of applied voltage. Also, at a fixed $V_{RF}$ above the threshold, the
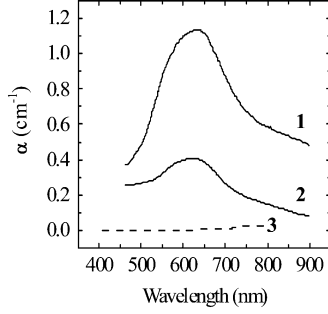
Fig. 5. UDM quenching of 650 nm F-center absorption band in $\gamma$–irradiated KBr at a dose of 10 krad. Plot 1—initial before UDM; plot 2—after UDM by ultrasound of 2.16 MHz, $V_{\mathrm{RF}} = 25$ Volts. Plots 1 and 2 are calculated from experimentally obtained transmission spectra by using (1) and (2) at $n = 1.55$. For comparison, plot 3 shows water absorption coefficient from a table in [37], [44].



Fig. 6. Change in the concentration of F-centers in KBr with $V_{\mathrm{RF}}$ after 30 minutes UDM [plot (a)], and with time of UDM at $V_{\mathrm{RF}} = 25$ Volts [plot (b)].

rate of F-center destruction becomes increasingly smaller with time, as illustrated in panel (b) of Fig. 6.

## IV. DISCUSSION

The experimental results presented in Figs. 3–6 illustrate the annealing effect of ultrasound on the radiation defects in ionic crystals. In order to understand the likely origin of the observed decrease in the defect concentration due to UDM, the threshold behavior of the effect should be discussed in more details.

Speaking qualitatively, when the interstitial ion goes to its native site in one half cycle of acoustic wave, it still remains at its regular site in the next opposite half cycle of acoustic wave. This is due to the fact that the ultrasonic energy is much less than the nuclear radiation energy. Therefore, the ultrasonic energy is not large enough to remove the atom from its regular site, whereas it is thought to be enough to promote the atom to move back from the interstitial position. As the energy barrier is known to exist between the interstitial and the regular site, it would determine the threshold acoustic energy (and the threshold voltage $V_{\mathrm{RF}}$) for the UDM effect. Significantly, the fact that the energy needed to displace the atom/ion residing in the regular site to nearby interstitial positions exceeds the one for the backward motion is indeed well-documented [22].

Taking a simple quantitative estimate, we first remark that the UDM annealing can facilitate an overheat of a local crystal volume during the compression cycle of ultrasonic load, as evidenced, for instance, by sonoluminescence experiments [23]. In terms of the heat flux $\Phi$ flowing from warm sample areas to cooler ones [24]

$$\Phi \approx (k \cdot \Delta T / d) \qquad (4)$$

where $k$ is the thermal conductivity, $d$ is the heat diffusion distance, and $\Delta T$ is the temperature variation. To take a rough estimate of $\Delta T$, suppose now that the temperature varies due to acoustic power flow $W$, and a $Q^{-1}$ part of the flow is absorbed by the lattice, with $Q$ being the mechanical quality factor of our samples. Combing this with (4) we obtain

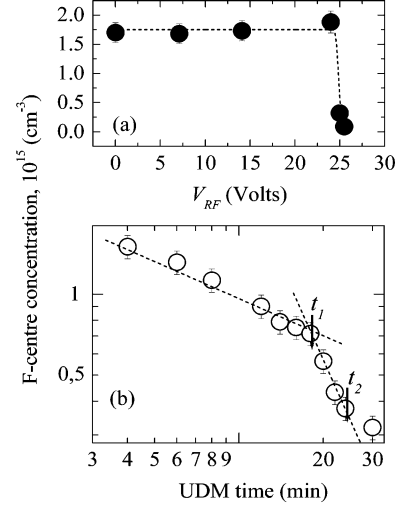$$\Delta T \approx (W \cdot d / Q \cdot k). \qquad (5)$$

