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Progress Report No. XIII
Laboratory for Insulation Research
Massachusetts Institute of Technology

O.N.R. Contracts N5ori-07801
N5ori-07858

June, 1953

Progress Report No. XIII

Laboratory for Insulation Research
Massachusetts Institute of Technology
Cambridge, Massachusetts

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June, 1953

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ORGANIZATION CHART
LABORATORY FOR INSULATION RESEARCH

June 1, 1953

Professor A. R. von Hippel, Director
Dr. Alexander Smakula, Associate Director
S. M. Kingsbury, Business Manager

DIELECTRIC MEASUREMENTS

William B. Westphal, Group Leader
Anne C. Miller
Benjamin R. Moon

SPECTROSCOPY

Jay T. Last, Grad. Student, Physics

X-RAY DIFFRACTION

Dr. Sidney C. Abrahams, Group Leader
Dr. H. Judith Grenville-Wells
William R. Blackmore, M.Sc., Grad. Student, Chemistry
Jean H. Epstein, Grad. Student, Physics

HIGH VOLTAGE

Dean A. Powers, E.E., Group Leader
Max E. Caspari, Grad. Student, Physics
Myer Geller, M.S., Grad. Student, Physics
William B. Green, Grad. Student, E.E.

FERROMAGNETICS

David J. Epstein, M.S., Grad. Student, E.E., Group Leader
Bertram A. Calhoun, M.A., Grad. Student, Physics
Donald O. Smith, M.S., Grad. Student, Physics
Boguslaw Frackiewicz
Lee E. Johnson, M.S., Grad. Student, E.E.

DRAFTSMAN

John J. Maccarrone

MACHINISTS (Five)

FERROELECTRICS

Elizabeth D. Alden, Grad. Student, Physics

CRYSTAL GROWTH

Dr. Alexander Smakula
Edgar Rudzitis
Victors Sils
Juris Smiltens
Donald G. Wickham, M.S., Grad. Student, Chem.

SEMICONDUCTORS

Professor F. Ralph Kotter

THEORETICAL PHYSICS

Dr. Eugene P. Gross

HIGH PRESSURE

Dr. Peter W. Forsbergh, Jr.
Dr. C. S. E. Phillips

CERAMICS

George Economos, M.S. Grad. Student, Metallurgy
John H. Baldrige

INORGANIC CHEMISTRY

Janis Kalnajs, Dipl. Eng. Chem.

ORGANIC CHEMISTRY

Dr. Laurence G. Wesson

DESIGN ENGINEER

Lloyd B. Smith

SECRETARY

Monica R. Woodman

TYPIST

Aina Sils

List of Publications, November 1, 1952 to June 1, 1953Reports

- Progress Report No. XII, November 1, 1952
- Technical Report 55 "Behavior of Slow Electrons in Polar Crystals,"
by E. P. Gross.
- Technical Report 56 "The Growing of Lead Titanate Crystals and Some of
Their Properties," by H. H. Rogers.
- Technical Report 57 "Tables of Dielectric Materials, Vol. IV," by the
Dielectrics Measurements Group, W. B. Westphal,
Group Leader, Lab. Ins. Res.
- Technical Report 58 "An X-Ray Diffraction Study of Barium Titanate,"
by H. T. Evans, Jr.
- Technical Report 59 "Photocurrent, Space-Charge Build-Up and Field
Emission in Alkali Halide Crystals," by A. von
Hippel, E. P. Gross, J. G. Jelatis and M. Geller.
- Technical Report 60 "On the Disorder in Crystalline 2, 2-Dinitropropane,"
by S. C. Abrahams.
- Technical Report 61 "The Low-Temperature Transition in Magnetite,"
by S. C. Abrahams and B. A. Calhoun.
- Technical Report 62 "A Low-Temperature Adaptor for the Norelco High-
Angle Spectrometer," by B. A. Calhoun and S. C.
Abrahams.
- Technical Report 63 "A Shielding Device for X-Ray Diffraction Cameras,"
by S. C. Abrahams and W. R. Blackmore.
- Technical Report 64 "A Moving-Strip Fourier Analyzer," by H. J. Gren-
ville-Wells.
- Technical Report 65 "A Graphical Method of Evaluating Certain Crystal-
lographic Structure Factors," by H. J. Grenville-
Wells.
- Technical Report 66 "Upper Level Precession Photography and the Lorentz-
Polarization Correction. Pt. II," by H. J. Grenville-
Wells and S. C. Abrahams.
- Technical Report 67 "Inhomogeneity of Thallium Halide Mixed Crystals and
Its Elimination," by A. Smakula, J. Kalnajs and
V. Sils.

Published Papers

- "The Optical Absorption of Sulfur," A. M. Bass, J. Chem. Phys. 21, 80 (1953).
- "Investigation of Crystal Growth by Thermal Etching and Oriented Overgrowth," A. Smakula and M. W. Klein, J. Chem. Phys. 21, 100 (1953).
- "The Low-Temperature Transition in Magnetite," S. C. Abrahams and B. A. Calhoun, Acta Cryst. 6, 105 (1953).
- "The Crystal Structure of Cesium Hexasulfide," S. C. Abrahams and E. Grison, Acta Cryst. 6, 206 (1953).

Papers Presented at Technical Meetings

- "Conduction and Breakdown," A. von Hippel, Conference on Electrical Insulation, Nat. Academy of Sciences and Nat. Research Council, Lenox, Mass., Oct. 2-4, 1952.
- "Electrical Conductivity of Magnetite at Low Temperatures," B. A. Calhoun, American Physical Society, Cambridge, Mass., Jan. 22-24, 1953.
- "Photoemission from Silver into AgCl, KBr, NaCl, and New Bands of Photosensitivity in AgCl," M. A. Gilleo, American Physical Society, Cambridge, Mass., Jan. 22-24, 1953.
- "Photocurrent, Space Charge Build-Up and Field Emission in Alkali Halide Crystals," A. von Hippel, E. P. Gross, J. G. Jelatis and M. Geller, American Physical Society, Cambridge, Mass., Jan. 22-24, 1953.
- "Electron Motion in Polar Crystals," E. P. Gross, American Physical Society, Durham, N. C., Mar. 26-28, 1953.
- "Ferroelectrics for Dielectric Amplifiers," A. von Hippel, Symposium on Dielectric Amplifiers, Carnegie Institute of Technology, Pittsburgh, Pa., Apr. 16-17, 1953.
- "Materials for Dielectric Amplifiers," W. B. Westphal, Symposium on Dielectric Amplifiers, Carnegie Institute of Technology, Pittsburgh, Pa., Apr. 16-17, 1953.

