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Radiation Damage Studies in Solids;
Nuclear Resonance Absorption Technique

by

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Department of Physics

Brown University

Providence 12, Rhode Island

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Table of Contents

Abstract

Nuclear magnetic resonance (NMR) studies of neutron-irradiated LiF crystals have resulted in the detection of colloidal lithium metal and molecular fluorine gas. Quantitative determinations of the amount of damage and the behavior of the displaced metal and halide ions appear to be possible.

The defect density in neutron-irradiated KBr has been checked as a function of dosage and annealing time. A low-intensity satellite on the low-frequency side of the normal bromine NMR in unirradiated KBr has been detected and studied as a function of the magnitude and orientation of the magnetic field. Second-order quadrupolar effects arising from the strain fields of dislocations are probably responsible for the effect.

Experimental and theoretical studies of the preferential orientation of dislocations in single crystals of NaI and KI have progressed. With increased spectrometer sensitivity which detects the broad "wings" or "tails" on the I^{127} NMR, integrated line intensities are now more meaningful.

NMR studies of proton-irradiated (160 Mev) crystals of NaI, RbI, RbBr, CsI, CsBr and other alkali halides have been started.

The Li^7 and B^{11} resonances in Li_2O - B_2O_3 glasses have been studied as a function of the glass composition. Departures from the behavior of the Na_2O-B_2O_3 system are noted. The Li^7 resonance displays satellite transitions arising from nuclei with electrical quadrupole coupling constants of an average value of 187 Kcps. Neutron radiation damage has been detected in the Li_2O - B_2O_3 glasses by NMR techniques.

Gamma ray radiation effects in hexamethylenetetramine reveal a hyperbolic tangent dependence on radiation dosage and emphasize the importance of recombination processes in molecular solids.

I. Report of Work Completed or in Progress

The major research effort during the past year has been concentrated in seven areas:

1) studies of radiation damage in alkali halides;
2) studies of the effects of dislocations in alkali halides;
3) investigation of the structure of glasses;
4) study of radiation damage in glasses;
5) extension of the neutron radiation damage studies to damage caused by fast charged particles;
6) investigation of gamma ray radiation effects in organic solids and use of gamma radiation to make possible nuclear magnetic resonance investigations of certain inorganic solids;
7) improvement of spectrometer sensitivity;
8) completion of several phases of proposed work and preparation — including theoretical analyses -- of resulting papers for publication. Six papers have been completed and one other brought almost to completion during the period covered by this report.

The principal results obtained in the first six areas listed above are summarized in the following sections.

(1) Alkali Halides

(a) LiF

Nuclear magnetic resonance (NMR) studies of LiF samples subjected to neutron dosages of the order of 10^{18} nvt have revealed [(1)] the presence of constituent nuclei in environments other than those of the normal lattice sites. These nuclei produce narrow lines which are superimposed on the normal broad lines.

The more narrow F^{19} line is shifted about 0.07% to a frequency higher than that of the broad line. This is in agreement with the shift [(2)] expected for molecular fluorine gas on the basis of a larger degree of covalent bonding than exists in the ionic crystal of LiF. Approximately 7% of the fluorine nuclei contribute to this line for a radiation dosage of 1×10^{19} nvt.

The narrow lithium line is shifted by 2.8% to a frequency higher than that of the broad line. This agrees with the 2.49% shift [3] observed by Knight in lithium metal.

We conclude that the narrow Li^7 and F^{19} lines arise from colloidal lithium metal and molecular fluorine gas, respectively. These findings are consistent with observations [4,5] arising from x-ray work.

Further studies of the lithium and halogen resonances, as functions of radiation dosage and post-irradiation annealing, are in progress on both single crystals and powders of LiF, LiBr, and LiI. Higher neutron dosages (perhaps 10^{20} nvt of fast neutrons) may yield the same type of information from alkali halides other than lithium halides. Quantitative determinations of the amount of damage and the behavior of the displaced metal and halide ions appear to be possible.

These studies are being carried out in correlation with electron-spin resonance (ESR) investigations (under Contract AT(30-1)-2024) of neutron-irradiated LiF and other materials. The detection and study of metallic lithium and molecular fluorine in these LiF samples may help clarify the origin of the multi-component (>27) ESR pattern previously reported [6] in neutron-irradiated LiF but not explained.

