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WAYNE STATE UNIVERSITY

Department of Physics

TECHNICAL PROGRESS REPORT

on

Electron Paramagnetic Resonance Studies of Radiation Effects In Solids and Chemical Compounds

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I. Introduction

Respectfully submitted herewith is a technical progress report of the work entitled "Electron Paramagnetic Resonance Studies of Radiation Effects in Solids and Chemical Compounds", conducted under AEC Contract No. AT(11-1)-1054. The contract work is effective for the period September 16, 1968 through September 15, 1969. The present technical progress report covers the work period of June 1, 1968 through May 31, 1969. A previous technical progress report (COO-1054-32), which was submitted on May 31, 1968, covered a one-year period prior to June 1, 1968.

As planned in the Proposed Technical Programs which were submitted on May 31, 1968 and subsequently approved (PTP-68), experimental studies have been conducted on the following three groups of materials:

(1) Alkali halides

(2) Phosphorus materials, and

(3) Superconducting materials.

For the most part, samples have been irradiated in beams of x-rays and gamma-rays for varying dosage at temperatures controlled near or below 300°K. The investigations of these samples have been performed between 2°K and 300°K, depending upon the need of individual investigations. If necessary, samples were also subjected to optical and/or thermal bleaching treatments. Interesting and significant results have been, and are being, obtained from these study programs.

The over-all efficiency of the capital equipment and supporting facilities have been improved by purchasing commercially or by building new pieces of equipment in this laboratory. Particularly significant are (i) the addition of a new 9-inch Varian magnet and its power supply, (ii) the completion in assembling a variable-temperature cryogenic dewar, and (iii) a mechanical manifold for the dewar to facilitate x-irradiation of superconducting materials held in the dewar near 4°K.

In personnel, Dr. C. Aseltine, the post-doctoral research associate supported under the contract for the 1967-68 year formally joined the U. S. Army Ballistic Research Laboratory, Aberdeen Proving Ground, Maryland, upon completion of his one-year appointment under the contract. A new post-doctoral research associate joined the present group. Six graduate students have been participating in the contract work in various capacities. One is near completion of his Ph.D. thesis; two other graduate assistants successfully passed the Ph.D. qualifying examination; and one graduate student is preparing for the Ph.D. qualifying examination.

A summary of individual study programs is given in Section II. Equipment, facilities, and the personnel activities are described in the following sections, III and IV respectively. A list of publications is given in section V. Copies of publication materials are also being appended to this progress report.

II. Report of Work Completed or in Progress

(1) Alkali Halides

As planned in the PTP-68, the delayed luminescence of single crystal samples subjected to extremely weak radiation treatments at controlled temperatures has been investigated in order to understand the conversion mechanism of incident photons into those photons of the delayed luminescence as well as the relative mobilities of isolated defects and charge-neutral defect pairs. The results have been briefly summarized in the following abstract (COO-1054-43):

C. Manilerd and Y.W. Kim, "Delayed Luminescence in Alkali Halides Under F-bleaching"

The abstract has been presented at the American Physical Society meeting, Washington, D.C., 28 April through 1 May, 1969. A copy of the abstract plus the text of the presentation is appended to the present progress report. The details of this work are being summarized, at the moment, into a manuscript for possible publication.

In short, the experimental results show that the delayed luminescence in the very early stage of radiation-defect formation is quite distinct from the direct luminescence to be observed in the F to M conversion by F-bleaching as reported recently by Compton and his co-worker.[1] The delayed luminescence is the same for the common halide ions, and does not appear to be due to the F_3^+ as suggested recently by other people.[2] A third possibility is V_2 centers being responsible for the delayed luminescence. However, the experimental results are not compatible with this speculation. In the case of LiF the delayed luminescence was observable only from a group of samples which had previously been heavily gamma-irradiated, while it was not observed from un-irradiated samples. This fact suggests that the delayed luminescence probably has something to do with positive ion vacancies (α centers), since it has been believed that gamma-irradiation of LiF produces large concentrations of α centers.[3] Comparative analyses of similar experimental results obtained from various alkali halides favor the following model for the delayed luminescence. F light photo-ionizes F centers and some of the halogen ions, thus producing negative-ion vacancies (α centers), electrons and holes. The holes are probably trapped to charge-neutral pairs of positive-ion and negative-ion vacancies. This latter configuration is referred to as Seitz's H_s centers.[4] The delayed luminescence then is the consequence of the recombination of the electron with the hole of the H_s center.

