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To be submitted to: 1975 IEEE Annual
Conference on Nuclear and Space
Radiation Effects, Arcata/Eureka, Calif.
July 14-17, 1975.

BNL 20253

Copy. 150703--13

**RADIATION INDUCED COLORING OF GLASSES MEASURED
DURING AND AFTER ELECTRON IRRADIATION***

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Abstract

The growth of color centers during irradiation, and the decay after irradiation, has been studied in two glasses using recently developed equipment for making optical absorption and luminescence measurements during and after electron irradiation. The glasses studied were NBS 710, a soda-lime silicate glass and NBS 711, a lead silicate glass. Both glasses exhibit similar coloring characteristics. The radiation induced absorption spectra consists of a weak gaussian shaped band in the visible, a stronger gaussian band in the ultra violet and a band edge "shift" which may be accurately approximated by a third gaussian band. For all absorption bands the color center v.s. dose (or irradiation time) curves can be accurately resolved into two saturating exponential and one linear component. The decay curves obtained after the irradiation is terminated can be accurately expressed by three exponential components. Coloring and decay curves made at different dose rates indicate that the processes responsible for decay after irradiation and electron hole recombination during irradiation play important roles in determining the rate and extent of coloring. The results are qualitatively in agreement with some very simple kinetic treatments for color center formation. In some, but not all, respects the quantitative agreement is also good. Lastly, the results appear to provide strong evidence to conclude that it is necessary to make measurements during irradiation to establish the formation kinetics of color centers that are unstable at the bombardment temperature.

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*Research supported by U. S. Energy Research and Development Administration

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Introduction

In the past, almost all radiation-effect studies on glasses, as well as most other materials, have been made by first irradiating a sample and then transporting it to a separate instrument for measurement at a later time. Recently, equipment has been completed for making simultaneous optical absorption and luminescence measurements on optical materials during 0.5 to 3 MeV electron irradiation. With this equipment, in addition to determining the absorption and luminescence present during irradiation, one determines changes in absorption and any phosphorescence which may occur after the irradiation is terminated. Described below is, as far as can be determined, the first report on measurements of the coloring of glasses during continuous, or steady-state, electron irradiation.

Previously, similar studies were made on Barium Aluminoborate glasses during steady-state ^{60}Co gamma-ray irradiation.¹ Also, a number of glasses intended for fibre optic applications have been studied during pulse and steady-state x-ray irradiation and pulse electron irradiation.^{2,3} The completed studies have provided a number of qualitative and quantitative results which indicate that electron-irradiation experiments would provide appreciable new information on the physical processes contributing to the coloring of glasses by radiation. For example, electron irradiations can be made at dose rates comparable to those used previously, as well as at rates thousands of times higher. Also, by varying the energy of the bombarding electrons one can determine its atom displacements are important contributors to the damage process in glass. Numerous additional examples could be cited to demonstrate the usefulness of electron irradiation studies.

Most importantly, however, measurements on the growth of the coloring during irradiation, and the changes occurring after irradiation, are the best possible ways of studying the kinetics of the radiation-induced processes in glasses - and other materials. In fact, except for a few exceptional cases, it is essential to make the measurements during irradiation to determine coloring kinetics in a reasonably thorough manner. This paper will describe studies designed to emphasize the kinetic aspects of the radiation-induced coloring of glasses.

Experimental Equipment and Methods

The equipment used for these measurements, which has been described previously,⁴ will be outlined briefly in this section. A detailed description will be published in the future.

The bombarding electrons are produced in a vertical electron accelerator which can be operated at approximately 0.5 to 3 MeV. The vertical beam is deflected into a long horizontal-beam tube toward the sample area. This tube is equipped with focusing coils, Faraday cups, and a thin gold foil scatter to insure that the sample is uniformly irradiated. During irradiation the beam incident on, and passing through, the sample is monitored by a Faraday cup behind the target. The current detected by this cup is recorded throughout the measurement.

Optical absorption and luminescence measurements are made with a computer controlled double-beam spectrophotometer shown schematically in Fig. 1. One monochromator provides a monochromatic light beam for absorption measurements. This beam is split, sequentially chopped, and conveyed to the target area by an optical relay system. The sample is placed at the image point of the "sample" beam. The image point of the "reference" beam appears 12 mm away at a symmetrical point on the system's axis. Thus both beams are subjected to equal changes in window absorption, air glow, and any other perturbations along the optical path which might occur during irradiation.