We have now set up, with the cooperation of the Department of Chemistry, a program of optical studies of the color centers in all of our irradiated samples.

(b) RbI, RbBr

These materials are of particular interest since the Rb^{85}, Rb^{87}, Br^{79}, Br^{81}, and I^{127} nuclei all have large electrical quadrupole moments and the ions have large Sternheimer [7] antishielding factors. The NMR responses are consequently sensitive to crystal defects and the amount and type of damage in irradiated specimens can be checked by comparison of data from the three (RbI) or four (RbBr) NMR responses.

Samples of RbI which have received total neutron dosages of 1.6 — 1.8×10^{19} nvt are at present being kept at dry ice temperature in a shielded container. Within two months the activity should decrease to a safe level and NMR, ESR, and optical studies will be started.

Samples of RbI were included in an initial selection of alkali halides which received 10^{11} protons/cm^2/sec for one hour in the 160 Mev proton beam of the Harvard University Cyclotron.

The irradiated portions exhibited heavy coloring but all of the alkali halide samples were misaligned in the beam and were only partially irradiated. A second group has been irradiated and are under investigation.

RbBr has been secured from Korth of Germany and is under investigation.

(c) KBr

The initial investigations of this material have been reported [8] in a paper completed during the past year. That paper presents the background for NMR studies of radiation damage and details the method of analysis employed. The original KBr samples had annealed for two months at room temperature. Data from two newly irradiated samples yield, from analysis of the Br^{81} NMR,

$$\Delta I = 0.30 \pm 0.02$$

and

$$\rho_r = 7.8 \times 10^{-4} \text{ defects/atom}$$

for a fast neutron flux of 3.5×10^{17} nvt at room temperature. ($\Delta I = \frac{I_o - I}{I_o}$ where I_o is the NMR intensity in the "perfect" crystal and I is the intensity in the irradiated crystal. ρ_r is the defect density deduced from the measured ΔI using the model [8] of randomly distributed defects. The fast neutron flux is taken as $\frac{1}{10}$ of the total integrated flux and assumed to be 1 Mev neutrons.)

Another KBr sample which received a fast neutron flux of 1.4×10^{17} nvt at room temperature yielded

$$\Delta I = 0.17$$

and

$$\rho_r = 4.7 \times 10^{-4} \text{ defects/atom.}$$

These results should be contrasted with the earlier [8] values

$$\Delta I = 0.27$$

$$\rho_r = 6.9 \times 10^{-4} \text{ defects/atom}$$

$$\text{fast flux} = 6.6 \times 10^{17} \text{ nvt}$$

The number of defects per atom per incident fast neutron is, for the three cases:

Sample	defects/atom/fast neutron
A	$1.0^{+} \times 10^{-21}$
B	2.2×10^{-21}
C	3.3×10^{-21}

A = original samples which annealed at room temperature for two months after irradiation and before NMR studies.

B = two samples kept at dry ice temperature after irradiation and brought to room temperature just before NMR studies.

C = one sample handled as in B.

The difference in values between cases B and C is not important since the agreement is well within the uncertainties arising in flux measurements and within the theoretical analysis.

A set of KBr crystals which received 160 Mev proton irradiation is under investigation with NMR, ESR, and optical techniques.

A low intensity "satellite" has been found on the low frequency side of the normal Br^{81} resonance (NMR) in unirradiated crystals of KBr. The position and intensity depend on orientation of the crystal in the magnetic field and on the field strength. Second-order quadrupolar effects arising from the strain fields of dislocations are suspected and a thorough experimental and theoretical study is in progress for both Br^{79} and Br^{81}.

(d) NaI and KI

A high degree of preferential orientation of dislocations in single crystals of NaI and KI has been established [8,9]. Because of the consequent large intensity anomalies yielding essentially perfect-crystal intensities for certain orientations of magnetic field, the NMR responses in these crystals are particularly susceptible to radiation damage effects. Consequently, the study of 160 Mev proton irradiation damage has been concentrated in large part on NaI to date. These initial investigations -- involving both intensity and spin-lattice relaxation time (T_1) measurements -- apparently do not yield the large defect influence on line intensity reported for NaCl [10]. This disagreement is being checked thoroughly with further investigations of NaCl, NaI, and other alkali halides. The studies involve NMR data on line intensity and T_1 values, ESR data on concentration of paramagnetic centers, and optical studies of the color centers.

