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J. Tatarkiewicz, A. Król, A. Breitschwerdt, and J. Dobrowolski

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# Optical absorption spectroscopy of hydrogen- and deutron-implemented cadmium fluoride

J. Tatarkiewicz<sup>a)</sup>

Max Planck Institut für Festkörperforschung, Heisenbergstrasse 1, D-7000 Stuttgart 80,  
Federal Republic of Germany

A. Król

Institute of Experimental Physics, Warsaw University, ul. Hoza 69, 00-681 Warszawa, Poland

A. Breitschwerdt

Max Planck Institut für Festkörperforschung, Heisenbergstrasse 1, D-7000 Stuttgart 80,  
Federal Republic of Germany

J. Dobrowolski

Research Institute for Industrial Chemistry, ul. Rydygiera 8, 01-793 Warszawa, Poland

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A full-range (visible and infrared) study of the optical properties of  $\text{CdF}_2$  implanted with hydrogen and deuterium is presented. One strong absorption band was found at 280 nm (4.2 eV), whose intensity is proportional to the radiation damage introduced. Oxidation of  $\text{CdF}_2$  produces a similar absorption band, which suggests that the electronic transition takes place in an extended defect containing a cadmium vacancy. In the infrared region implanted samples exhibit two bands at about 3200 and 1600  $\text{cm}^{-1}$ . We assign these modes to water molecules vibrating on the surface. Measurements at low temperatures (after polishing) revealed weak localized vibrational modes of hydrogen and deuterium at 1550 and 1160  $\text{cm}^{-1}$ , respectively. Because of their temperature behavior we assign these lines to the second harmonics of implanted ion vibrations. Calculated oscillator strengths correspond to an effective charge of about  $0.5e_0$  and show decreasing values for higher radiation damage.

## I. INTRODUCTION

Although the optical properties of fluorites have been intensively studied during the last 30 years (for a review see the monograph edited by Hayes),<sup>1</sup> cadmium fluoride received considerably less attention. On the other hand  $\text{CdF}_2$  is the only fluorite which can be rendered semiconducting by doping with trivalent rare-earth ions.<sup>2</sup> This has motivated quite a number of optical investigations of the material (for a review see Ref. 3). However, none of them reported observation of hydrogen vibrations, because the standard procedure of introducing hydrogen into fluorites (cf. Ref. 4) converts  $\text{CdF}_2$  into a semiconductor which shows strong IR absorption due to the photoionization of donors.<sup>5</sup> Recently, hydrogen has been used to passivate various defects in a variety of semiconductors and its presence has also been detected by optical absorption (for a review see Ref. 6). One can ask if it is possible to introduce into  $\text{CdF}_2$  sufficiently high concentrations of hydrogen to be able to detect it optically. In this paper we positively answer the above question by implantational doping and low-temperature IR measurements. Optical phenomena accompanying implantations were also studied, showing that due to the hygroscopic properties of cadmium fluoride<sup>7</sup> large quantities of water can be adsorbed on its surfaces. Irradiation introduces optical centers, which change the color of implanted samples into a brownish one (undoped  $\text{CdF}_2$  is transparent in visible light). Thus, one can see the results of the implantation with the naked eye,

which is not possible for many other semiconductors. This fact gives us an instantaneous method for determination of the quality of irradiation (implanted area and its homogeneity). In the case of ionic materials, which are poor heat conductors, this is important because it is very easy to burn the surfaces of these materials during irradiation (for a description of problems encountered during implantation of ionic crystals, the reader is referred to the recent review in Ref. 8). Preliminary results on hydrogen vibrations in  $\text{CdF}_2$  have already been presented<sup>9</sup> and compared with other proton-implanted semiconductors. We found some trends in hydrogen vibrational energies as a function of crystal ionicity (see Ref. 10). This study gives additional information on *how* hydrogen is bonded (i.e., what is the energy of its vibrations) in highly ionic semiconductors. Still, there is a question of *where* this hydrogen is bonded, but optical measurements alone cannot answer it. For  $\text{CdF}_2$ , which is a very soft material, uniaxial stress experiments are hard to perform. EPR measurements, which usually are helpful in providing information about defects, are in this case inconclusive (too many lines from radiation defects<sup>11</sup>). Hence, our knowledge of defects in  $\text{CdF}_2$  containing hydrogen is rather limited. In order to further ascertain that the observed localized vibrations are really due to hydrogen, we also performed deutron implantations. Because of the well-known isotope shift<sup>4</sup> (by the factor of  $\approx\sqrt{2}$ ) one can easily identify hydrogen-localized vibrational modes (LVMS) in the material studied. In this paper absorption spectroscopy [in the visible (VIS) and infrared (IR) regions] is used to study proton radiation effects in  $\text{CdF}_2$ . Raman scattering experiments have already been