In order to consolidate the proposed model, an interesting experiment is in progress with respect to the so-called Dember effect[5] in these x-irradiated samples. Experimentally, an x-irradiated sample containing F-centers is suspended at an end of a torsion balance which is held vertically. The sample is placed at the cross-point of two mutually perpendicular electric fields which are sinusoidal at 60 cps and have a 90° phase difference. When the F-light sweeps across the sample, electrons are liberated from F-centers, leading to a non-uniform distribution of excess charges across the sample. This leads to the formation of an effective dipole moment in the sample. The external time-dependent electric fields then act on the dipole moment, thereby generating an effective torque. This torque would rotate the sample plus the torsion balance until it is counter-balanced by the torsion of the balance. Preliminary data in this laboratory seem to necessitate further work on this effect. In this respect, it is interesting to note that related studies on the Dember effect are being currently made elsewhere.[6-7]

The content of the abstract and the results of some additional experiments including Dember effect are to constitute the major portion of the Ph.D. thesis for Mr. Chaleo Manilerd, who has been supported under the contract. The thesis is expected to be completed late in the fall of 1969. Most parts of the experimental set-up employed for the delayed luminescence and the Dember effect have been designed, constructed, and assembled successfully in this laboratory.

(2) Phosphorus Materials

Main efforts were made successfully to understand the apparent failure of the Kroeger's theoretical model[8] of the luminescence in the case of $CaWO_4$ subjected to sustained x-irradiation. The results and their interpretation are summarized in the following thesis (COO-1054-44):

Glen P. Double, "Luminescence in Calcium Tungstate".

In short, the thesis work deals with systematic investigations of direct luminescence under sustained x-irradiation, thermal luminescence following the x-irradiation at 77°K and possible photoconductivity during the x-irradiation. The data pertaining to the direct luminescence and thermal luminescence are carefully correlated in order to extract basic information relevant to the operation of the Kroeger's model. The photoconductivity data turned out to be not necessarily consistent among different runs of experiments. The results showed that, under x-irradiation, at least two or more types of luminescent defects are simultaneously produced. The temperature dependence of individual luminescences is different, and all of them contribute to the measured luminescence of the sample. Accordingly, the over-all temperature dependence of the resultant luminescence is not necessarily to be accounted for by the Kroeger's model. A copy of the thesis is being appended to this progress report.

Subsequent efforts have been made with little success for the purpose of understanding the apparent inconsistency of the x-ray-induced photoconductivity data. Possible roles of the space charge, the Dember effect, and the contact potentials are currently being examined in this laboratory. In this respect, it is significant to note that other investigators of the same phosphor have recently reported similar difficulties in understanding the thermally-induced electric conductivity of their samples.[9]

(3) Superconducting Materials

As planned in the PTP-68, three mutually correlated study programs are being developed. One of them deals with microwave interactions of type II superconducting materials in the mixed state, the second with electron tunneling properties through superconducting junctions, and the third with DC interactions of type II superconducting materials. These study programs are being conducted with controlled x-irradiation near cryogenic temperatures. Basic equipment required for conducting these study programs have been successfully assembled during the past year. Most pieces of the equipment have been calibrated and/or sensitivity-checked and are fully operational. The others require further modifications, which are currently being made. The results of these investigations so far have been encouraging. Brief summaries are given in the following:

(i) Microwave Studies

Thin film samples of superconducting tin have been employed for this study program. Tin as a bulk matter has the superconducting transition temperature close to 3.72°K. When prepared in the form of thin films of thickness between 200 Angstrom to 2,000 Angstroms, these samples exhibit the type II transition through the mixed state in an external magnetic field. The mixed state corresponds to a partial penetration of the magnetic flux through the sample below the transition temperature.[10] The flux penetration is accomplished by means of quantized magnetic fluxoids. The core of each fluxoid contains the normal conduction electrons, and a superconducting current surrounds the core. The reason the thin film samples are employed is to find the microwave electric power dissipation in these normal core electrons as functions of controlled defects including the radiation defects and temperature.

