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Radiation Damage in BaF_2 Crystals

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ABSTRACT

The effects of radiation damage and recovery have been studied in BaF_2 crystals exposed to ^{60}Co radiation. The change in optical transmission and scintillation light output have been measured as a function of dose up to 4.7×10^6 rad. Although some crystals exhibit a small change in transmission, a greater change in scintillation light output is observed. Several 25 cm long crystals which have been irradiated show large changes in both transmission and light output. Recovery from radiation damage has been studied as a function of time and exposure to UV light. A long lived radiation induced phosphorescence has been observed in all irradiated samples which is distinct from the standard fast and slow scintillation emissions. The emission spectrum of the phosphorescence has been measured and shows a peak at ~ 330 nm, near the region of the slow scintillation component. Results are given on the dependence of the decay time of the phosphorescence with dose.

1. INTRODUCTION

Barium fluoride has long been believed to be one of the most radiation resistant inorganic scintillators known [1]. Because of its excellent radiation hardness and fast time response (0.6 ns for the fast component), it is of considerable interest for applications involving very high counting rates, such as at the SSC or LHC. However, most studies of radiation damage in BaF_2 in the past have involved only the measurement of the internal transmission loss in crystals due to radiation. Although transmission loss in the region of the scintillation emission will certainly affect the amount of light reaching the detector, other factors, such as geometry, light collection efficiency, surface absorption and scatter, as well as the overall scintillation efficiency (i.e., the amount of scintillation light produced per unit energy deposit) can affect the amount of light detected by the readout device.

The change in optical transmission of a number of BaF_2 crystals has been measured as a function of exposure to ^{60}Co radiation up to a dose of 4.7×10^6 rad. The scintillation light output was also measured as a function of dose, and the correlation between the change in transmission and loss in light output is given. The transmission and light output were also measured over a period of time after irradiation to study the effect of natural recovery from radiation damage. Recovery was also studied as a function of exposure to UV light.

Finally, a long lived phosphorescence, or afterglow, was observed in all samples exposed to radiation. This phosphorescence is characterized by a component with a

short decay time which dies away within a few seconds after irradiation, along with another component with a much longer decay time which persists for weeks or even months. The emission spectrum of the phosphorescence has been measured and is found to occur at nearly the same wavelength as the slow component scintillation emission. The phosphorescence appears to be related to the other radiation induced effects in the crystals.

2. EXPERIMENTAL PROCEDURE

The radiation measurements were carried out at two different facilities at Brookhaven National Lab. One, the Gamma Ray Radiation Facility [2], consists of a multipurpose visible and UV spectrophotometer used in conjunction with a high intensity (~ 20 KCi) ^{60}Co source. This system can be used to carry out optical measurements on crystals before, during and after irradiation. Another facility, HIRDL [3], consists of a collection of ^{60}Co sources totaling ~ 90 KCi arranged in a pool of water containing long exposure tubes which provide uniform doses for samples up to 30 cm in length. We have used the HIRDL facility to irradiate several small samples at the same time, as well as to irradiate larger samples one at a time. The dose rate used at HIRDL was 3.4×10^4 rad/hr, and typically 3.8×10^4 rad/hr at the Gamma Ray Facility. It should be noted that these dose rates are considerably higher than would be expected in any high energy collider experiment.

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The samples used in this study were obtained from several sources. A number of 1" dia. x 1" long polished cylinders were obtained from ingots grown at Korth Monokristalle [4] between 1989-1990 for the TAPS project [5] which were machined and polished at Optovac, Inc. [6]. Two samples, henceforth referred to as 1 and 2, were used for the transmission and light output study, along with a third sample (3) grown by Harshaw [7] in around 1985. The Harshaw sample was produced by a so-called slab growth method developed a number of years ago, and was chosen in order to have a sample with a completely different origin than the Korth samples. Two additional samples (4 and 5) were produced from the Korth ingots and were used for measuring the scintillation emission spectrum and studying the phosphorescence. Two large samples, each a 25 cm long trapezoid measuring $3 \times 3 \text{ cm}^2$ at the front and $4 \times 4 \text{ cm}^2$ at the back, were also studied. One was obtained from Korth and was cut from an ingot similar to the one used to produce the small samples. The second was obtained from the Shanghai Institute of Ceramics (SIC) using an ingot grown at the Beijing Glass Research Institute (BGRI).