<sup>a)</sup> Permanent address: Institute of Physics, Polish Academy of Sciences, al. Lotników 32/46, 02-668 Warszawa, Poland.

reported.<sup>12</sup> We found no Raman lines directly connected with hydrogen or deuterium in irradiated CdF<sub>2</sub>.

## II. EXPERIMENT

Samples with a thickness of about 2 mm were cut from the same single crystal of nominally pure CdF<sub>2</sub>, polished on both sides and implanted with 2-MeV protons or deuterons in the Warsaw van de Graaff accelerator. Implantation currents were varied between 0.1 and 1.0  $\mu\text{A cm}^{-2}$  for times between 20 and 100 h, giving total proton or deuterium doses of 1.8 and  $3.6 \times 10^{17} \text{ cm}^{-2}$ . Although the samples were glued with a silver paste onto the holder and the latter was water cooled, we found that it was very easy to get flocking of the implanted layer. This happens because the layer is highly strained.<sup>13</sup> Some of our samples were broken during implantation without apparent reason except for the above-mentioned stresses. Hence, we were unable to implant doses higher than about  $4 \times 10^{17} \text{ cm}^{-2}$ , which made our IR measurements much more complicated than in the case of other semiconductors.<sup>9</sup>

Optical measurements were performed using Varian Carry 17 (VIS region) and Perkin Elmer 580B (IR region) conventional spectrometers as well as Nicolet MX-1 Fourier transform spectrometer (FIR region). Standard Oxford Instruments model 100 flow-type cryostats with suitable windows (sapphire or KRS-5) were used for low-temperature measurements.

The range of 2-MeV protons in CdF<sub>2</sub> is about 30  $\mu\text{m}$  with a spread of about 5  $\mu\text{m}$  (Ref. 14) (slightly less for deuterons), so our doses correspond to hydrogen/deuterium concentrations of the order of 1 at. %.<sup>9</sup> These concentrations are comparable to those studied in other fluorites<sup>4</sup> although our surface concentrations are two orders of magnitude smaller, making detection of hydrogen vibrations relatively cumbersome.

## III. RESULTS AND DISCUSSION

### A. VIS region

As already mentioned, after irradiation, samples change their color. This is due to the tails of the strong absorption

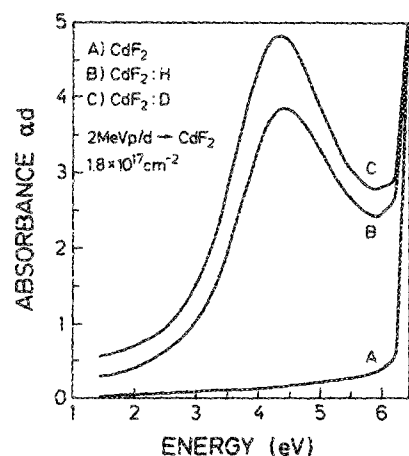


FIG. 1. Comparison of the absorption in the visible region of pure and proton- or deuteron-implanted cadmium fluoride. Room-temperature measurements.