After passing through the target area, beams are focused by the second leg of the optical relay system on monochrometers which serve as tunable optical band-pass filters. (Actually, for the measurements described in this paper, a single "filter" monochromator was used in the sample beam. The reference beam was focused directly on a phototube. This arrangement is quite adequate for these studies since it was immediately determined that the glass samples did not produce strong luminescence.) The filters prevent all luminescence light from reaching the phototube except in the wavelength region specified. In the presence of luminescence this increases the signal to noise ratio for absorption measurements by factors of 100's to 1000's. Also, the filter monochromator in the sample beam is used to measure the spectrum of the emitted luminescence. The filter monochromator in the reference beam can be used to determine the spectrum of the light emitted by the target-chamber windows, the air glow, etc.

At each wavelength, four measurements are made. The actual sequence of measurements is given in Fig. 2. The dwell time at each wavelength may be specified, so that a number of separate measurements may be made at each point for signal averaging. This data, as well as information such as sample temperature beam current, etc., is stored in the computer. At the end of each scan, which occurs every 40 sec. or longer, the data is transferred to magnetic tape. The computer specifies the wavelengths at which measurements are to be made, time interval between scans, etc.

The data, recorded on tape, is processed using a large computer. Each scan which can consist of up to 256 points in the wavelength region 200 to 400 nm or 400 to 800 nm, provides both an absorption spectrum and a radioluminescence emission spectrum. These spectra are fully corrected for radiation-induced absorption and luminescence in the target-chamber windows, air glow, etc. Figure 3 was constructed from two separate absorption measurements on NBS 710 glass; one in the visible and the other in the ultraviolet. The two measurements are "joined" during computer processing. They show the growth of the coloring during irradiation and the decay occurring after the irradiation is terminated. Such "3-D" plots are very useful for planning detailed analysis of the data.

Once a data set, e.g. Fig. 3, has been obtained additional analysis is straightforward (but occasionally laborious). The first step in a complete analysis is the resolution of each spectrum into component absorption bands. There is now considerable evidence to support the assumption that the bands in glass are Gaussian shaped^{5,3,6}. Also, the resolution into Gaussian bands is readily accomplished by using a computerized best-fit procedure.⁷ Next, growth and decay curves may be constructed by plotting the observed absorption near or at the band maximum vs. irradiation time or dose. Or, if the resolution into Gaussian bands has been undertaken for enough scans, a more accurate growth, or decay curve, may be constructed. In the following sections, the analysis of such curves obtained with NBS 710 soda-lime silicate and NBS 711 lead silicate glasses obtained by making measurements during and after 1.5-MeV electron irradiations is considered in detail.

Experimental Results

Radiation-Induced-Absorption Spectra:

The absorption spectra induced by electron bombardment of NBS 710 and NBS 711 glasses are shown in Figs. 4 and 5. In each case, the spectra can be resolved into three components: a weak Gaussian band in the visible, a stronger Gaussian band in the u.v. and an "absorption edge shift", represented by the low-energy wing

of a third Gaussian (Table 1). A discussion of the specific nature of the centers responsible for the absorption lies outside the scope of this paper. However, the spectra shown resemble those obtained by other investigators for a variety of irradiation conditions^{2,3,8} and are evidently typical of the two glass types.

Growth of Radiation Induced Absorption:

The irradiation-induced absorption in both NBS 710 and NBS 711 glasses shows an initial rapid growth at the onset of electron bombardment and increases more slowly as the irradiation progresses. The relative growth rates differ slightly at different photon energies, reflecting the presence of multiple components in the spectrum.