Before irradiation, the samples were cleaned lightly, but not repolished, using ethyl alcohol and wrapped in aluminum foil. Whenever possible, the samples were kept in total darkness to minimize their exposure to external light. Preparation of the samples for transmission and light output measurement after each irradiation was carried out under a dim red light. All measurements were typically completed within 3 hours after irradiation and the samples immediately returned to the ^{60}Co pool to continue the exposure. For the large samples, a few days elapsed between each exposure in order to allow time for the phosphorescence to sufficiently die away in order that light output measurements could be made.

The transmission of each sample was measured within approximately one hour after irradiation using a Hitachi U-3210 Spectrophotometer. This instrument was equipped with a large sample compartment which permitted measuring the transmission of samples up to 30 cm in length. It also utilized an integrating sphere which helped collect all refracted and scattered light from the sample which greatly improved the accuracy of determining the transmission. However, due to other systematic effects, such as cleaning and handling the samples, errors in repositioning the samples inside the spectrophotometer, etc., the accuracy of the transmission measurements was $\sim \pm 0.5\%$.

The scintillation light output was measured using a special Hamamatsu R2059 2" diameter photomultiplier tube equipped with a Cs-Te solar blind photocathode and quartz window. The solar blind photocathode has a peak sensitivity in the region of the BaF_2 fast component and greatly suppresses the signal from the slow component. In addition, it also has very low sensitivity to the phosphorescence, as described later. Before each measurement, the samples were lightly wiped again with alcohol and

wrapped in 2 layers of white reflecting teflon. The crystal was placed immediately in front of the phototube with only a thin air gap ($\leq 0.1 \text{ mm}$) in between. Whenever possible, no optical grease was used to couple the crystal to the phototube in order to reduce the systematic error introduced by the grease joint. However, as the light output decreased with increasing dose, it became necessary to use a UV transmitting grease (GE Viscasil 600M) to increase the detectable signal.

The light output was measured in terms of the number of photoelectrons produced on the photocathode of the phototube per MeV of energy deposited in the crystal by a ^{137}Cs gamma ray source. The signal from the phototube was digitized using two separate LeCroy 2249W ADC's, one with a 20 ns effective gate width, used to measure the fast component, and a second with a 1 μsec gate, used to measure the total light output (fast plus slow). The photopeak from the ^{137}Cs source was used to determine the charge output from the phototube at a given voltage for an energy deposit of 662 keV in the crystal. The charge per photoelectron for the phototube operating at the same voltage was determined independently using an LED. Although the absolute number of photoelectrons was measured in each case, our results, for the most part, are given in terms of the relative change in light output with respect to the unirradiated sample.

The gain of the phototube, as well as other sources of systematic errors, were monitored continuously throughout the experiment using a control BaF_2 sample which was not irradiated, but treated in every other way the same as the irradiated samples. The overall systematic error in the determination of the light output was a maximum of $\pm 5\%$, with an rms of $\sim 2\%$. This variation essentially determines the limit of our sensitivity to measure changes in light output due to radiation.

3. TRANSMISSION AND LIGHT OUTPUT RESULTS

Samples 1, 2 and 3 were simultaneously irradiated at the HIRDL facility to doses of 10^3 , 10^4 , 6×10^4 , 2.7×10^5 , 9×10^6 and $4.7 \times 10^6 \text{ rad}$. The transmission and light output were measured after each irradiation as described above. Figure 1 shows the transmission spectra for the three samples as a function of dose. All three samples developed noticeable absorption bands in the wavelength region between 300-700 nm. The intensity and position of the bands vary from sample to sample, with the most prominent band produced in the Harshaw sample around 550 nm. The absorption spectra in samples 1 and 2 are similar, although not identical. Sample 1 showed a very small change in transmission in the region of the fast component ($\sim 1\%$ at 200 nm), and a much larger change in the region of 500-700 nm. All three samples exhibited saturation in the amount of induced absorption after $\sim 10^4 \text{ rad}$. This type of saturation has been observed before [8] and is indicative of radiation damage caused