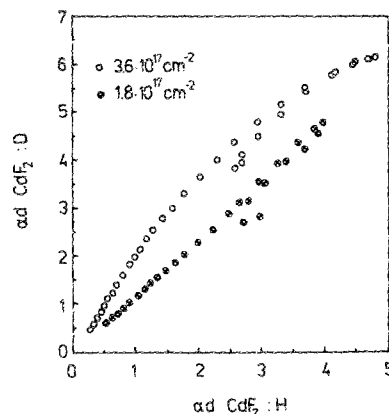


FIG. 2. Absorption of a deuteron-implanted CdF<sub>2</sub> sample plotted vs absorption of a proton-implanted one (each point denotes absorption for the same photon energies).

band at around 280 nm (4.2 eV). In Fig. 1 we present this band for proton- and deuteron-irradiated samples. That the band is radiation induced can be inferred from the fact that for the same ion doses it is stronger for deuterons which should produce roughly twice as many defects as protons.<sup>14</sup> The band, similar to other proton-irradiated semiconductors, appears on a slanting background<sup>9</sup> (the so-called near-edge band or NEB). This background also originates from radiation effects as shown by the linearity of the correlation plot in Fig. 2. For higher ion doses ( $3.6 \times 10^{17} \text{ cm}^{-2}$ ) the plot is curved because of the saturation of defect production ("on beam" annealing). Knowing that the NEB is exponential in energy<sup>15</sup> one can subtract it and obtain the shape of the absorption band at about 280 nm. Figure 3 shows that the band is Gaussian with a half-width of about 1 eV and a center at 4.2 eV. Low-temperature measurements have shown that this band shape does not change. Similar bands, observed in other fluorites after x-ray irradiation<sup>1</sup> are narrower, but protons introduce many more defects. Thus, the width of the band is probably due to inhomogeneous defect broadening. To estimate the density of these defects we use the oscillator strength<sup>16</sup> of the band at 280 nm. For the sample implanted with  $1.8 \times 10^{17} \text{ p/cm}^2$  we obtain an integrated intensity  $\int [\alpha(\omega)d/\omega]d\omega$  (where  $\alpha d$  is the absorbance) of about 0.86.

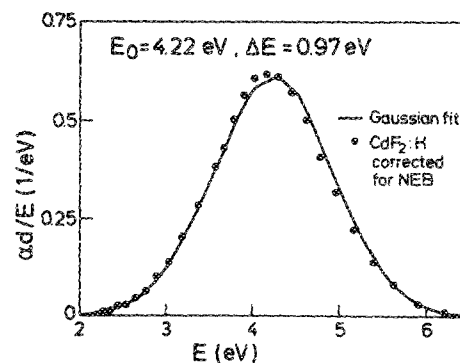


FIG. 3. Shape of the band at 280 nm (4.2 eV) after subtraction of the exponential background due to NEB. Normalization by energy taken into account.

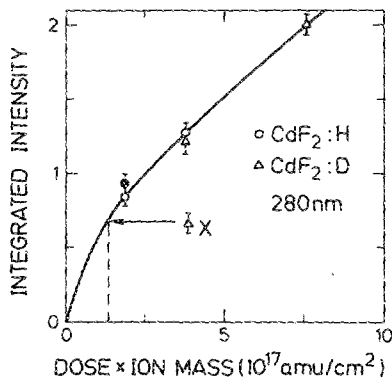


FIG. 4. Integrated intensity of the band at 280 nm (4.2 eV) as a function of radiation damage [ion mass in atomic mass units (amu) multiplied by implanted dose].  $\times$  denotes sample broken during irradiation. Full point denotes sample implanted with low protons current.

If one assumes that the oscillator strength is one, then the above value gives a density of centers higher than  $5 \times 10^{16}$  per  $\text{cm}^2$  or more than  $\frac{1}{4}$  center per incoming ion. These values seem to be reasonable—cf., density of divacancies in silicon after proton irradiation.<sup>17</sup> Hence, the intensity of this band can be used as an approximate measure of lattice damage. Figure 4 shows the nonlinearity of the integrated intensity due to the annealing of defects during irradiation. This effect has been extensively discussed in the case of covalent semiconductors<sup>18</sup> and is even more pronounced for ionic ones, which are very poor conductors of heat. However, an estimate of the radiation damage from the intensity of the band at 280 nm can be useful as is proved in the case of one of our samples, which was broken during implantation (Fig. 4, point marked with  $\times$ ). The integrated intensity for this sample indicates that the dose was roughly three times smaller than that expected from the time of irradiation. This fact has been confirmed by IR measurements (see the next section), where the sample exhibited only weak deuterium vibrational lines.