The growth of the absorption at 4.0 eV in NBS 710 glass is shown in Fig. 6. This data can be accurately fitted by the expression

$$\alpha(t) = \sum_{i=1}^2 A_i (1 - \exp(-a_i t)) + \alpha_L t \quad (1)$$

where $\alpha(t)$ is the absorption coefficient and t the irradiation time. The fitting is accomplished by a computerized least-squares procedure illustrated in Fig. 7. First, a straight line is fitted to the data in the "plateau" region occurring at long irradiation times. This line is extrapolated to the origin and the data subtracted from it. The difference is then plotted on a normalized semi-log scale (upper curve, Fig. 7) and resolved into components by a successive subtraction process similar to that used in radioactive decay analysis. The straight lines in Fig. 7 clearly demonstrate the existence of two exponential components and the a 's and A 's are determined from the slopes and intercepts of the straight lines. The constants obtained from this procedure were used in Eq. (1) to compute the solid line through the data points in Fig. 6.

Similar growth curves have been constructed at various photon energies in the absorption spectra of NBS 710 and NBS 711 glasses. In every case it is possible to clearly resolve the absorption growth curves into two saturating exponential components and one linear component. Moreover, the relative growth rates, as indicated by the a_i coefficients, are quite similar in all cases (at a given dose rate). For example, the a_i coefficient in NBS 710 glass at 4.0 and 2.6 eV and in NBS 711 glass at 3.15 eV are 10.48×10^{-4} , 13.46×10^{-4} and $14.12 \times 10^{-4} \text{ sec}^{-1}$, respectively, at $3.7 \times 10^6 \text{ F/H}$. Further discussion and interpretation of these results will be presented in the next section.

Decay of Radiation Induced Absorption:

In all cases, the absorption immediately begins to decrease once the electron bombardment is terminated. The decay of the absorption subsequent to irradiation in NBS 710 glass at 4.0 eV is shown in Fig. 8. In all cases the decay can be resolved into three decaying exponential components as given by the expression

$$\alpha(t) = \sum_{i=1}^3 D_i \exp(-d_i (t-t')) \quad t \geq t' \quad (2)$$

where $\alpha(t)$ is the absorption coefficient and t' the time at which the irradiation is terminated. The fitting procedure (Fig. 9) is similar to that discussed above, with the exception that the linear component is absent. The data are plotted directly on a normalized semilog scale and resolved into three components as

shown by the straight lines in Fig. 9. Values of d_1 and D_1 were determined from the straight line slopes and intercepts. In turn they are used in Equation (2) to compute the solid line through the data in Fig. 8.

The decay curves can be resolved into three decaying exponential components at all photon energies in both NBS 710 and NBS 711 glasses. The decay curves contain a long lived component with $d \sim 10^{-6} \text{ sec}^{-1}$ and two short lived components with $d \sim 10^{-4}$ and 10^{-3} sec^{-1} . In NBS 710 glass, the visible absorption decays somewhat more quickly than the u. v. absorption. Also, the short lived component in the visible is a larger fraction of the total absorption than the short lived component in the u. v.

Growth and Decay Curves at Various Dose Rates:

The parameters in Equations (1) and (2), describing the growth and decay of the radiation induced absorption, depend on the dose rate. This dose rate dependence has been studied in NBS 710 glass. Figure 10 shows a family of growth curves at 2.6 eV for different dose rates in NBS 710 glass. The curves were plotted at 2.6 eV since only one absorption band contributes to the absorption in this region (Fig. 4). The solid curves in Fig. 10 were generated from Equation (1) using the best fit parameters shown in Table 2.

From the figure, it is evident that the saturation level increases with increasing dose rate. This corresponds to the increase in $A_1 + A_2$, in Equation (1), with dose rate. This is shown in Table 2 (although the individual A_1 coefficients vary somewhat). At higher dose rates saturation is reached more quickly; the a_1 and a_2 coefficients are roughly linear with dose rate. Finally, the linear term α_1 generally increases with increasing dose rate.

Decay curves at 2.6 eV are shown in Fig. 11 for different dose rates. The solid lines were computed from Equation (2) using the parameters in Table 3. Also, all curves were normalized to unity at $t = t'$. Figure 11 shows that the rapidly decaying component immediately following the termination of the irradiation increases with increasing dose rate. More specifically, Table 3 shows that this effect is due to an increase in the amplitude of the short lived component with increasing dose rate. The table does not show that the d coefficients depend on dose rate in a systematic manner.