Progress Report No. XIII

on

Projects N5ori-07801 and N5ori-07858

SURVEY

In glancing over the Table of Contents and the Organization Chart of this report, the reader may feel like the proverbial Hussar who is compelled to gallop simultaneously in all directions. But while we are spread thin indeed over the wide field of dielectrics, and the chorus of good friends is not missing that urges us to concentrate and strike a mighty blow, our present position is dictated by long-range objectives.

Our laboratory was started about fifteen years ago to help develop a new field, "dielectrics", and, in doing so, to train graduate students of physics, chemistry, and electrical engineering in a broad common research effort, free of departmental boundaries. This charter implies dissipation of effort over a wide field in order to gain broad perspectives. It also requires that after the research is progressing with some success, the knowledge gained is set to work in a revitalized teaching program.

We are just entering this second phase of our effort. Two books, "Dielectrics and Waves" and "Dielectric Materials and Applications", will go to print next month. The former tends to convey how we might think today in a unified approach about polarization, magnetization, and conduction from the macroscopic and from the molecular standpoint. The latter book represents the outcome of last summer's training course and tries to tell in a sequence of contributions by numerous authors about the properties of dielectric materials, their measurements and a variety of applications.

Simultaneously, the Electrical Engineering Department under the

vigorous leadership of Professor Gordon S. Brown has entered the scene with a new philosophy concerning the training of our undergraduate students. It is our common consent that the classical education of the electrical engineer in fields and circuits has to be complemented by an understanding of the properties of materials and their interaction with fields and circuits. Our contribution to this program will be a new course, "Molecular Electrical Engineering", offered to the Juniors next spring.

The teaching of engineers requires that we think about the practical implications of our fundamental research effort. A similar obligation arises from our Government relations, where we provide useful practical information in our "Tables of Dielectric Materials" and advice when asked about dielectric problems. We believe that this aspect of applications, when kept within bounds, is not disturbing for a fundamental research program but vitalizing, since it provides a merciless test as to how well we have understood a given set of phenomena.

In glancing over the group of reports in the following pages the reader will find that our fundamental research is not guided by thoughts of applications. But here and there phenomena emerge which obviously should be followed up until their applicability is clarified. Our ferrite research is a case in kind, and the long-range applied research on ferroelectric ceramics should be pushed in a similar way to ascertain what kind of materials can actually be made for dielectric amplifiers, memory devices, etc. Our research on field emission in solids will soon approach a similar stage.

If we could set up our laboratory today on a final basis, guided by the experience of the past years, we would consolidate it in an Institute of Dielectrics. Fundamental research would form the strong base, long-range

applied research establish the feasibility of new approaches, and development work pioneer the engineering of new devices. In addition, an information center would keep fully abreast of the field of dielectrics and be available to Government and Industry for impartial advice, backed up by the first hand knowledge and unique facilities of the various groups of the laboratory.

A. von Hippel

DIELECTRIC SPECTROSCOPY

Dielectric measurements (W. B. Westphal and A. C. Miller)

Volume IV of the "Tables of Dielectric Materials" has been issued as Technical Report 57. Over six hundred dielectrics are included, of which about 250 are new materials. Also the following dielectrics have been measured since Volume IV appeared:

Dilecto GM-1	Glass mat, melanine	Continental Diamond Fibre
Dilecto GM-PE	Glass mat, polyester	"
Hysol XL-6060	Soft Epoxy resin	Houghton Labs.
Hysol XE-6080	" " "	"
Hysol 6030 B	" " "	"
Rexolite 1422 *	Cross-linked polystyrene	Rex Corp.
SE-450 *	Silicone rubbers	General Electric
SE-460 *	" "	" "
SE-972 *	" "	" "
Alsimag T-111-4	Titania body	American Lava
" T 128	Titania "	" "
" 475	Zircon porcelain	" "
" 491	Alumina porcelain	" "
" 513	Alumina porcelain	" "
" 576	Alumina porcelain	" "
" 577	Wallastonite porcelain	" "
Ferramic 1326	Square loop ferrite	General Ceramics

*Materials included in Vol. IV for which temperature data have now been obtained.

Ferramic 1331	Gyrator ferrite	General Ceramics
Corning 0014 *	Glasses	Corning Glass
" 7055 *	"	" "
" 7570 *	"	" "
" 8830 *	"	" "
" 9010 *	"	" "
Cesium bromide	Crystal	Ins. Lab.
Cesium chloride	"	Ins. Lab.
Barium titanate	Crystals	Ins. Lab.
Fluorochemical 754	Liquid	Minnesota Mining

Special samples were measured for the Cambridge Research Laboratories and for Project Lincoln.

Improvements have been made in the 25,000 Mc equipment and several coaxial lines have been built for measurements on ferrite rings (2 in. dia.) in the frequency range from 30 Mc to 150 Mc.

It is expected that the main effort of the dielectric measurements group for the next half year will be concerned with frequency response characteristics of ferroelectrics and ferromagnetics.

Pressure broadening of microwave resonance lines (C. S. E. Phillips)

The equipment mentioned in the previous Progress Report (p. 3) is largely completed. The pressure system (Fig. 1) has been tested and found to work satisfactorily under a vacuum and at pressures up to 700 atmospheres. A higher-pressure pump working up to 2,700 atmospheres has been obtained and connections provided. The bomb and the oil-mercury

* Materials included in Vol. IV for which temperature data have now been obtained.