The preferential orientation [8,9] of dislocations in NaI and KI is receiving further experimental and theoretical treatment. Spectrometer sensitivity has been further increased, yielding very broad "tails" or "shoulders" on the I^{127} resonance in KI at all orientations of the magnetic field, $\vec{H}_o$, and in NaI at all orientations of $\vec{H}_o$ other than along [1,0,0] type directions. Numerical integrations of the derivative-type experimentally recorded NMR curves are now much more reliable. Comparison of such integrated intensity curves (as a function of $\vec{H}_o$ orientation in the (0,0,1) plane) for NaI yields the same result as the earlier [8] plot of $D_{p-p}(\delta\nu)^2$ as a function of angle (D_{p-p} is the measurement from the positive maximum to the negative maximum in the the derivative of a line; $\delta\nu$ is the separation in frequency between these two maxima in the derivative trace). $D_{p-p}(\delta\nu)^2$ plots for I^{127} in KI were not reported [8] in light of their negligible meaning for NMR responses showing broad "tails" or "shoulders". Numerical integration of the I^{127} derivative curves for KI as a function of $\bar{H}_o$ orientation in the (0,0,1) plane does not yield either a constant intensity value or the narrow intensity anomalies found in the I^{127} NMR of NaI. Over certain ranges of $\bar{H}_o$ orientation, satellite splittings are obviously being pushed out into the noise by quadrupolar effects and are unobservable (i.e. the observed "shoulders," which are most apparent [8,9] when $\bar{H}_o$ is along [1,0,0] type directions, extend well out into the noise at most orientations of $\bar{H}_o$.)

Progress is being made in extending the theory of the effect of oriented dislocations on NMR responses to all $\bar{H}_o$ orientations, partial preferential alignment of dislocations, and dislocations more complex than the simple edge and screw types.

(e) CsBr, CsI, and NaF

CsI and RbBr are of particular interest because they are systems of almost equal masses (Cs^{133} and I^{127}; Rb^{85} or Rb^{87} and Br^{79} or Br^{81}). Present theories for the number of atomic displacements caused by each bombarding particle assume a lattice of equal masses (see, however, reference 11).

The cesium compounds cannot be investigated after even very small neutron dosages because of the resultant activity. Hence, we have commenced study of crystals which have received 160 Mev proton irradiation. These soft and difficult crystals are not oriented by the supplier (Harshaw Chemical Company), but after much experimenting with techniques of handling and crystal polishing, we are now able to orient them with standard x-ray methods.

Pre-irradiation of these crystals with up to 50 megaroentgens of cobalt gamma rays yields very suitable bromine and iodine nuclear magnetic resonances. Unirradiated crystals give difficulty in this respect [12].

The Na^{23} and F^{19} resonances in NaF are under investigation in crystals which have received 10^{18} nvt of neutron irradiation. Dosages up to 10^{20} nvt are being secured at the Materials Testing Reactor in order to follow the behavior of the displaced ions, as in the case of LiF.

(2) Glasses

A study[13] has been made of the Li^7 and B^{11} NMR in the Li_2O - B_2O_3 glass system at room temperature as a function of Li_2O molar concentration up to 25%. Beyond this concentration the system forms a crystalline mass instead of a glass. As in the Na_2O - B_2O_3 system [14], it is possible to follow the change from threefold to fourfold bonding of the boron atoms as the Li_2O concentration is increased. However, in the Li_2O - B_2O_3 system, the initial rate of change of the boron coordination as a function of alkali oxide content is approximately four times as large as in the Na_2O - B_2O_3 system. Further, the fraction of four-coordinated boron atoms saturates at a lower percentage of the modifier (Li_2O in this case). This behavior may be attributable to the fact that the lithium ion is smaller than the sodium ion, thereby allowing a closer packing of the boron-oxygen network.

The Li^7 resonance has been observed in samples ranging in composition from 2.5 to 25% Li_2O. Weak and broad first-order satellites on the Li^7 NMR in the samples containing 20 to 25% Li_2O are observable. An average quadrupole coupling constant for the nuclei contributing to these satellite "wings" is 187 Kcps. We are attempting to determine from these data whether the lithium ions go into the glass in a random manner or are primarily in ordered positions. These two alternatives are currently of considerable interest to workers in the field of glass structure.