Analysis and Interpretation of Results

Several of the results presented in the previous section can be qualitatively understood on the basis of a simple kinetic model for color center formation. This model, which has been discussed in detail in conjunction with other coloring-kinetic studies on glasses and other materials,^{9,1,5} will be developed only briefly here.

Fundamental to the model is the concept of a color center precursor, i.e. a site in the glass lattice which is capable of trapping charge carriers. In some cases the number of precursors may be changed by radiation damage processes. Very simple assumptions regarding the kinetics of the carrier trapping and radiation damage processes lead to growth curve equations of the form (1). If one assumes that: 1) P_{0i} is the initial precursor concentration of the i th type, 2) additional precursors are formed at a constant rate K_i during irradiation, 3) the rate of carrier trapping at precursors, during irradiation is proportional to the concentration of empty precursors with proportionality constant F_i , 4) the rate of thermal untrapping from filled precursors is given by the constant

U_i (normally $U = s \exp(-E/KT)$) and 5) the electron-hole recombination rate, which also removes trapped charges from precursors is R_i , one obtains

$$\frac{dN_i}{dt} = F_i(P_{oi} + K_i t - N_i) - (R_i + U_i)N_i \quad (3)$$

where N_i is the concentration of filled trapping sites of the i^{th} type. The simplest solutions of (3) are obtained by assuming that each of the i components is independent. The solutions, for $N_i = 0$ at $t = 0$, are

$$N_i(t) = N_{oi}(1 - \exp(-a_i t)) + N_{Li}T \quad (4)$$

If the observed absorption is assumed proportional to a linear combination of two different carrier trap concentrations N_1 and N_2 whose behavior is given by (4) growth curve expressions of the form (1) are obtained immediately.

This last assumption is extremely general and may be satisfied in several ways. Perhaps the simplest arises when the N_i are two distinct color centers, each with a separate absorption band. If these bands overlap, the observed absorption is simply the sum of the individual contributions at the measured photon energy. As an example of an alternative situation leading to the same observed kinetics, consider that the absorption of only a single type of center is observed, e.g. a trapped electron center. For each trapped electron there must exist in the glass a trapped hole. If the number of electron traps is large compared to the number of stable hole traps, the electron center kinetics will simply mirror the kinetics of hole trapping and untrapping. If there are two important hole traps, the observed absorption due to the electron center is given by

$$\alpha(t) = C_e N_e = C_{1h} N_{1h} + C_{2h} N_{2h} \quad (5)$$

where the C 's are constants. If N_{1h} and N_{2h} are two independent solutions of (3), the kinetics are indistinguishable from the two band case discussed above. At this point, it is not possible to determine if either of the two possibilities discussed above applies to the glass data. Preliminary attempts to resolve the growth kinetics into contributions from overlapping bands have been unsuccessful, possibly because all bands grow at nearly the same rate.

The relationships between the constants appearing in Equation (3) and the A_i and a_i and α_L coefficients are exhibited in Ref. (9). The dose rate dependence found for the a_i , or exponential coefficients, is in qualitative agreement with the expression

$$a_i = F_i + R_i + U_i \quad (6)$$

obtained in Ref. (9), if it is assumed that F_i and R_i are linear with dose rate. The data in Table 2 indicates that the U_i are not zero, which is consistent with the fact that the absorption decays when the irradiation is terminated. Without more complete knowledge of the values of the coefficients in Equation (3) critical comments cannot be made on either the dose rate dependence of the saturation level ($A_1 + A_2$) or the precise estimate of P_{oi} . For the strong bands in the u. v., one obtains a lower bound of $\sim 10^{17}/\text{cm}^3$ for P_{oi} , assuming an oscillator strength of unity.

Inasmuch as α_L was non zero the model indicates that defects are introduced by radiation damage processes and play a role in the coloring kinetics. This result does not rule out the possibility that impurity centers^(3,15) are responsible for the coloring. However, it does require that some of the observed centers are

stabilized or converted to precursors by radiation damage processes. Also, it is possible that the linear term in the growth curves may represent the critical portion of a third slowly growing saturating exponential.