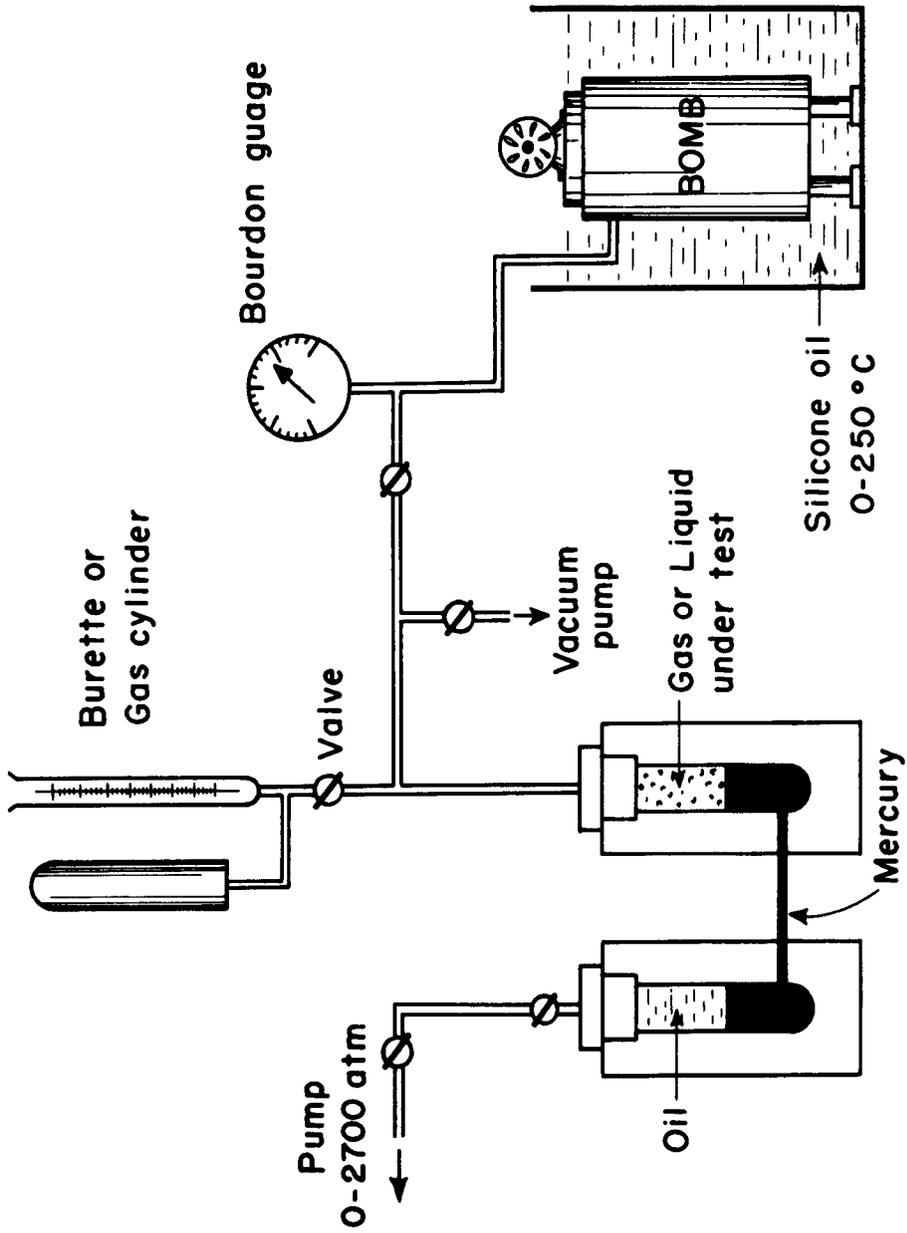


Fig. 1. High pressure system (schematic).

separator are installed in a pit in the concrete floor for reasons of safety. The temperature control system also has been installed, but awaits the heat transfer fluid (silicone oil 550 R). The remote control system works satisfactorily, but up to the present the resonator has been tuned manually.

A set-back was encountered with the microwave system, which has not worked satisfactorily. The long lengths of cylindrical waveguide used to separate the experimenter from the bomb have introduced more loss than anticipated, probably because the bends are not in the same plane. A more serious problem was encountered in the bomb itself. Direct coupling (of the input and output wave) between the input and output windows was observed, and difficulties in adjusting the H_{01} resonator system led to the decision to change to a transmission cavity of the H_{11} type. Bench tests have shown that such a system should work more easily within the bomb. An ordinary commercial cavity is being adapted for this purpose.

Once the microwave problems are solved, initial runs are planned on carbon tetrachloride for the purpose of testing the whole equipment and in order to investigate the possibility of creating semipermanent dipole moments by means of pressure in a normally nonpolar molecule.

Pressure broadening of resonance lines in the infrared and microwave region (E. P. Gross)

Theoretical work is being carried out in connection with the pressure broadening program reported by Dr. C. S. E. Phillips in the previous paragraphs. A survey is in preparation of the present theoretical and experimental information on pressure broadening in the micro-

wave and infrared regions in gases and the dielectric properties of simple liquids. We are attempting to assess the value of pressure-broadening experiments in elucidating the forces between molecules and the dynamic order in liquids. Theoretical calculations have been undertaken of the static dielectric constant in polar gases under pressure. These calculations are generalizations of the analysis presented by Kirkwood¹⁾ and van Vleck²⁾ of the "translational fluctuation effect". Experimental data are scarce at present ; reliable measurements would be very valuable for the study of intermolecular forces.

A theoretical study of a dense dipolar gas (Progress Report XI, p. 12) has been continued, and an attempt is being made to formulate a theory for the widths of microwave rotational lines.

OPTICAL SPECTROSCOPY

(J. T. Last)

Instrumentation

At the date of our last progress report, the Beckman I R-3 spectrophotometer had been received and was undergoing performance tests. A number of difficulties arose in the optical as well as in the electronic part of the equipment. With the close co-operation of the Beckman Instruments Co., the necessary changes and improvements are being made to realize the inherent capabilities of this powerful equipment. An accurate calibration of the spectrophotometer in the range of the CaF₂ optics from 0.6 to 8.2 microns has been about completed. The monochromator housing will be exchanged shortly to allow an easy removal of the KBr lenses; the wire recorder has been exchanged for a tape recorder; an accurate

1) J. G. Kirkwood, J. Chem. Phys. 4, 592 (1936).

2) J. H. van Vleck, J. Chem. Phys. 5, 556 (1937).

calibration over the spectral range covered by KBr optics will be started as soon as possible; and a new micro-adaptor developed by the Beckman Company will be tried out for measurements with small crystal samples.

This period of equipment consolidation was used to return our Cary spectrophotometer, after four years of excellent service, to the manufacturer for a thorough overhaul since a number of minor troubles had developed in the course of operation. The spectrophotometer has just been returned to us. A new powerful hydrogen lamp has been installed for ultraviolet measurements, and the instrument is back in operation.

X-RAY CRYSTALLOGRAPHY

In the continuation of our study of the crystal chemistry and molecular configuration of oxygen, sulfur, selenium, and tellurium compounds in their various valence states, we have now nearly completed the work on a group of important substances.

A variety of means for explaining the relations between magnetic behavior and crystal structure and texture in the ferrites have been examined; and our search for new techniques has been maintained.