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Fig. 1: Transmission vs. wavelength for samples 1-3 (a-c) for doses of (1) unirradiated, (2) 10^3 , (3) 10^4 , (4) 6×10^4 , (5) 2.7×10^5 , (6) 9.0×10^5 and (7) 4.7×10^6 rad. Note expanded vertical scale.

by either impurities or defects in the crystal. However, it is not possible to uniquely determine what kind of impurities or defects could be causing the damage from these absorption spectra alone.

Figure 2 shows the relative change in light output for the 20 ns gate (fast) and 1 μ sec gate (total) for the three samples as a function of dose. The first few points were

Fig. 2: Relative change in light output of samples 1-3, (a) fast, (b) total, for doses of (1) unirradiated, (2) 10^3 , (3) 10^4 , (4) 6×10^4 , (5) 2.7×10^5 , (6) 9.0×10^5 (7) and 4.7×10^6 rad.

measured without grease coupling, but as the dose increased, the light output dropped to a point which was too low to measure reliably without better optical coupling. Therefore, the higher dose points were measured with grease, and are normalized to their respective preirradiation values, also measured with grease. A decrease of 30-40% in the light output is observed for both the short and long gates which is well outside the range of systematic error. The loss in light output also shows the same saturation effect, at approximately the same dose, as the transmission data.

From the data given in Figures 1 and 2, it is apparent that the loss in light output exceeds that which would be expected from the observed loss in internal transmission. This effect has also been recently reported by another group [9]. In the case of sample 1, the transmission loss for the fast component is $\sim 1\%$, while the loss in light output is $\sim 30\%$. It is clear that a simple transmission loss cannot explain this effect. However, as mentioned above, the amount of light detected by the phototube is determined by numerous factors, such as geometry, light collection efficiency, and the amount of scintillation light

Fig. 3: Scintillation emission spectrum for samples 4 (a) and 5 (b) measured during irradiation. Curves 100 rad and 10^6 rad for sample 4 are overlapping. Data are uncorrected for the spectral response of the spectrophotometer.

produced within the crystal. We have used a ray tracing simulation program [10] to study the effects of changes in the attenuation length and light collection in the crystal to see if the increased internal absorption could explain the observed loss in light output. We have found that the observed increase in absorption alone *cannot* explain the decrease in light output using a simple model. This conclusion was also reached in ref. [9]. We are presently using this program to try and determine if changes in surface reflectivity, or perhaps increased absorbance at the surface, could further explain this effect.

In order to determine if the amount of scintillation light produced with a crystal changes as a result of radiation, two other samples (4 and 5) were irradiated at the Gamma Ray Facility where the scintillation emission spectrum was measured *during* irradiation. Figure 3 shows the results. The spectra have not been corrected for the spectral response of the spectrophotometer, and hence do not give the actual shape of the emission spectrum. Also, the arrangement of the spectrometer is not the same as that used for making light output measurements,

Fig. 4: Transmission spectra for samples 4 (a) and 5 (b) measured before and after irradiation of 10^6 rad. Note expanded vertical scale.

and the light collection efficiencies in the two setups are quite different. However, the data do give a relative comparison of the spectra for the different doses. Sample 4 shows no change in either the shape or magnitude of the spectrum, while sample 5 shows a change of $\sim 15\%$. This change could be within the systematic error of the measurement, and tests are currently under way to verify this with additional samples and improved calibration of the spectrometer. However, these results indicate that the intrinsic scintillation efficiency is *not* dramatically changed during radiation, at least not in these samples.