Several samples of 20% Li_2O content were irradiated in a high thermal neutron flux region of the Brookhaven National Reactor to take advantage of the high cross sections of B^{10} and Li^6 for thermal neutrons. The resultant fission products yield a high degree of radiation damage. Samples receiving one and two duty cycles of irradiation showed reductions of 5% and 10%, respectively, in the intensity of the four-coordinated boron NMR. With the understanding gained of glass structures and establishment of the ability to observe radiation damage effects in glasses by NMR techniques, we are proceeding with a program of damage studies as a function of glass composition and radiation dosage. Some theoretical predictions are available but much more work in this area is necessary. The NMR studies are being supplemented with ESR and optical investigations.

We have begun work on the K_2O - B_2O_3 glasses and aluminum-containing glasses.

(3) Lithium Compounds, Boron Compounds, and Molecular Solids

Successful NMR studies of the Li^7 resonance in LiOH, LiOH • H_2O, $LiNO_3$, $LiNO_3$ • $3H_2O$, $LiIO_3$, Li_3PO_4, Li_2TiO_3, and Li_2ZrO_3 have been carried out [15]. Investigations of neutron radiation damage in these ionic compounds is in progress by NMR, ESR, and optical techniques.

The structure of crystalline boron is under investigation through study of the B^{11} NMR in small single crystals. Neutron radiation damage studies of boron carbide, boron nitride, and several metal borides are in progress. Successfull efforts during the past year to increase spectrometer sensitivity will greatly facilitate these investigations.

One phase of the work with irradiated molecular solids has been completed with a study of neutron-irradiated hexamethylenetetramine. Results of interest concern the dependence of damage on dosage and the importance of recombination processes in this non-aromatic compound. Since a paper [16] giving experimental procedure, data, and conclusions is available as a preprint from the author of this report and has been submitted to the "Journal of Chemical Physics" for publication, only the abstract is reproduced here:

"The electron spin and N^{14} pure quadrupole resonances of eleven samples of $(CH_2)_6 N_4$ have been studied as a function of Co^{60} gamma ray irradiation. The results show that the number of free electrons in the samples increases as the hyperbolic tangent of the amount of radiation received. As expected, the pure quadrupole resonance intensity decreased as the number of free electrons increased. This decrease is due to a broadening of the resonance which is characterized by a linear decrease of T*, the line width parameter, with the number of free electrons. Finally, the dependence of the intensity on the number of charged defects created by the gamma rays is found to be different from that found previously for neutral impurity molecules."

II. Equipment and Facilities

Three complete NMR spectrometers, with associated magnets, are now in operation in the laboratory. Two systems are being used full-time for radiation damage studies under Contract AT(30-1)1880. Part of the research effort employing the third system, though supported by other grants, involves structure and electron distribution studies of materials which are of interest for radiation damage studies (selections of lithium and boron compounds, glasses, and semiconductors).

Spectrometer sensitivity has been further increased since the preceding report, NYO - 7624, was issued.

Additional equipment for low and high-temperature studies has been completed.

Through the cooperation of the Departments of Chemistry and Applied Mathematics, excellent facilities for optical and x-ray studies have been made available to the personnel of this contract.

The electron-spin resonance equipment of Contract AT(30-1)2024, and the cooperation of the research assistant personnel of that group, have greatly facilitated the nuclear magnetic resonance studies of radiation damage.

III. Personnel

Professor P. J. Bray, the principal investigator, is devoting at least $\frac{1}{4}$ time to the project during the academic year under support from Brown University funds. During the period between 1 June and 1 September 1958, at least the equivalent of one full-time month was spent in work on the project.

Two research assistants, P. J. Ring and J. G. O'Keefe, were employed at a 5/8 time rate during the academic year and for three summer months of 1958. The work of both men on the contract has been highly satisfactory.

During 1958, Mr. P. A. Casabella has worked on radiation damage problems in conjunction with Mr. Ring and Mr. O'Keefe. His financial support was provided by an Ohio Oil Company Fellowship awarded through Brown University. Mr. Casabella's investigations added approximately a $\frac{1}{2}$ time research assistant to the project without drawing funds from the contract. This fortunate arrangement has resulted in a completed study [16] of a gamma-irradiated molecular solid. Mr. Casabella is now finishing his Ph.D. thesis and will remain at Brown in the NMR group as a Research Associate. His principal work will be in other NMR areas under support from the National Science Foundation, but he will continue his cooperation with the radiation damage project.