Experimentally, the surface of a thin film sample is placed in parallel with the microwave electric field of the cylindrical cavity operated in the TM_{011} mode at the frequency of 9.6×10^9 cps. Of course, the sample plus the cavity are placed in a metallic helium dewar. An external steady magnetic field is applied perpendicularly to the film. The magnetic field is modulated with variable amplitude and frequency. The temperature of the sample is controlled near 1.5°K, corresponding to a reduced temperature of approximately .4. The microwave (power dissipation) signal is phase-detected at the modulation frequency. When the external magnetic field is scanned at a controlled rate from below the lower critical field (H_{c1}, a few gauss) to above the upper critical field (H_{c2}, approximately 200 gauss), the sample goes through the type II transition from the perfect superconducting state through the mixed state to the normal state. In the mixed state, the number of quantized fluxoids increases (decreases) as the magnetic induction increases (decreases). The phase-detected microwave signal is therefore expected to reflect the microwave interaction of the normal electrons of the fluxoids, thus revealing superconducting transition characteristics of the sample. The defect concentration has been varied from 10 parts per million (ppm) to 1,000 ppm.

Two very interesting and strange phenomena have been observed. First, the polarity of the detected signals for an increasing magnetic field is opposite to that for a decreasing magnetic field. This opposite polarity means the phase of the detector output has 180° difference between the two cases. For convenience, this change in the phase is referred to as phase reversal. Secondly, when one scans the magnetic field from H_{c1} up to a certain value and then suddenly reverses the sign of the field scanning rate, dH/dt, where H and t are the magnetic field and time respectively, the signal reverses its phase immediately. Similar phase reversal is also noticed when one scans the magnetic field from above H_{c2} to a lower field and then suddenly reverses the sign of dH/dt. These signals show very intricate dependencies on the rate of field scanning, and on the field modulation. If the scanning rate is either too small or too large, the signal would be very hard to observe. An optimum scanning rate used in the present experiment is approximately 1 gauss per second. Likewise, an optimum modulation frequency was found to be between 200 cps and 400 cps. It has been found that the amplitude of the time rate of change of the modulating magnetic field has to be larger than the optimum rate of magnetic field scanning. Otherwise, the signal intensity would decrease drastically and disappear almost completely in an extreme case.

Phenomenologically, the observed behaviors of the superconducting thin film appear to suggest that, at a given external magnetic field, the film has a memory of two distinct states: one state pertaining to the positive polarity of the signal, and the other to the negative polarity of the signal. A transition between the two memory states can be triggered by reversing the sign of the dH/dt.

The present author is inclined to believe that the observed memory behavior is, at least in part, due to the magnetic hysteresis of the superconducting thin film in the mixed state.[11] It appears, however, that the hysteresis alone does not easily explain the observed phenomena. Efforts in this laboratory to account for the phenomena by using the time-independent Landau-Ginsburg equation,[12] which is valid near H_{c2}, have turned out to be rather disappointingly unsatisfactory. The present author feels that a next approach is to make use of the time-dependent Landau-Ginsburg equation.[13] J. G. Park[14] recently investigated magnetic transitions of tin alloys subjected to time-dependent RF magnetic fields, and encountered similar difficulties in explaining the experimental results in terms of conventional hysteresis mechanism.