To determine the possible origins of the observed band at 280 nm (4.2 eV) we made additional measurements on an oxidized cadmium fluoride sample. Figure 5 shows the spectrum of the sample oxidized for 1 h at 200 °C in pure oxygen as compared with the spectrum of a deuterium-implanted sample. Except for the different background due to the scattering of light on surface irregularities both samples exhibit Gaussian bands in the same region between 4 and 5 eV, al-

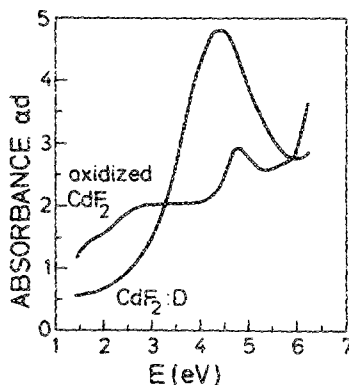


FIG. 5. Comparison of visible absorption of  $\text{CdF}_2$  implanted with deuterons and of the oxidized crystal.

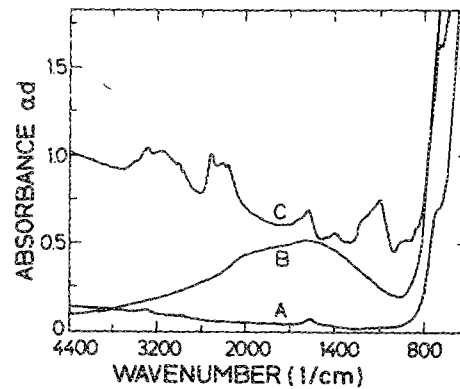


FIG. 6. Absorption spectra of: A— $\text{CdF}_2$  implanted with  $1.8 \times 10^{17}$  p/ $\text{cm}^2$ , B—oxidized  $\text{CdF}_2$ , C— $\text{CdF}_2$  kept over heavy water. Note change of the scale at  $2000 \text{ cm}^{-1}$ .

though the band for the deuterium-implanted sample is much broader. Oxidation of the surface of cadmium fluoride leads to the production of cadmium oxide. Hence, the surface layer of the crystal becomes cadmium deficient which means that by oxidation we create cadmium vacancies. Thus, the similarity of the observed optical bands suggests that the main radiation defects in proton- or deuterium-implanted  $\text{CdF}_2$  are cadmium vacancies. On the other hand it is well known that typical radiation defects in fluorites are F centers or fluorine vacancies.<sup>1</sup> It is quite possible that the observed band is due to electronic transitions in *both* types of defects. However, we were unable to see this band after 1.5-MeV electron irradiation<sup>19</sup> up to the doses of  $10^{18}$  electrons/ $\text{cm}^2$ . During irradiation, electrons produce only simple defects (vacancies and interstitials) which annihilate "on beam" at room temperature. This observation leads to the final conclusion that the band at 280 nm (4.2 eV) is due to electronic transitions in extended defects, containing many cadmium and fluorine vacancies.

## B. IR region

The most pronounced features in the infrared spectra of  $\text{CdF}_2$  implanted with proton bands appear at 1600 and 3200  $\text{cm}^{-1}$  (see Fig. 6, spectrum A). In the same figure we present the spectrum of our oxidized sample (spectrum B), which shows only one broadband at about 1500  $\text{cm}^{-1}$ , probably originating from vibrations of cadmium oxide on the surface. To verify the source of the two bands always seen after irradiation, one of our samples has been kept over heavy water  $\text{D}_2\text{O}$  for 5 months. Spectrum C in Fig. 6 shows two additional bands, shifted by the factor of about 1.4. This experiment suggests that the strong bands are due to surface vibrations of  $\text{H}_2\text{O}$  molecules (basic vibrational frequencies 1587 and 3178  $\text{cm}^{-1}$ ).<sup>20</sup> We note also that low-temperature measurements (Fig. 7) show a large increase of the band at 3200  $\text{cm}^{-1}$ , probably due to the hardening of bonds in water molecules contained in the surface layer of  $\text{CdF}_2$  crystal or due to the water adsorption into the surface defects during cooling. Since the main vibrational modes seen in our spectra were due to surface effects we decided to always polish our samples after implantation as well as before each measurement. Because the depth of the implanted layer containing