Barium tetrasulfide monohydrate (S. C. Abrahams)

The unit cell size and space group, as well as the approximate positions of the barium atoms, were given in Progress Report No. XII. The atomic co-ordinates of the sulfur and oxygen atom have now been determined largely by desymmetrizing the Fourier series based only upon the superlattice planes. A portion of the symmetrized $hk0$ projection, computed on XRAC through the kindness of Prof. R. Pepinsky, is given in Fig. 2b. All co-ordinates are still undergoing refinement. The double Fourier

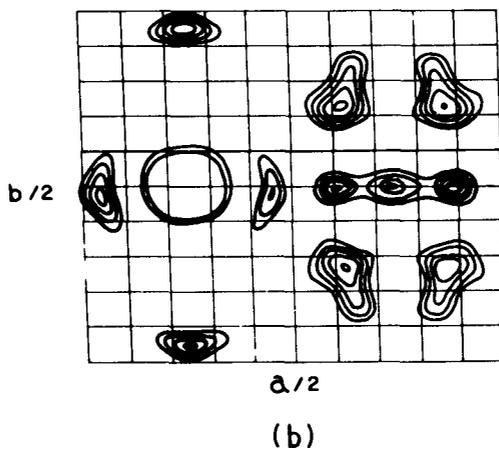
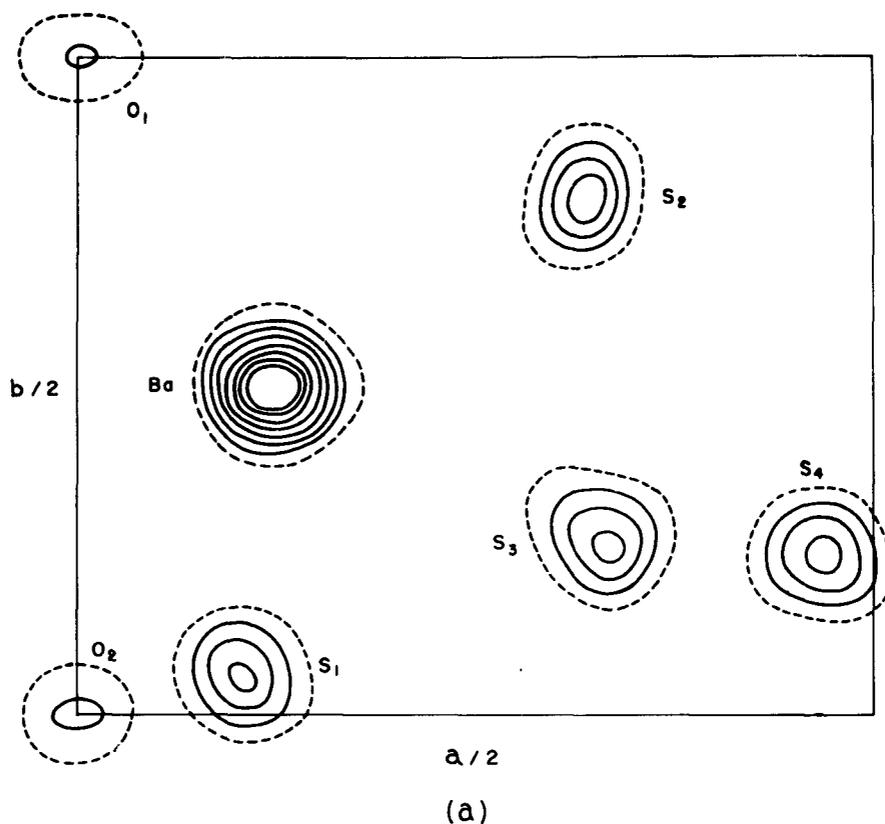


Fig. 2a. Electron density projection of one quarter of the unit cell of $\text{BaS}_4 \cdot \text{H}_2\text{O}$ on (001). Contours are at intervals of $10 \text{ e}\text{\AA}^{-2}$ for the oxygen and sulfur atoms, and $20 \text{ e}\text{\AA}^{-2}$ for the barium atom.

2b. Symmetrized electron density projection of the same part of the unit cell, as viewed on XRAC.

series method was used to its limit, further refinement being obtained by the least-squares process. The value of R_1 for $hk0$ and $0k\ell$ is at present 11.0 and 10.0 percent, respectively, a value considerably better than is usually found in crystals such as this. A very encouraging feature of this analysis is the definition of the oxygen atoms, considering the presence of barium. The $h0\ell$ zone gave rather poorer agreement, presumably because these intensities were measured using Cu K radiation. Much improvement is expected on substituting Mo K radiation (the linear absorption coefficients for these radiations are respectively 670 and 82 per cm). It is of interest to note that the tetrasulfide group possesses an exact crystallographic two-fold axis. An electron density map of this crystal is shown in Fig. 2a.

Di-p-tolyl ether, sulfide, selenide and telluride (W. R. Blackmore and S. C. Abrahams)

It has recently been found that crystals of each of these four compounds form a series whose unit cells are within one percent in size, and with a common space group. It is thus reasonable to infer that these crystals are iso-structural. The positions of the tellurium atoms were soon established, and rough co-ordinates assigned to the two tolyl groups. These co-ordinates were then used with the selenide data and further refinement of the tolyl groups obtained using double Fourier series. R_1 for the $h0\ell$ zone is about 30 to 35 percent for the two crystals. The ether was prepared by E. Rudzitis and photographs are now being recorded. The sulfide is in the course of preparation. It is planned to determine the whole series accurately and to observe the change in the divalent VI_b angle and bond length as the series is ascended.

Tellurium tetrabromide and iodide (W. R. Blackmore and S. C. Abrahams)

No further work has been performed on the tetrabromide since the last Progress Report.

In order to obtain good intensities for the tetragonal form of TeI_4 , an extensive search was instituted for new tetragonal crystals. This has been without success thus far, but it has been shown that the form previously described as triclinic (Progress Report IX), is in fact orthorhombic, with $a = 13.54$, $b = 16.73$, and $c = 14.48 \text{ \AA}$, space group Pnma . The calculated density is 5.145, the observed is 5.056, corresponding to 16 molecules per unit cell. One batch, prepared from the elements, yielded a form which is apparently rhombohedral, but since a chemical analysis of this form has not yet been made, it is not known whether they are pure TeI_4 . Further work on both the iodide and bromide will be resumed soon.

Diphenyl sulfoxide and selenoxide (H. J. Grenville-Wells and S. C. Abrahams)

On the basis of magnetic measurements³⁾ and Patterson projections computed from our intensity data, a trial structure has been set up for diphenyl sulfoxide. This is now in the process of refinement, using the calculating devices described in Technical Reports 64 and 65. The value of R_1 is about 50 to 60 percent. Difficulties were encountered in obtaining good crystals of the selenoxide, but these have now been overcome, and the structure of this isomorphous compound will be determined concurrently with the sulfoxide.

Experimental determination of atomic scattering factors for S, Se and Te
(S. C. Abrahams and H. J. Grenville-Wells)

The difficulties previously met in the preparation of the sample (Progress Report No. XII) have been largely disposed of, and some experi-

3) Mrs. E. W. Toor, University College, London. Private communication.

mental data has been obtained for PbS, PbSe and PbTe. The PbS results are the most satisfactory, since the atomic scattering factors are obtained from the experimental sum (Pb + S) and difference (Pb - S) curves, for in the case of PbTe, the difference curve cannot be interpreted with certainty. The curve for PbS is given in Fig. 3.

Further work on several S, Se and Te compounds is planned.