(ii) Electron-tunneling

If radiation defects and other controlled defects are to affect the transition of superconducting materials, they are expected to affect the superconducting (energy) gap parameter (Δ) of those materials. This is so, because the transition temperature (T_c) is related to the gap parameter $\Delta(T)$ at a temperature p as follows:

$$\Delta(t) \cong 3.2\ kT_c \sqrt{1 - \frac{T}{T_o}}\ , \quad \text{where } T < T_c\ .$$

where k is the Boltzmann constant.[15] The gap parameter Δ can be very reliably determined by means of the electron-tunneling of a junction consisting of superconductor-insulator-superconductor (or normal metal).[16]

Samples of Pb-oxide-Al junction have been successfully prepared in this laboratory, and are being employed for the purpose of radiation damage studies. Pb and Al have the transition temperature $T_c = 7.23°K$ and $T_c = 1.18°K$ respectively.[17] Thus, as far as the junction is kept between 1.18° and 7.23°K, Pb is superconducting while Al is normal. The measured Δ therefore pertains to Pb. Careful calibration measurements of the tunneling properties without radiation treatments have been conducted under controlled magnet fields at low temperatures.

X-ray beams (40 kV - 20 ma) are currently being employed for the purpose of radiation treatments of these junctions which are cooled in a He dewar. The x-ray beams produce defects not only in the superconducting Pb side of the junction but also in the normal metal Al side, plus all the electrical lead wires and electrical contacts attached to the junction for the measuring circuits. It is known[18] that radiation defects produced in these metals near 4.2°K do not anneal noticeably and affect significantly electrical properties of these metals. Thus, before any meaningful measurements can be made on x-irradiated Pb, the radiation effects on the other metallic parts of the junction have to be carefully determined. Efforts are being made toward this background calibration. It should be noted here that irradiation of these materials at temperatures higher than 77°K is practically of no use, since most of radiation defects anneal out, leaving little effect on the materials electrical conductivity.[18] Recent measurements in this laboratory have shown that the electrical resistivity of Pb can be increased by 15% after a 24-hour x-irradiation near 77°K. Thus it is expected that similar x-irradiation near 4.2°K will affect the resistivity of Pb more noticeably. Besides, the insulating layer of the junction does not seem to be affected significantly by the x-ray treatment. These preliminary results are very encouraging for further work.

(iii) DC Measurements

Impirically, various defects in type II superconducting materials have been known to act as <u>pinning</u> centers for the quantized magnetic fluxoids penetrating through the samples in their mixed state.[16] The mechanism and/or nature of the pinning by the defects appear to be little understood but of much interest both in the theory of type II superconductor and for practical applications in superconducting magnets. Therefore, it is significant to investigate possible effects of radiation defects on the fluxoid motion.

Experimentally, one may compare the D.C. current(I) vs voltage (V) relation of the samples with or without radiation treatments. If their comparative studies are conducted under controlled magnetic field but at a fixed $T < T_c$, the comparative data on critical currents can be obtained, among other things.[19-20] The critical current data may be used for understanding the pinning effect. If the experiments are conducted at controlled temperatures near T_c the observed I-V curves would yield information on the radiation effect on T_c. This data then may be correlated with the corresponding data obtained in (ii).

Preparations for conducting these experiments are currently in progress. Initial studies will be dealing with Nb ($T_c = 9.1°K$), Pb ($T_c = 7.23°K$), and Sn ($T_c = 3.72°K$), with controlled x-ray treatments. As mentioned in (ii), these studies require careful calibrations of the electrical properties of lead wires and electrical contacts under the x-ray treatments, prior to any meaningful experiments.

III. Equipment and Facilities

Several pieces of equipment have been commercially purchased under the contract, in order to upgrade the operational efficiency of the existing capital equipment in this laboratory. They are one infra-red grating, one trace recording camera unit, one metal film deposit control unit, one variable-temperature cryostat tail section, one high vacuum gate valve and one liquid nitrogen baffle for a high vacuum mercury diffusion pump. Also purchased under the contract are one photomultiplier tube for optical work, one mercury xenon lamp, and one ultrasonic cleaner.

In particular, the completion of assembling the variable-temperature helium cryostat has been of great help in facilitating the study programs dealing with superconducting materials.