The threshold value $W_{\mathrm{th}}$ achieved at $V_{RF} \approx 25$ V in Fig. 6(a) is found to be $\sim$5 W/cm$^2$ and the $Q$ factor is of the order of 200, as obtained by the resonance-antiresonance method [25]. With $k = 1.1$ J/m·K·s [26] and $d = 38$ mm in CsI, we obtain $\Delta T \approx 9$ K. Considering the additional contribution of the heat flux from the input transducer (1 in Fig. 1) this value is enhanced, exhibiting the agreement with the experimentally detected $\Delta T \approx 30$ K to be fairly good. This consideration is only valid in a perfect lattice. Taking into account lattice defects locally decreases $Q$ giving rise enhanced $\Delta T$ in (5). In the worst case $(Q = 1)$ we find $\Delta T \approx 1700$ K, which amounts to the greatest possible value of the overheat of a local crystal volume. Next, the escape time of the heat flux is given by [24]

$$\tau \approx (\mathrm{d}^2 \cdot \rho \cdot C / k) \qquad (6)$$

where $\rho$ is the density and $C$ is the heat capacity. Taking $\rho = 4.5 \times 10^3$ kg/m$^3$ and $C \approx 100$ J/kg·K in CsI [27], we find $\tau \approx 600$ s. With $\rho = 2.75 \times 10^3$ kg/m$^3$, $\kappa = 5$ J/m·K· s, $C \approx 400$ J/kg· K [27], and $d = 10$ mm in KBr we obtain $\tau \approx 25$ s. Hence, the escape time and the likely overheat are estimated to be large enough to facilitate the defect migration process.

However, if UDM is perceived as a purely thermal process, then it is difficult to explain the data of Fig. 5(a). The decrease in the F centres concentration above the threshold $V_{\mathrm{RF}}$ appears to be unexpectedly fast, and this is vital to the UDM model to be discussed. We thus conclude that, although the thermal effect likely contributes to UDM, there probably exists another accompanying process. Turning next to identifying the process involved, we may stress that ultrasonic load in ionic crystals has been shown to produce electric fields of considerable strengths due to dislocations which move in ultrasonic fields [20]. The moving dislocations, in turn, are shown to drag the point defects [28], which may decrease the concentration of color centers. The local heating can facilitate dislocation vibrations.

Developing a viable explanation of the ion diffusion process due to UDM it may be suggested that ultrasound can act through

the electronic subsystem. This is the key to previous findings explaining the thermal annealing and optical bleaching of F centers by a two-step process. It involves the excitation of electron from the center followed by ion diffusion [29]. Therefore, implicit in our model is that elevated local temperature and large electric field periodically kick F center thus providing an attachment of ultrasonic energy flux to the defect system with complex surroundings. The dynamics of this mixed system, which can be separated into the heavy, slowly changing classical system of vacancy-interstitial ion pairs and the fast quantum mechanical system of electrons moving in a complex potential, would then determine the amplitude dependence and the kinetics of UDM. The quantum mechanical system influences the diffusion properties of the classical system, and this is frequently termed as quantum diffusion [30], [31].