On the disorder in crystalline 2, 2-dinitropropane at room temperature
(S. C. Abrahams)

A recent study of the dielectric constant and loss of 2, 2-dinitropropane⁴⁾ led to interest in the crystal structure of this substance. A consequent study of this phase has been described in Technical Report 60.

A shielding device for X-ray diffraction cameras (S. C. Abrahams and W. R. Blackmore)

The need for adequate protection against stray radiation emitted between the X-ray tube and the diffraction camera has led to the design of a simple shielding device. This is described in Technical Report 63.

A graphical method for evaluating structure factors and electron density
(H. J. Grenville-Wells)

Standard charts for the graphical evaluation of structure factors and related functions have now been prepared; the principles employed and the practical applications of the method are discussed in Technical Report 65.

A single-crystal adaptor for the Norelco high-angle diffractometer
(S. C. Abrahams and H. J. Grenville-Wells)

A preliminary account of this adaptor was given in Progress Report No. XI. The equipment has now been put into use and a typical diffraction

4) J. G. Powles, D. E. Williams and C. P. Smyth, J. Chem. Phys.
(to be published).

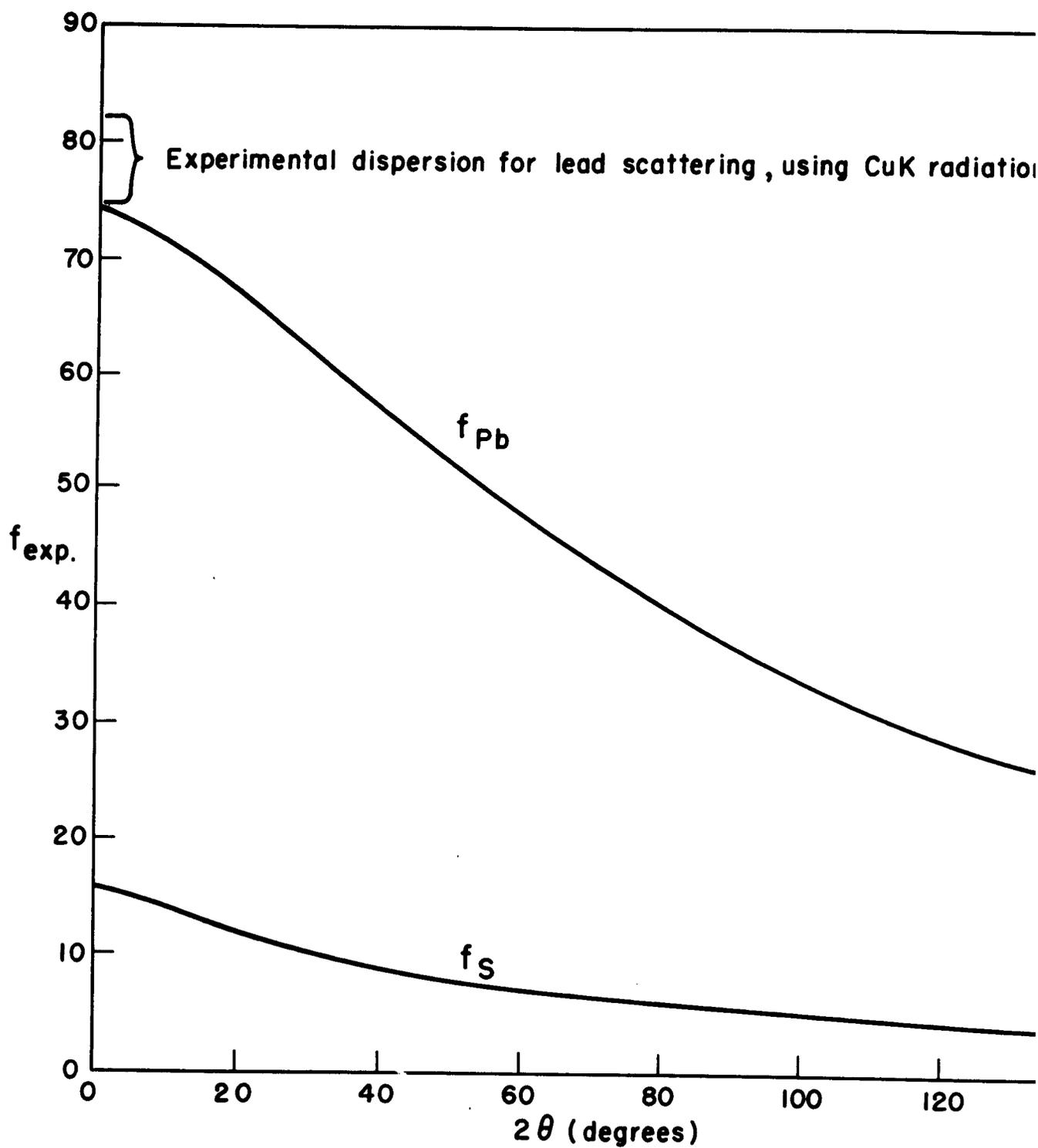


Fig. 3. Experimentally determined atomic scattering factors for Pb and S in PbS.

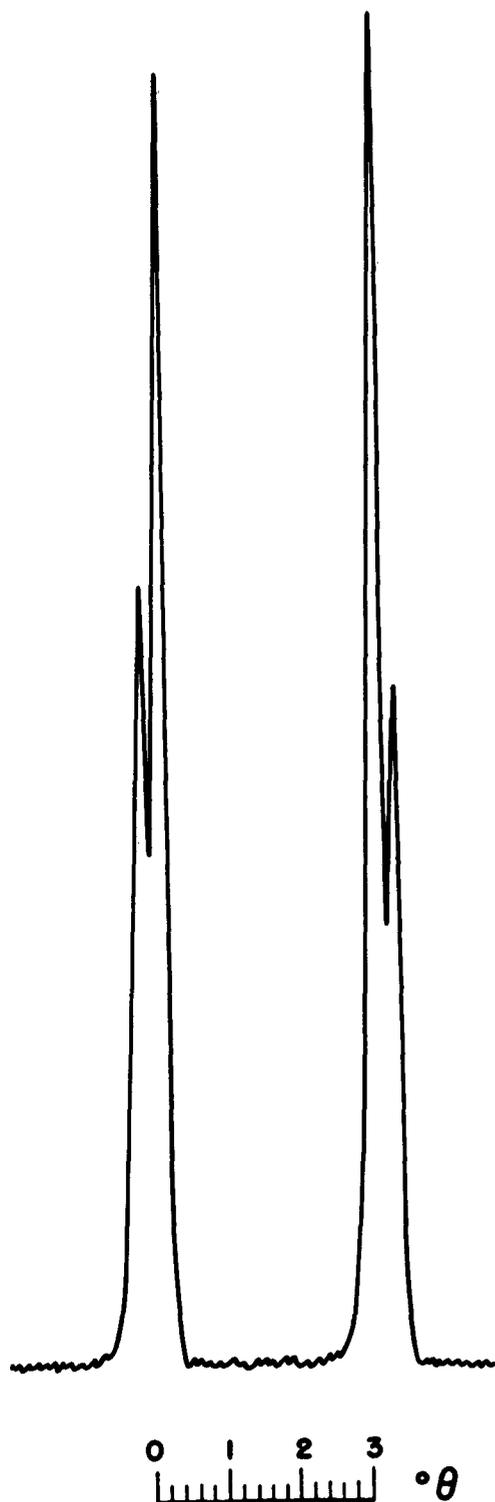


Fig. 4. Oscillation record of (800) line of magnetite using single-crystal adaptor.