In order to produce a sizeable concentration of radiation defects in these materials, the x-irradiation has to be conducted near 77°K or lower, preferably near 4.2°K, as mentioned in the section II(iii).[18] This requires that the sample be kept in the He dewar while the X-irradiation is in progress. Consequently, the heavy metallic He dewar plus the sample has to be brought very close to the x-ray beam port. A large mechanical manifold has been successfully constructed in this laboratory in order to facilitate satisfactory x-irradiation as required. Since the x-ray source is permanently installed in another part of the Physics Research Building here, the manifold is made mobile. The present setup for the radiation treatment is reasonably suitable but it has some practical drawbacks. Moving bodily the cryostat containing liquid nitrogen and liquid helium for cooling the sample between the laboratory and the x-ray room leads to undesirable excess loss of the He and N coolants and also possibly to accidental damage to the expensive cryostat.

Three other important pieces of equipment have been successfully constructed in this laboratory and have been in full operation. One of them is a very delicate fiber suspension torsion balance for the purpose of measuring the Dember effect [Ref: Sec.II(1)] plus the electronic part for generating the crossed sinusoidal electric fields. Irradiated samples of alkali halide are suspended at the bottom of this vertically held torsion balance which is then placed at the center of the crossed sinusoidal electric fields. Secondly, an electronic device plus a probe for measuring the He level in the cryostat has been constructed employing a single transistor and a carbon resistor. Pending the calibration, the device is in partial operation. Thirdly, an electronic unit to measure degree of vacuum down to 10^{-6} torrs is being assembled in this laboratory. The unit consists of a thermo-couple circuit and an ion-gauge circuit and a temperature-controlled filament circuit.

On the basis of matching funds, the Department of Physics, Wayne State University, Detroit, Michigan, has contributed a capital fund of $12,000 to the present contract work as an additional support to the original agreement in the 1968-69 budget (modification No. 8) for the contract work. The U. S. Atomic Energy Commission allowed the present author to apply an amount of $4,000 from the contract budget to the equipment section of the budget, for the purpose of matching funds. (Reference: my letter of November 11, 1968 to Dr. Louis C. Ianniello, Division of Research, AEC, Washington, D.C. and the November 26, 1968 letter of Dr. Harold N. Miller, Director, Contracts Division, AEC Chicago Operations Office). Subsequently, the Department of Physics increased voluntarily its share of contribution by another $2,000. Thus the total of the matching funds from the Department of Physics amounts to $14,000. These funds facilitated the purchase of the following items:

(i) One Varian Associates Model V-3401 9-inch laboratory electromagnet with a pair of cylindrical ring-shim pole caps (V-3484), a rotating base (V-3421-2), and a mobile base (V-3472).

(ii) One Varian Associates Model V-FR2803 MK-I Fieldial magnet power supply with a zero sweep unit (V-FR-2512).

(iii) One Hewlett-Packard model 7004A X-Y recorder and its accessories.

(iv) One RFL Industries, Inc., Model 220 Gaussmeter and its accessories.

All the items have been recently delivered. The magnet and its power supply are being installed. The other items are in operation fully.

The addition of these new pieces of equipment during the current contract year are expected to facilitate more efficient conduct of the study programs described in Section II, which have been overloading the present 12-inch magnet system and other supporting equipment.

IV. Personnel

The present contract work has been supervised by the principal investigator, Dr. Yeong Wook Kim in the Department of Physics, Wayne State University, Detroit, Michigan 48202. Dr. Clifford L. Aseltine successfully completed his one-year appointment as a postdoctoral research associate supported full-time under the contract. His appointment under the contract was formally terminated as of September 15, 1969. Subsequently he formally joined the U.S. Army Ballistic Research Laboratory, Aberdeen Proving Ground, Maryland. His major contribution to the contract work consists of the modification of the microwave spectrometer system for the purpose of initiating microwave studies of superconducting thin films, and actually conducting initial phase of the microwave study of these films by means of the modified equipment as described in the section II (3). The vacated postdoctoral position was assigned formally to Dr. Kevin Cornell from the University of Illinois, Urbana, Illinois, as approved in the 1968-69 contract budget (modification #8), as of October 21, 1968.