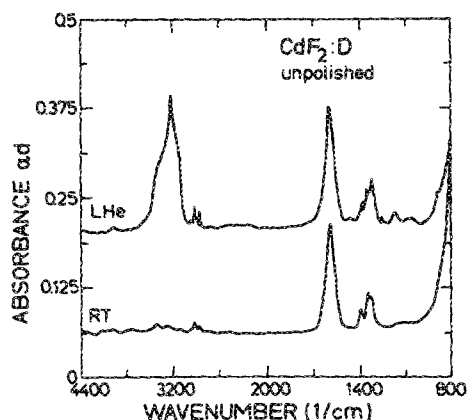


FIG. 7. Spectra of  $\text{CdF}_2$  implanted with deuterons, measured unpolished in room (RT) and about liquid helium (LHe) temperatures. Note change of the scale at  $2000\text{ cm}^{-1}$ .

hydrogen is about  $25\text{ }\mu\text{m}$ , careful polishing will remove only surface contaminations, leaving the hydrogen in the crystal. Figure 8 shows that, indeed, polishing completely removes adsorbed water; but introduces some additional lines between  $1000$  and  $1100\text{ cm}^{-1}$ . Since our polishing has been done in an alcohol bath we assign these lines to the vibration of alcohol molecules on the surface of the crystal (according to Ref. 20, ethyl alcohol has vibrational frequencies between  $1000$  and  $1300\text{ cm}^{-1}$ ). Figure 9 presents our data for proton- and deuteron-implanted cadmium fluoride, measured at three different temperatures. One can clearly see, even at room temperature, two lines at  $1570$  and  $1155\text{ cm}^{-1}$ . Due to their frequency ratio of about  $1.36$  we assign these lines to hydrogen and deuterium vibrations. We have already shown that<sup>9</sup> hydrogen/deuterium vibrations in irradiated semiconductors exhibit isotope shifts of less than the expected value of  $\sqrt{2}$  because of anharmonicities. Here, the factor is even smaller than in the case of other semiconductors which leads us to the assumption that we observe second harmonic hydrogen/deuterium vibrations. Figure 10 shows details of the temperature dependence of the hydrogen vibrations for a

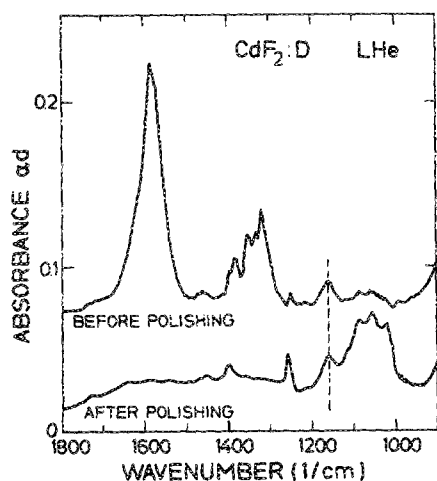


FIG. 8. Spectra of the  $\text{CdF}_2$  sample implanted with deuterons, measured in LHe temperature before and after RT polishing.