record obtained with it is shown in Fig. 4. The integrated intensity is obtained by allowing the diffracted beam to sweep across the stationary entrance slit of the Geiger tube. This slit is wide enough to admit the entire diffracted beam. By using the rate meter, and subtracting the background, the integrated intensity may be obtained rapidly and with high accuracy. This adaptor has the advantage that the Norelco diffractometer may be interchanged for use either as originally designed, or as a single crystal camera within a matter of minutes. It has been found that the entrance slit may be within $\pm 0.2^\circ 2\theta$ of the correct position without loss in accuracy of the intensity measurement. A diagrammatic view and photograph of the adaptor are shown in Fig. 5.

X-ray studies on the composition of the ferrites
(J. H. Epstein)

Attempts have been made to measure the magnetostrictive coefficient in some ferrites by means of X-rays. The change in line position was within the experimental error of estimating the diffraction angle, and new attempts will be made on substances with higher magnetostrictive coefficients. Analysis of the

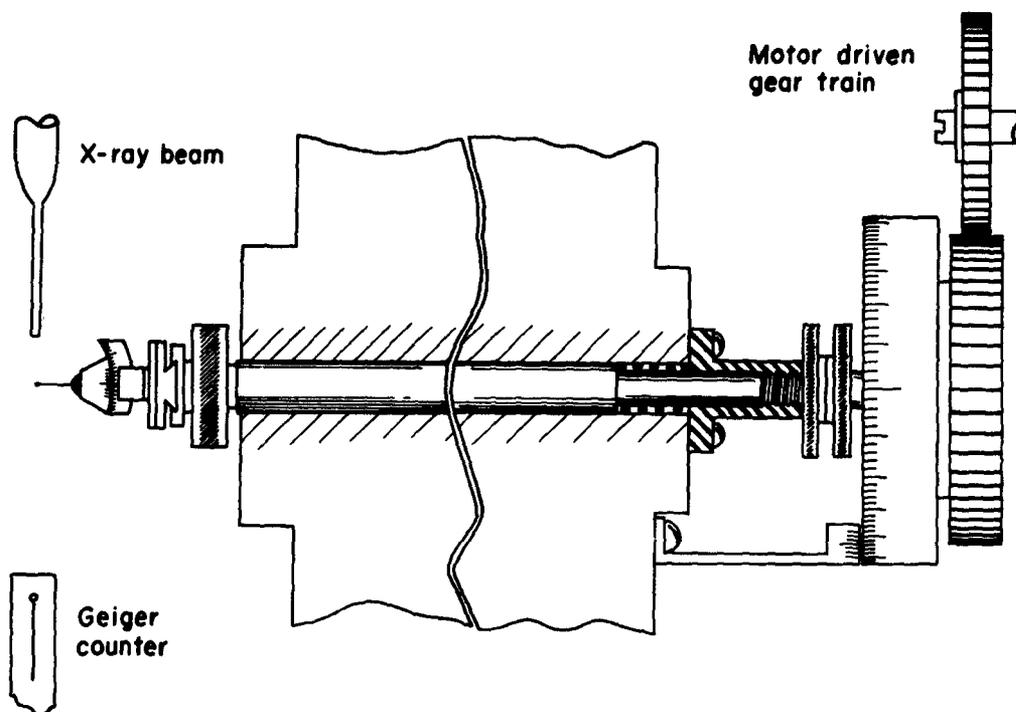


Fig. 5. (upper) Diagrammatic view of the single-crystal adaptor for the Norelco High-Angle Diffractometer.
(lower) Photograph of the driving mechanism for the adaptor.

strain present in some ceramic ferrite samples has not led to conclusive results, and experimental methods of changing the strain while the sample is being examined with X-rays, are being developed. A study of the extent to which the ferrite components have been reacted has been made in close co-operation with G. Economos. We have clearly shown that the degree of inversion in ferrites other than those of Fe, Ni, Co and Mn may be readily estimated with some accuracy. Although particle sizes may be measured by an X-ray method, it seems that for these ceramic specimens, this is not very satisfactory.

INVESTIGATIONS ON BINARY CRYSTAL SYSTEMS

(A. Smakula, J. Kalnajs, and V. Sils)

Inhomogeneity of thallium halide crystals and its elimination

Prisms, cut from mixed crystals of thallium halides, as they are used at present for infrared spectroscopy, show optical inhomogeneity which causes an undesirable decrease of resolution. By chemical, X-ray diffraction, and thermal analysis it was possible to trace the cause of this defect to the use of incorrect data of the minimum melting points of the compositions from which the crystals were grown. The melting points of the thallium halides and of their binary systems have been redetermined, and crystals grown from compositions corresponding to the accurate minimum melting points have been found to be entirely homogeneous. The lattice constants and thermal expansion coefficients of the new mixed crystals have been measured.

Binary systems of thallium iodide and cesium or rubidium halides

The cubic phase of TII can be stabilized at room temperature by

replacing a fraction of the iodine ions by the smaller bromide ions. This brings the mean ratio of cation to anion radius close to the minimum value (0.73) required for the body-centered cubic system. Another method of stabilizing might be to replace the Tl ions by larger cations. For this purpose we selected Rb and Cs which are also monovalent and crystallize as halides in the body-centered cubic structure. Simultaneously, we hoped to extend the transparency for infrared still further than is possible with our KRS5 crystals.

CsI, commercially unavailable, was prepared from CsBr by conversion into CsIO_3 and subsequent thermal decomposition.

The tentative phase diagram (Fig. 6) of TlI + CsI shows a portion in solid solution on the TlI side with a eutectic point at 398.4°C and ca. 25 mole % CsI. It was determined by thermal analysis (Fig. 7).