After irradiation, the transmission and light output of samples 4 and 5 were compared with their pre-irradiation values. Both samples exhibited induced absorption, as shown in Figure 4. The light output of sample 4 decreased by $\sim 30\%$, and sample 5 by $\sim 50\%$. Since this is significantly greater than the observed change in scintillation intensity in either of the two samples, the apparent loss in light output must be due to other effects. One possibility is that the radiation causes changes in the surface reflectivity or absorption, or otherwise changes the light collection efficiency, as mentioned above. It is also possible, however, that the apparent scintillation efficiency

Fig. 5: Transmission spectra of 25 cm BaF_2 trapezoids measured after 10^3 and 10^4 rad, (a) SIC sample (b) Korth sample.

during irradiation may be different than after irradiation due to the large amount of ionization occurring in the crystal while it is being irradiated. We are presently exploring both of these possibilities with further measurements and calculations with the simulation program to try and fully understand this effect.

The radiation damage was also measured in the two 25 cm trapezoids. Figure 5 shows the change in transmission of the two samples measured along their 25 cm axes for doses of 10^3 and 10^4 rad. In both cases, a large amount of induced absorption is observed. The SIC sample exhibited a small absorption band ~ 290 nm before irradiation which is believed to be due to cerium contamination at the level of a few ppm [11]. There was an indication of the same absorption band in the Korth sample as well, although much weaker. A prominent absorption band developed at ~ 530 nm in the SIC sample after 10^3 rad, and continued to grow after 10^4 rad. The Korth sample developed a more complex structure of absorption bands which became more pronounced at 10^4 rad.

The light output as a function of position along the length of each crystal is shown in Figure 6. The crystals were wrapped in white reflecting teflon for these measurements, although no special effort was made to make the

Fig. 6: Change in light output as a function of position along the 25 cm BaF_2 trapezoids after 10^3 rad, (a) SIC sample (b) Korth sample. Measurements were made ~ 2 days after irradiation.

light collection particularly uniform over the length before irradiation. After 10^3 rad, the light output of the SIC crystal dropped by $\sim 30\%$ at one end and $\sim 40\%$ at the other. The light output of the Korth sample decreased by $\sim 20\%$ at one end and $\sim 35\%$ at the other. The light output was too low to measure after 10^4 rad in either sample. The difference in the amount of damage from one end to the other would be expected if the damage were related to impurities in the crystal which segregate to one end during growth.

4. RECOVERY

Recovery from radiation damage, both in the transmission and light output, has been studied in two ways. One is the natural recovery with time, and the second is the recovery induced by exposure to UV light. Figure 7 shows the change in transmission of the three 1" cylinders measured at various times over a period of 60 days after irradiation. All samples were kept in the dark at room temperature during this period. Some natural recovery is seen, although the amount is generally quite small. The

Fig. 7: Recovery in transmission with time for samples 1-3 (a-c): (1) unirradiated, (7) 1 hr. after irradiation, (8) 8 hrs., (9) 1 day, (10) 2 days, (11) 7 days, (12) 16 days (13) 50 days (14) 60 days. Note expanded vertical scale.

one exception is sample 1, which shows significant recovery in the 400-800 nm range. Somewhat more recovery is observed in the light output, as shown in Figure 8. The recovery is approximately the same for both the fast and total, gaining back ~ 5-10% of the original light output.

Fig. 9: Recovery in relative light output with time for samples 1-3: (a) fast, (b) total. Time after irradiation: (7) 1 hr., (8) 8 hrs., (9) 1 day, (10) 2 days, (11) 7 days, (12) 16 days (13) 50 days (14) 60 days.

Recovery was also studied as function of exposure to UV light. A strong mercury lamp was used to illuminate several samples for short periods of time, and the transmission and light output were measured immediately after each exposure. Sample 1 was illuminated in a continuous series of measurements starting with 30 seconds and continuing up to a cumulative exposure of 127.5 minutes. Figure 9 shows the transmission spectrum just after irradiation, after 60 days of natural recovery, and after 3.5 and 127.5 minutes of exposure to the mercury lamp. Figure 10 shows the recovery in light output as a function of exposure time. Essentially complete recovery is achieved after the ~ 2 hour exposure.