It is not the objective of this study to give a cohesive picture of the underlying mechanism in UDM. However, several considerations support the idea that the physical process giving rises to UDM effect likely to be a quantum diffusion stimulated by ultrasound. First, experimentally it appears that applying UDM at $V_{\mathrm{RF}}$ just slightly above the threshold value produces a catastrophic collapse of the F centers. This is illustrated by a dramatic decrease in $N_F$ at $V_{\mathrm{RF}} \approx 25$ V seen in Fig. 6(a). This type of a threshold behavior is discussed to be characteristic of deformation-induced catastrophic phenomena [32], which may lead to a very rapid decreasing in activation energy under external deformation displacing an ion from its initial location. A phononassisted catastrophic lattice relaxation is described in [33]. Second, the system of vacancy-interstitial ion pairs coupled to the system of electrons separated from F centers would respond to an underlying dynamical behavior of the electron system. Recent computations of the quantum diffusion in complex surroundings with interaction of a heavy system coupled to a quantum mechanical light system [30] reveal three time scales in the fast system dynamics. Following [30], the resulting diffusive spread of the energy varies with time $t$ as $Dt^{\eta}$, where D is the "diffusion constant," and the potency $\eta$ is given by the slope of this time dependence in a log-log scale. Theoretically, the diffusive behavior of a heavy system strongly depends on the complexity of a light system, resulting in a few time scales $(\eta)$ observed in the time dependence of the energy diffusion. Noticeably, the energy diffusion saturates at a great time instant. Quite remarkably, this type of the behavior is observed experimentally. It is seen in Fig. 6(b) that the time variation of $N_F$ exhibits two nearly straight lines with different slopes (at $t < t_1$ and $t_1 < t < t_2$), and eventually saturates after some larger time value $(t_2)$.

As such, we believe that the quantum diffusion model gives the correct picture of the UDM in KBr. The data obtained are seemingly in accord with the results of elaborate theoretical treatments. However, since our present model is still crude, previously proposed mechanisms of acoustically activated diffusion processes in semiconductors will need to be considered in developing a more elaborated model of UDM. Among them: (1) Defect pair reorientation by resonantly absorbed ultrasound giving rise to the pair dissociation [34]. (2) Ultrasonically activated charge transfer between point defects [35], leading to atom migration [36].

Finally, we discuss potential contribution of the surface effects into UDM. Main sources of errors may be associated with moisture and roughness that can spoil the sample surfaces. Comparison of water absorption coefficient (plot 3 in Fig. 5) [37], [44] with our data on UDM (plots 1, 2 in Fig. 5 and plots 4, 5 in Fig. 3) leads to a conclusion that water or moisture on crystal surface can not give the observed changes in the spectra after UDM. For instance, water absorption coefficient is small and flat in comparison with the color center bands detected from our samples in Figs. 3, 4 and 5. This proves that UDM can not be explained by moisture effects. In addition, initial samples were optically polished, and surfaces were clean and free of moisture. Before UDM the samples were carefully examined, and they were without any surface damages that can be detected by optical microscope. A quality of the samples is confirmed by the experimentally measured optical absorption coefficients, which agree with previously published data [3], [36], [37], [44], [38]–[41]. For example, plot 2 of Fig. 5 (KBr after UDM) gives $\alpha = 0.1$ cm$^{-1}$ at 800 nm, and $\alpha = 0.27$ cm$^{-1}$ at 500 nm; the last coincides with classical measurements for KBr [38]. In different colored alkali halide crystals, the absorption coefficient $(\alpha)$ is measured to be from 0.75 cm$^{-1}$ to as high as 2500 cm$^{-1}$ in prolonged X-irradiated KBr [3], [38]–[43]. Note, in these publications, the sample dimensions are mainly about the same as we used. Consequently our measurements of $\alpha$ from irradiated samples are in agreement with known data. Another question is can ultrasound remove the surface spoiling factors like moisture or roughness that could arise during the measurements? It is not likely to occur since we optically examine the samples three times, before irradiation, after coloring or before UDM, and after UDM. The samples remained of the same quality.

## V. Conclusion

It is shown for the first time that the radiation-induced optical absorption in ionic crystals can be remarkably removed by ultrasonic treatment of the crystals at room temperature. This new method of a cold annealing of radiation defects in solids can be termed as UDM. The effect of UDM can be used for particle detector systems to decrease or cure a radiation damage of crystal scintillators.

The data suggest that UDM involves defect migration in ultrasonic fields. Experimental results indicate that the UDM effect is not the result of a purely thermal process. Many potentially relevant factors including ultrasound-mediated inter-ion charge exchange and tunneling effects seem so far to be the less important parameters. The data are consistent in several aspects with a recent model of the quantum diffusion.

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