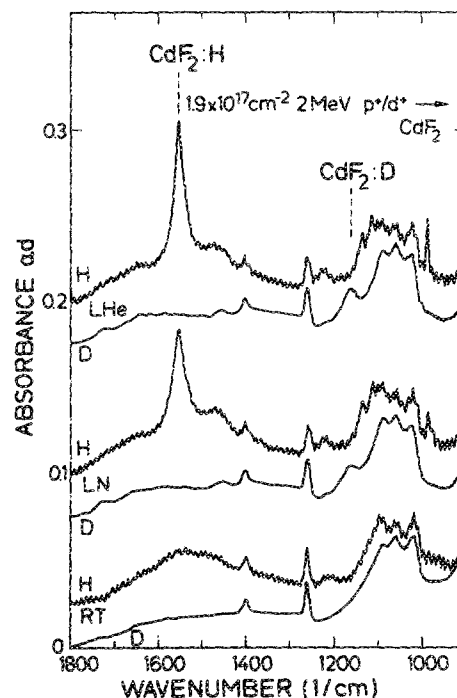


FIG. 9. Comparison of room, about liquid nitrogen (LN), and about liquid helium temperature measurements of proton- and deuteron-implanted  $\text{CdF}_2$  samples after polishing. Lines at about  $1250$  and  $1400\text{ cm}^{-1}$  are due to KRS-5 windows.

sample irradiated with low ion current. The vibrational frequency changes from  $1570$  (at RT) to  $1550\text{ cm}^{-1}$  (at LHe) and the line strongly sharpens. We note also that the isotopic ratio changes from  $1.36$  at RT to  $1.34$  at LHe. We do not see the fundamentals of these vibrations because of lattice phonons. Two-phonon absorption in  $\text{CdF}_2$  starts<sup>21</sup> rapidly at about  $805\text{ cm}^{-1}$  (see also Fig. 6), so it masks possible weak hydrogen-localized vibrational modes. On the other hand we can see (Fig. 10) lattice combination bands associated

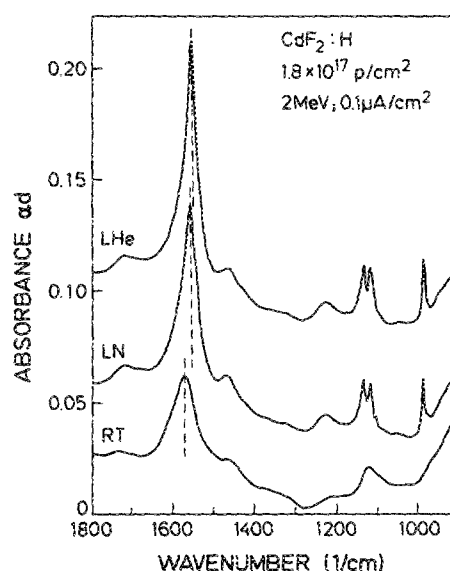


FIG. 10. Comparison of RT, LN, and LHe spectra of proton-implanted  $\text{CdF}_2$  for a sample implanted with very low ion current.

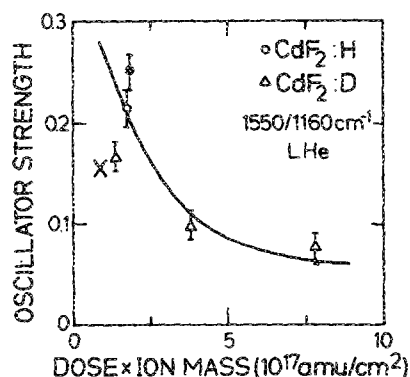


FIG. 11. Calculated oscillator strengths for proton- and deuterium-implanted cadmium fluoride, about liquid helium temperature measurements.  $\times$  denotes sample broken during irradiation. Continuous line is only a guide to the eye. Note that for the same proton dose we get higher oscillator strength for the sample irradiated with lower ion current (full point).

with the second harmonic of hydrogen in  $\text{CdF}_2$ : the most pronounced are at about  $1470$  and  $1720 \text{ cm}^{-1}$ . According to Elliot *et al.*<sup>4</sup> these bands arise as combinations of localized vibrations with lattice phonon states. The energy differences (about  $80$  and  $170 \text{ cm}^{-1}$ ) suggest that we observe combinations with TA and LA modes, because TO and LO modes in  $\text{CdF}_2$  have much higher energies.<sup>22</sup>