Sample 2 was also exposed to the mercury lamp, but the exposures were interrupted several times during the process, once for 34 days after 16 minutes of exposure, again for 2 days after 23 minutes, and finally for 1 day after 25 minutes. The sample was kept in the dark during each interruption. The transmission and light output

Fig. 9: Recovery in transmission of sample 1: (1) unirradiated, (2) 1 hr. after 4.7×10^6 rad, (3) after 60 days (4) after 60 days plus 3.5 minute exposure to mercury lamp (5) after 60 days plus 127.5 minute exposure to mercury lamp.

Fig. 10: Recovery in relative light output as a function of time of exposure to mercury lamp for sample 1.

were also measured before and after each interruption, and were found to be unaffected by the pauses in the recovery. Finally, after 80 minutes of total exposure, the recovery in the transmission and light output were found to agree with the results obtained for sample 1. These results indicate that, although there may be no permanent damage in the crystal, the effects are in fact long term, and recovery does not occur rapidly without external stimulation.

Fig. 11: Phosphorescence emission spectrum measured with a sample kept at room temperature and a sample heated to 100°C . Both curves are normalized to 1.0 at the peak.

5. PHOSPHORESCENCE

A strong phosphorescence, or afterglow, was observed in all samples after irradiation. This phosphorescence was first noticed when trying to measure the light output from several irradiated crystals using a phototube with a standard bialkali photocathode and quartz window. The phosphorescence produced a severe background in the phototube, causing the anode current to reach several hundred microamps at the normal operating voltage. The phototube with the solar blind photocathode, used for most of the measurements described above, was far less sensitive to the phosphorescence, and the background current was generally not a problem. However, the suppression of the background is not complete and some background current is produced even in the solar blind tube. The gain of this tube was checked to be stable to within a few percent up to a current of $10 \mu\text{amp}$, and at no time did the current exceed $1 \mu\text{amp}$ while measuring the light output of the small crystals. However, the background current was much greater with the larger crystals, and reached several tens of microamps immediately after irradiation. Because of this, the light output of the large samples was measured several days after irradiation to allow sufficient time for the phosphorescence to die away and reduce the background current to a tolerable level.

The emission spectrum of the phosphorescence was measured after irradiation at the Gamma Ray Facility. One measurement was done keeping the crystal at room temperature and signal averaging over many wavelength scans. Another spectrum was obtained by heating the crystal to 100°C , which increased the intensity of the phosphorescence and improved the signal to noise ratio. The spectra obtained by these two methods are shown in Figure 11. In both cases, the peak of the spectrum

Fig. 12: Phosphorescence decay spectrum after doses of 600, 4×10^3 , and 1.0×10^6 rad.

Table I

Components of Phosphorescence Decay in BaF_2

Dose (rad)	Components (τ in seconds, () = %)		
	τ_1	τ_2	τ_3
600	2.6 (23)	12.3 (10)	184 (67)
8.8×10^3	1.9 (2)	14.1 (1)	215 (97)
1.5×10^5	1.8 (2)	16.4 (2)	219 (96)
1.0×10^6	1.8 (1)	15.2 (2)	208 (97)

occurs at ~ 330 nm, which is near the region of the slow component scintillation emission (~ 310 nm). Since the quantum efficiency of the Cs-Te photocathode cuts off sharply above ~ 300 nm, the solar blind tube sees only the tail of this spectrum.

Figure 12 shows the decay time of the phosphorescence measured immediately after irradiation for several doses. For a low dose, most of the phosphorescence decays away very quickly with a time constant on the order of a few seconds. At larger doses, the decay curve develops a long tail with a much longer decay time. Table I gives the decay curve separated into several components with their respective $(1/e)$ decay times for various doses. Although there was no direct attempt to measure them, it was apparent that there were some components of the phosphorescence which persisted for very long times. Some crystals kept at room temperature still showed signs of phosphorescence even months after irradiation.