IV. Personnel Activities and Publication of Results

Abstracts

1) "Nuclear Magnetic Resonance in Pile-Irradiated LiI," J.F. Hon and P.J. Bray, Bull. Am. Phys. Soc. II, 2, 344 (1957).
2) "Nuclear Quadrupole Coupling Constants of Li^7 in Lithium Compounds," P.J. Bray and J.F. Hon, Bull. Am. Phys. Soc. II, 2, 345 (1957).
3) "B^{11} Nuclear Magnetic Resonance in Planar Hexagonal Compounds," A.H. Silver and P.J. Bray, Bull. Am. Phys. Soc. II, 2, 345 (1957).

4) "Pure Quadrupole Resonance of Gamma Irradiated Hexamethylenetetramine," P. A. Casabella, A.H. Silver, and P.J. Bray, Bull. Am. Phys. Soc. II, 2, 384 (1957).

5) "Anomalous Nuclear Magnetic Resonance of B^{11} in Boron Carbide," A.H. Silver and P.J. Bray, Bull. Am. Phys. Soc. II, 2, 387 (1957).

6) "Nuclear Magnetic Resonance of B^{11} and Li^7 in the Binary Glass $Li_2O - B_2O_3$," A. H. Silver, J.G.O'Keefe, and P.J. Bray, Bull. Am. Phys. Soc. II, 3, 21 (1958).

7) "Nuclear Magnetic Resonance Studies of Pile-Irradiated NaI and KI," P.J. Bray and J.F. Hon, Bull. Am. Phys. Soc. II, 3, 22 (1958).

8) "Nuclear Magnetic Resonance and Dislocations in NaI and KI," J.F. Hon and P.J. Bray, Bull. Am. Phys. Soc. II, 3, 22 (1958).

9) "Nuclear Magnetic Resonance Studies of Radiation Damage," P.J. Bray, Bull. Am. Phys. Soc. II, 3, 154 (1958).

Papers

1) "Nuclear Quadrupole Coupling Constants of Li^7 in Lithium Compounds," J.F. Hon and P.J. Bray, Phys. Rev. 110, 624 (1958).

2) "Nuclear Magnetic Resonance Absorption in Glass. I. Nuclear Quadrupole Effects in Boron Oxide, Soda-Boric Oxide and Borosilicate Glasses," A.H. Silver and P.J. Bray, J. Chem. Phys. 29, 984 (1958).

3) "Nuclear Magnetic Resonance Studies of Neutron-Irradiated Alkali Halides," J.F. Hon and P.J. Bray. Accepted for publication in the "Journal of the Physics and Chemistry of Solids."

4) "Nuclear Magnetic Resonance Studies of Dislocations in NaI and KI," J.F. Hon and P.J. Bray. Completed. To be submitted for publication.

5) "Li^7 and F^{19} Nuclear Magnetic Resonances in Neutron-Irradiated LiF," P.J. Ring, J.G. O'Keefe, and P.J. Bray. Submitted for publication in the "Physical Review Letters."

6) "Electron Spin and Pure Quadrupole Resonance Studies of Co^{60} Gamma Ray Irradiated Hexamethylenetetramine," P.A. Casabella and P.J. Bray. Submitted for publication in the "Journal of Chemical Physics."

A paper concerning nuclear magnetic resonance (NMR) studies of boron carbide (A.H. Silver and P.J. Bray) has been completed and will be submitted for publication. A paper concerning NMR studies of boron nitride, titanium boride, zirconium boride, and certain borofluorides and borohydrides is in progress (A.H. Silver and P.J. Bray).

References

(1) "Li^7 and F^{19} Nuclear Magnetic Resonances in Neutron-Irradiated LiF," P.J. Ring, J.G. O'Keefe, and P.J. Bray. Submitted to the "Physical Review Letters."

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(9) "Nuclear Magnetic Resonance Studies of Dislocations in NaI and KI," J.F. Hon and P.J. Bray. To be submitted for publication.

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(15) J.F. Hon and P.J. Bray, Phys. Rev. 110, 624 (1958).

(16) "Electron Spin and Pure Quadrupole Resonance Studies of Co^{60} Gamma Ray Irradiated Hexamethylenetetramine," P.A. Casabella and P.J. Bray. Submitted to the "Journal of Chemical Physics" for publication.

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