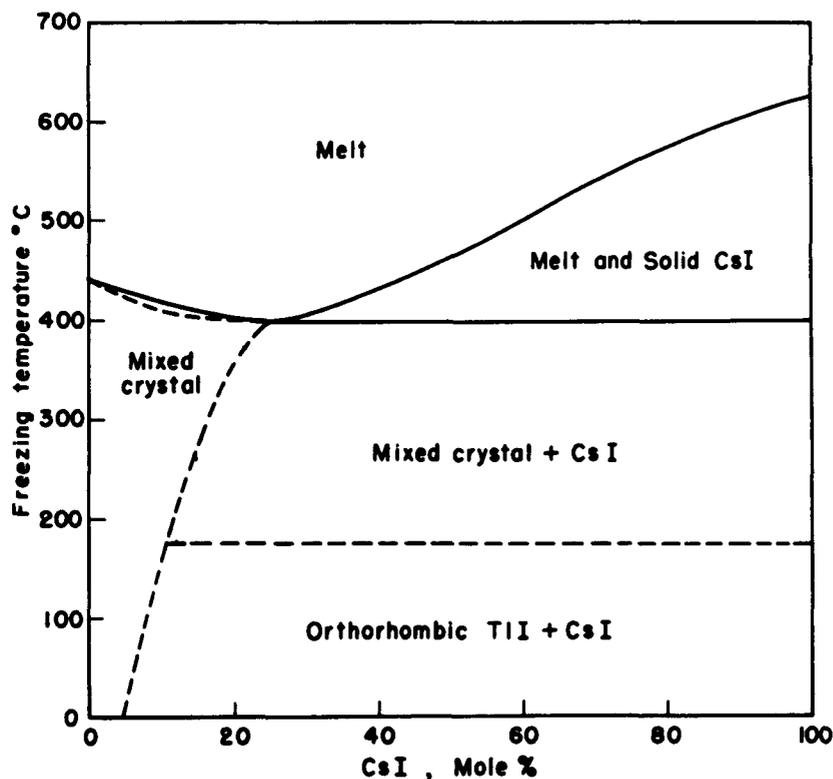


Fig. 6. Phase diagram of TlI + CsI binary system.

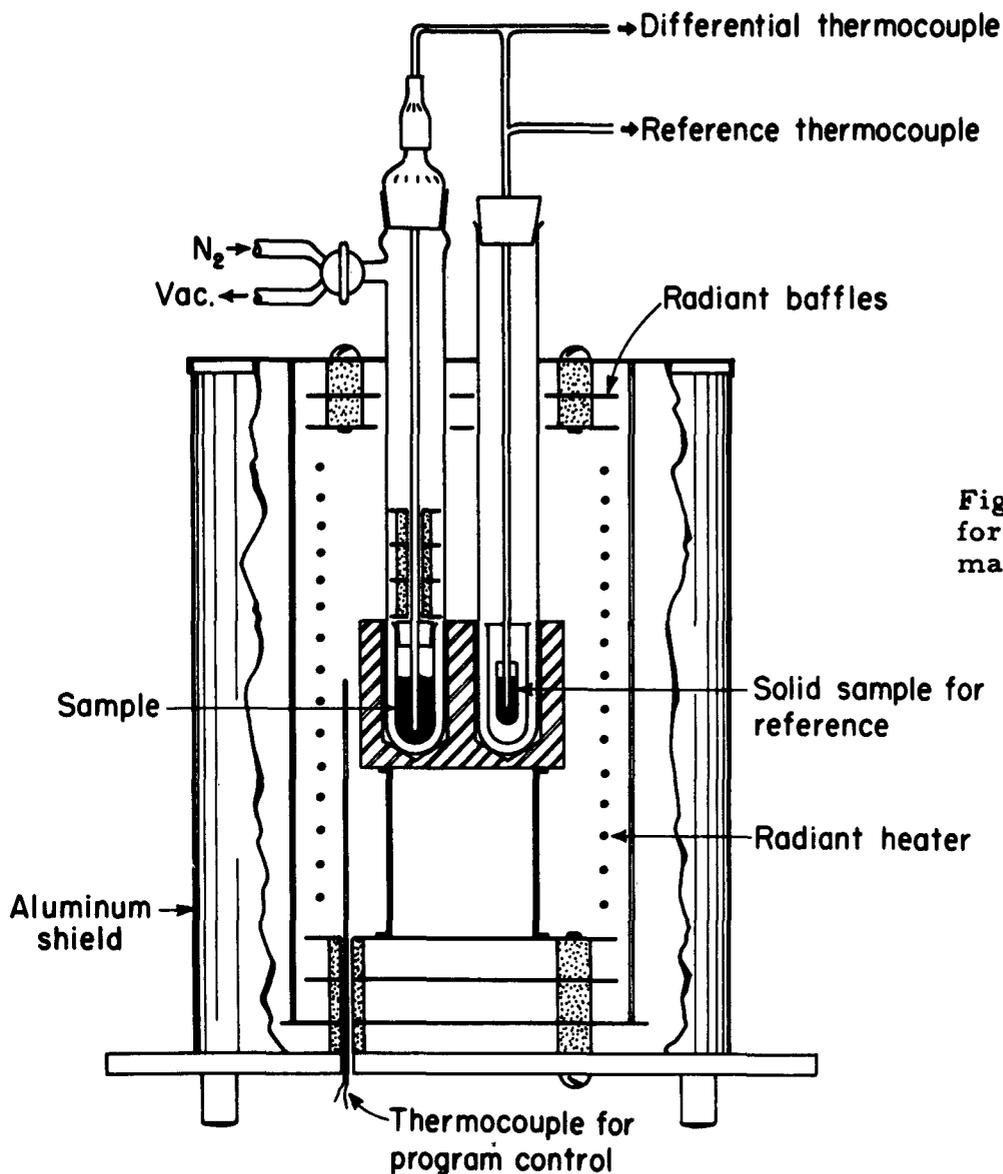


Fig. 7. Apparatus for differential thermal analysis.

The thermal analysis was complemented by an X-ray structure analysis of the solidified components. It was found that from 0.6 up to ca. 25 mole % CsI, only one lattice exists which corresponds to the expanded lattice of cubic TII. Then it remains constant while simultaneously, above 25 mole % (Fig. 8), the lines of pure CsI appear.