It was difficult to get an accurate measure of the relative intensity of the phosphorescence in the three 1" cylinders since the intensities were changing quickly with time during the measurements. The amount of activity did, however, differ between the three crystals by about a factor of ~ 3 , sample 1 being the highest

Fig. 13: Phosphorescence intensity and decay in an irradiated crystal after several exposures to roomlight. The intensity prior to exposure to roomlight is also indicated.

and sample 2 being the lowest. The intensity of the phosphorescence also appeared to saturate at about the same dose at which the transmission and light output saturated, implying that the three effects are somehow related.

It was also found that the amount of phosphorescence activity increased dramatically when the crystals were exposed to roomlight or sunlight. Figure 13 shows the increase in activity and subsequent decay after two short exposures of an irradiated crystal to roomlight. The activity increases sharply after the first exposure, then dies away, increases even more after the second exposure, then dies away again. It was found that after a long exposure to sunlight (~ 2 days), the phosphorescence completely died away and could no longer be reactivated. This effect is typical of phosphorescence in other materials where charges, which are liberated during irradiation, become trapped on sites within the crystal, perhaps impurities or defects, and require a minimum energy of a few eV to be released. Once released, either by raising the temperature or supplying external light, the charges are free to recombine on luminescent centers, giving rise to the phosphorescence. Once all of the trapped charges have been released, no further phosphorescence occurs. This result again supports the notion that the radiation effects observed here are related to either impurities or defects, and are not necessarily a property of the pure crystal.

6. CONCLUSIONS

Several radiation induced effects have been observed in BaF_2 crystals. The first is the production of absorption bands in the wavelength range from 200-800 nm. The position and intensity of these bands are highly sample dependent. All small samples exhibited a small amount of absorption in the region of the fast component, but different amounts of absorption at longer wavelengths. The larger samples showed much greater absorption at all wavelengths. The loss in transmission saturated at $\sim 10^4$ rad.

The second effect was a decrease in the amount of detected scintillation light, which saturated at $\sim 10^4$ rad, similar to the transmission. Again, the effect was worse in the larger crystals. The decrease in detected light is greater than can be accounted for by the simple loss in transmission at wavelengths in the region of the scintillation emissions. A Monte Carlo calculation shows that, even with multiple reflections, the increased internal absorption due to radiation cannot account for the observed loss in light. A measurement of the scintillation emission spectrum during irradiation shows little or no change in the shape and intensity of the emission spectrum. This implies that the loss in detected light is due to some other effect, such as a change in the light collection efficiency, or that the scintillation intensity measured during irradiation is not the same as after irradiation. Additional measurements and calculations are currently under way to try and better understand these results.

The third effect is that a strong phosphorescence was observed in all irradiated samples. The phosphorescence occurs at a wavelength near the slow component scintillation emission and has a decay time which is dependent on dose. For low doses, the phosphorescence dies away with a short decay time, while at larger doses, it develops a component which persists for much longer times. The phosphorescence can be reactivated by exposure to external light, but can be eliminated completely with a sufficiently long exposure to sunlight. The intensity of the phosphorescence varied from sample to sample, but showed the same effect of saturation as the loss in transmission and light output.

It is very likely that all of the radiation effects observed in these crystals are related. The common effect of saturation, which occurred in all the data, tends to indicate that the origin is related to impurities or defects inside the crystal. This is supported by the fact that the phosphorescence can be optically reactivated up to a point, but eventually disappears. Also, the non-uniformity in the loss in light output from the larger crystals is indicative of impurities or defects. Unfortunately, the information obtained in this study is not sufficient to identify any of the impurities or the nature of the defects. However, a great deal of work is being done, particularly at SIC, BGRI and Optovac, to identify the causes of radiation damage in BaF_2 . One possible cause is contamination by oxygen or OH^- ions which are left behind during growth.

Other possibilities may be traces of rare earth impurities, or defects related to residual stress in the crystal. If the radiation effects are, in fact, defect or impurity related, it should be possible to greatly improve the radiation hardness of BaF_2 with higher purity raw material and improved growth techniques. Work in these areas is currently under way. This effort to understand the causes of these effects will hopefully lead to improved, more radiation hard crystals for high energy physics, as well as other applications.

7. ACKNOWLEDGMENT

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