As shown previously (1) decay kinetics of the type observed result from equations of the form:

$$\frac{dN_i}{dt} = -d_i N_i - \sum_{j \neq i} d_j N_j \quad (7)$$

The first term on the right represents the release of carriers from the center under observation while the second term results from the thermally activated release of oppositely charged carriers from other traps. If one assumes that the release of carriers from these traps obeys the usual equation(1), and that a constant fraction of these carriers recombine at the color center under observation, one immediately obtains a solution of (7) which is in agreement with the data described by Equation (?).

In the simplest case, the d_i coefficients would be independent of dose rate or total dose, while the initial values of N_i and N_j would depend upon the previous irradiation history. This behavior is roughly in accord with the results given in Table 3. In addition, the d_i values for the two short lived components are not markedly different from U_i values estimated by applying Equation (6) to the growth curve data. It should be made clear, however, that multiple decay components, as well as multiple growth components, can be attributed to overlapping bands.

Summary

The results described above represent, as far as can be determined, the first study of radiation induced color center formation in glasses utilizing measurements made during and after continuous electron irradiation. Previously reported are similar studies utilizing absorption measurements made during and after gamma-ray irradiation. Aside from the obvious difference between radiations, these electron irradiations were made at dose rates between 10 and 100 times those used in the gamma-ray studies. Yet, qualitatively the results are quite similar.

1. The radiation induced absorption spectra consist of the superposition of one or more gaussian shaped absorption bands and an "edge-shift" which can be described by an additional band whose peak usually lies at the high energy side of the edge.
2. When measured during irradiation the color center growth curves can be described accurately by an expression, Equation (1), consisting of one or more saturating exponentials and a linear term. The latter term may represent the initial part of a very slowly growing saturating exponential.
3. When the irradiation is terminated the coloring immediately begins to decrease. During such decay, the color center concentration vs. time curves can be very accurately decomposed into one or more exponential components, i.e. is represented by Equation (2).
4. Color center growth studies at different dose rates, and decay measurements made after the irradiation is terminated, lead to the following conclusions:
  - a) The higher the dose rate the more rapid the approach to a "plateau" or saturation of the coloring.
  - b) Also, as the dose rate increases, the greater the short lived fraction of the total observed decay.
  - c) While theoretical

kinetic treatments are discussed below, the data shows that variables representing the rate of charge capture by traps (color center formation rate), the rate of trap introduction by the incident radiation (perhaps by atom movement processes) and the electron-hole recombination rate all increase with increasing dose rate.

One of the simplest possible kinetic theories^{1,9} which appears to apply to KCl and glasses, predicts that some of the parameters should be linear functions of the dose rate. The available data suggests that there is rough agreement with this behavior. However, really reliable testing of these predictions requires more data than available now. A large dose rate range should be used, greater accelerator beam stability should be employed, and additional experimental variations should be employed. For example, all decay measurements were made at roughly the same color center concentration irrespective of dose rate or irradiation time; additional measurements should be made at equal doses and/or irradiation time.

In conclusion, the data presented here appears to provide strong evidence to conclude that it is essential to make measurements during irradiation to unequivocally ascertain the growth and decay kinetics of radiation induced color center, and other defects, which are unstable at bombardment temperatures.

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Table 1

Peak energies and widths of the gaussian components in the absorption spectra of NBS 710 (70.5% SiO₂, 8.7% Na₂O, 7.7% K₂O, 11.6% CaO) and NBS 711 (46% SiO₂, 45.3% PbO, 7.5% Na₂O, 5.6% K₂O) glasses.

Glass	Region	Energy, eV	Full Width eV
710	Visible	1.93	0.40
	u. v.	4.27	2.47
	"edge"	4.50	1.27
711	Visible	1.60	0.57
	u. v.	3.11	1.56
	"edge"	4.42	1.52

Table 2

Growth curve parameters at 2.6 eV in NBS 710 glass from Equation (1), for several dose rates.

Dose rate (R/H) X 10 ⁻⁶	3.72	2.20	1.51	0.81
A ₁ (cm ⁻¹)	3.48	3.83	3.18	3.05
a ₁ X 10 ⁴ sec ⁻¹	13.5	9.54	5.88	3.10
A ₂ (cm ⁻¹)	2.72	1.64	1.90	1.92
a ₂ X 10 ³ sec ⁻¹	4.57	3.23	2.07	1.30
a _L X 10 ⁵ cm-sec ⁻¹	4.02	4.04	2.15	1.85
ΣA _i (cm ⁻¹)	6.20	5.47	5.08	4.97

Table 3

Decay curve parameters at 2.6 eV in NBS 710 glass from Equation (2), for several dose rates.