Using our standard method<sup>9</sup> we were able to determine the oscillator strengths for the observed hydrogen-localized vibrational modes. Figure 11 presents the values of oscillator strengths as a function of radiation damage (cf. Fig. 4). The decrease of the oscillator strength with increasing damage is clearly an artifact of our computational procedure<sup>9</sup>: we assume that *all* implanted ions are retained in the lattice, whereas for higher doses (higher damage) one expects their partial release. We can also calculate the so-called effective vibrating charge,<sup>9</sup> which in our case is about  $0.5e_0$  for low doses and decreases to about  $0.3e_0$  for the highest damage. It is worth noting that for the sample broken during irradiation we get a smaller oscillator strength. We note also that for deuterium implantations which produce more defects we always get smaller oscillator strengths (Fig. 11). On the other hand, for the sample implanted with low proton current we get a larger oscillator strength than for the same proton dose implanted with higher current. These facts further support the concept of hydrogen/deuterium release during irradiation because of the same mechanism of accumulation of defects that leads to crystal strains and to samples breaking.

#### IV. CONCLUSIONS

We have shown that it is possible to introduce hydrogen into cadmium fluoride without creating donors which hide the presence of localized vibrational modes. The observed hydrogen vibrational frequency corresponds to the second harmonic and agrees with frequencies observed for hydrogen vibrations in other fluorites. The somewhat lower value of the fundamental hydrogen vibrational frequency, inferred from our measurements of  $\text{CdF}_2\text{:H/D}$  as compared with  $\text{CaF}_2$  [ $785$  vs  $958 \text{ cm}^{-1}$  (Ref. 4), respectively] can be explained by the much higher mass of cadmium and, thus, hydrogen bonding by electrons from higher orbitals. In fact,

the fundamental hydrogen frequency in barium fluoride<sup>4</sup> has a similar value of about  $798 \text{ cm}^{-1}$  as in  $\text{CdF}_2$ . However, due to our inability to see the fundamental hydrogen frequency in cadmium fluoride we cannot make detailed calculations of the local potential (as in Ref. 4) to predict the symmetry of possible defects in which hydrogen is bonded. According to Newman<sup>23</sup> the observation of second and third harmonic transitions is the proof of  $T_d$  symmetry of the environment of vibrating hydrogen, i.e., that hydrogen occupies fluorine sites. For all other point symmetries considered (spherical, trigonal, tetragonal, and rhombic) there are more than one second harmonic transitions (with slightly different energies) and no third harmonic transitions. However, in the case of highly defective material, it is quite possible that hydrogen is bonded within complex defects, as suggested by our VIS measurements. In this situation much lower local symmetries of defects will also allow for second and third harmonic transitions. This interpretation for  $\text{CdF}_2$  implanted with protons or deuterons is further supported by the completely different temperature behavior of the position of hydrogen-localized vibrations: we measured (Fig. 10) a decrease of the hydrogen vibrational energies for decreasing temperatures, whereas Elliot *et al.*<sup>4</sup> reported an increase. Our lines are also extremely broad (about  $20 \text{ cm}^{-1}$ ) compared to those observed in other fluorites (between  $1$  and  $3 \text{ cm}^{-1}$  for second harmonic transitions). These differences can be explained by inhomogeneous defect line broadening, typical of extended defects.

Our spectra show only small traces of deuterium third harmonics (see Fig. 9, around  $1730 \text{ cm}^{-1}$ ) and no third harmonics of hydrogen can be observed (they coincide with a strong  $\text{CO}_2$  absorption band around  $2330 \text{ cm}^{-1}$ ). Hence, due to the lack of parameters, we cannot compute anharmonic forces in cadmium fluoride. Observation of third harmonic transition is according to Newman<sup>23</sup> typical of cubic symmetry, but again we believe that it appears because of high lattice distortions around extended defects. However, on the basis of the presented experimental data we cannot exclude the situation when only part of hydrogen is bonded in fluorine sites whereas the rest occupy extended defects.

The present work clearly shows that hydrogen can be bonded in cadmium fluoride in various positions (surface or bulk modes), some of them corresponding to water particles adsorbed by this hygroscopic material. Further studies of proton radiation effects in  $\text{CdF}_2$  with different experimental techniques will probably answer the question of the nature of the defects in which hydrogen is bonded.

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