Several graduate assistants have been participating in the current contract work. Mr. Chaleo Manilerd is normally supported at half-time rate by the contract during the academic year of 1968-69 (September 16, 1968 through June 14, 1969). Mr. Manilerd is expected to complete his Ph.D. thesis sometime late in the fall of 1969. Mr. Glen P. Double, a recipient of the National Science Foundation Traineeship effective for the academic year 1967-68, successfully completed his Master of Science thesis (COO-1054-44) in the fall of 1968. In September, 1968, Mr. Double was supported as a graduate research assistant by the contract. As of October 21, 1968, he left the contract work and took a position with IBM's East Fishkill Facility, Hopewell Junction, New York.

Two other graduate students successfully passed the Ph.D. qualifying examination in the fall of 1968 and have been working on their thesis projects full time since then. Their participation in the contract work has not been compensated for since one of them is a recipient of the National Science Foundation traineeship effective for the academic year 1968-69, and the other is a recipient of a National Defense Education Act fellowship effective for the academic year 1968-69. These two graduate students are expected to complete their Ph.D. theses approximately two to three years hence. Three other starting graduate students began to participate in the contract work on a limited basis. Their participation in the contract work, however, is not compensated for, since they are supported by either National Defense Education Act fellowships or departmental graduate teaching assistantships for the 1968-69 academic year.

During the summer of 1968 (June 19, 1968 to September 15, 1968), Dr. Clifford L. Aseltine and Mr. Chaleo Manilerd were supported on a full-time basis by the contract. As mentioned in the preceding paragraph, Mr. Glen Double, a graduate assistant, was paid on an hourly basis out of the contract nct more than four hours per week. Mr. Robert Skrocki, a graduating senior physics student, was supported on a full-time basis under the contract. Mr. Skrocki had been participating in the contract work since his junior year in this department, initially under a National Science Foundation undergraduate research participation program and voluntarily in his senior year. Mr. Skrocki graduated as an honor student in June, 1968. The present author wished very much that Mr. Skrocki would join the present research program, particularly because of his long association with the laboratory and his high scholastic record as an honor student. However, at the end of the summer, the draft status of Mr. Skrocki changed his mind and he gave up his graduate physics training.

V. Publication of Results and Personnel Activities

Several papers have been either published or are under preparation for possible publication. These will be listed in the following order according to their COO serial numbers.

1. Reprint (COO-1054-33).

   K. L. Vanderlugt and Y. W. Kim, "Conversion of F_3^+ Centers and Destruction of R Centers in LiF with R-light", Phys. Rev. 171, 1096 (1968).

2. Reprint (COO-1054-34)

   Ronald A. Andrews and Y. W. Kim, "Delayed Luminescence of M Center in F to M Conversion in NaF", Phys. Rev. 170, 793 (1968).

3. Reprint (COO-1054-35)

   R. A. Andrews and Y. W. Kim, "Impurity Effects on F to M Conversion in NaF", J. Phys. Chem. of Solids 29, 1909 (1968).

4. Abstract (COO-1054-43)

   C. Manilerd and Y. W. Kim, "Delayed Luminescence in Alkali Halide under F Bleaching", Bull. Am. Phys. Soc. Ser. II, Vol. 14, 516 (1969), plus the text of talk.

5. M. S. Thesis (COO-1054-44)

   Glen P. Double, "Luminescence in Calcium Tungstate" (September, 1968)

Professor Y. W. Kim attended the American Physical Society meeting in New York City, January 28, 1969 to February 1, 1969. Dr. Kevin Cornell, the postdoctoral research associate, attended the American Physical Society meeting in Philadelphia, Penn. March 24-27, 1969. Mr. Chaleo Manilerd presented the abstract (COO-1054-43) at the American Physical Society meeting in Washington, D.C. April 28-May 1, 1969.

Several members of the group have visited the Phoenix Nuclear Laboratory, University of Michigan, Ann Arbor and Michigan State University, East Lansing, Michigan for the purpose of irradiating various samples in beams of gamma-rays and/or electrons.

The principal investigator respectfully acknowledges the support of the present contract work by the U.S. Atomic Energy Commission.

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