Dose rate (R/H)X10 ⁻⁶	3.72	2.80	2.20	1.51	0.81
D ₁ (cm ⁻¹)	3.55	3.89	3.52	3.28	3.67
d ₁ X 10 ⁶ sec ⁻¹	4.75	3.20	4.88	4.20	3.72
D ₂ (cm ⁻¹)	1.62	1.62	1.43	1.24	1.19
d ₂ X 10 ⁴ sec ⁻¹	1.75	1.30	1.47	1.30	1.31
D ₃ (cm ⁻¹)	1.17	1.13	0.85	0.78	0.49
d ₃ X 10 ³ sec ⁻¹	1.98	1.49	1.90	1.21	1.20

Fig. 1. The experimental facility for measuring absorption and luminescence during and after electron irradiation. See text for details.

Fig. 2. The control and data acquisition system incorporated in the double beam spectrophotometer is illustrated. At each wavelength, specified by the computer, signals from the photomultipliers are synchronously processed at the interface and read into four 256 point arrays in computer memory. Two arrays contain sample beam information and two reference beam information. One member of each pair of arrays contains information recorded during the "dark" portion of the light chopping cycle, allowing both correction of the absorption spectra for luminescence and measurement of the sample luminescence itself.

Fig. 3. "3-D" plot of radiation induced absorption in NBS 710 glass. The measurements were made both during ("beam on") and after ("beam off") 1.5 MeV electron irradiation at room temperature. The individual spectral scans shown are made at least once every 40 seconds during and after the irradiation. For clarity every second scan has been omitted. The u.v. and visible portions of the absorption spectrum were obtained in different experiments and joined by computer processing. The data is subsequently used to construct absorption spectra and growth curves at specific photon energies, which are used in further analysis.

Fig. 4. Radiation induced absorption spectrum of NBS 710 glass. The total absorption is resolved into gaussian bands by a computerized least squares procedure. The spectrum contains two bands and an "edge shift" approximated by a third gaussian. The line through the data points is the computed superposition of the individual components shown.

Fig. 5. Radiation induced absorption spectrum of NBS 711 glass. The total absorption (upper curve) is resolved into gaussian bands (lower curves) which are qualitatively similar to those in Fig. 4: a weak visible band, a strong u.v. band and an "absorption edge shift."

Fig. 6. Growth curve of absorption at 4 eV vs. irradiation time in NBS 710 glass. The data can be accurately fitted by the expression

$$\alpha(t) = \sum_{i=1}^2 A_i (1 - \exp(-a_i t)) + \alpha_L t$$

shown as a solid line through the data.

Fig. 7. The figure shows the resolution of the growth curve data of Fig. 6 into two saturating exponential components, after first resolving the linear term. The remaining data (upper curve) is plotted on a semi-log scale and resolved into two components represented by straight lines. The slopes and intercepts of these lines determine the a 's and A 's used to compute the solid line in Fig. 6.

Fig. 8. Decay curve of absorption at 4 eV following irradiation in NBS 710 glass. The data can be accurately fitted by the expression

$$\alpha(t) = \sum_{i=1}^3 D_i \exp(-d_i(t-t'))$$

shown as the solid line through the data.

Fig. 9. The figure shows the resolution of the growth curve data of Fig. 8 into three decaying exponentials. The data is plotted on a normalized semi-log scale (upper curve) and resolved into three components represented by straight lines in the figure. The slopes and intercepts of these lines determine the d 's and D 's used to compute the solid line in Fig. 8.

Fig. 10. Growth curves of the absorption at 2.6 eV in NBS 710 glass at different dose rates. The dose rates are given at the right of each curve. The curves were generated by plotting the best fit analytic expressions for $\alpha(t)$ as given by Equation (1)

Fig. 11. Normalized decay curves of the absorption at 2.6 eV in NBS 710 glass following irradiation at different dose rates. The curves were generated from the best fit expressions for $\alpha(t)$ as given by Equation (2), normalized to unity at the origin.