Several crystals with compositions of 2 and 5 mole % CsI have been grown. They show a slight inhomogeneity in composition across their length: the bottom part, which crystallized first, contains less CsI than

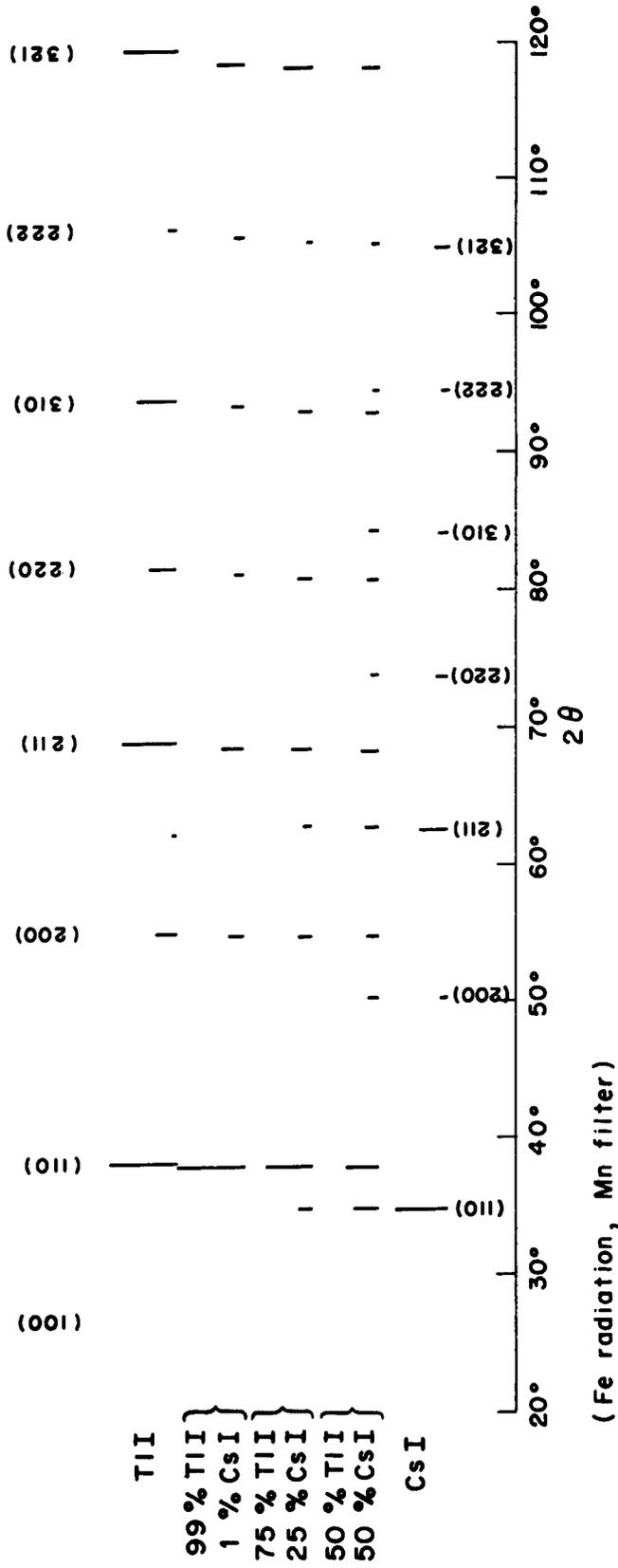


Fig. 8. X-ray diffraction lines of TlI + CsI binary system.

the top. A minimum of ca. 1 mole % of CsI is required for stabilizing the cubic phase of TII. A crystal containing only 0.6 mole % CsI, originally red, started at the bottom to turn yellow after a few weeks due to decomposition (Fig. 9). Filings of the crystal are even less stable, as would be expected, and also a crystal with 1 mole % CsI decomposes immediately on immersion in liquid nitrogen. On the other hand, crystals containing more than 5 % CsI lose their transparency at room temperature because of the separate crystallization of CsI.

The binary system TII-CsBr shows a behavior similar to TII-CsI, although somewhat more complicated since four different ions are involved. A crystal grown with 2 mole % of CsBr shows no decomposition after 3 1/2 months at room temperature. Unfortunately, the inhomogeneity in composition across its length seems to be somewhat higher than for CsI-TII crystals of corresponding composition and growing speed. The variation in lattice constant, however, amounts only to 0.007A over a length of 5 cm.

The cubic modification of TII can also be stabilized by the addition of RbI. A crystal grown from the composition 5 mole % RbI and 95 mole % TII is dark red and shows very slight inhomogeneity. The phase diagram has not yet been worked out.

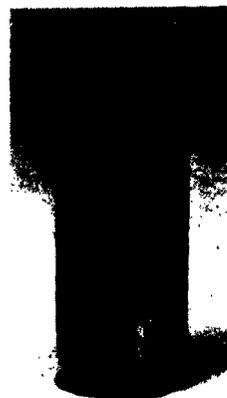


Fig. 9. Crystal of ca. 98.5 mole % TII + 1.5 mole % CsI, showing decomposition.

Although the discharge through the crystal was somewhat more destructive than that produced by point-plane systems because of the larger capacitance of the crystal sample, the destruction was small enough so that the directions of the breakdown paths could be ascertained. The discharge took place near the center of the tin-foil electrode and there were no visible signs of prebreakdown discharges, either at the edges of the foil or around the point where breakdown took place. It is thus safe to assume that the field prior to breakdown was not far from homogeneous.

The breakdown paths in both KBr and NaCl were always in the direction of the applied field, i. e., the $[100]$ direction. This is also the principal direction observed in KBr at room temperature in inhomogeneous fields, but in NaCl $[110]$ and $[111]$ directions are observed under these conditions. It seems therefore that under homogeneous field conditions the paths lie in the direction of the applied field. This means that the preferential scattering of the electrons by the crystal into certain directions can be counteracted by the action of a sufficiently strong field.

Experiments are in progress to determine the direction of the breakdown paths in fields of varying degrees of inhomogeneity.

Electric breakdown in hexane (W. B. Green)

Breakdown studies on liquids suffer from the disadvantage that fibers and impurities can be pulled into the gap, and that surface tension and bubble formation play a not clearly established role. On the other hand, a great simplification compared with the studies on solids results since no surface preparation of samples is required and the normal edge effects may be avoided easily. The presence of fibers has been eliminated and the difficulties caused by ion migration may perhaps be minimized by impulse studies.

It was therefore decided to make an extremely careful prototype study on n-hexane.

This liquid is especially suitable, since the investigation can be extended to other aliphatics for an evaluation of the influence of chain length, to aromatics by using cyclohexane as an intermediate compound, and to substituted hydrocarbons.

In Progress Report XI H. Wada reported on the design of a test cell for liquids which was used for a large number of breakdown tests on hexane with transient rise times and electrode metal as variables. Representative values obtained for steel electrodes were 750 kv/cm for a 10^{-5} - sec. rise time of the voltage wave, and 2700 kv/cm for a 10^{-8} sec. rise time. Considerable variations in value occurred for the different electrode metals used. Subsequent work indicates that the results might have been caused by the impurities present in the hexane.

To eliminate such impurities, a new test cell and purification apparatus were constructed. The cell is so designed that only glass and the gold-plated electrode holders contact with the liquid. The metal cell is coaxial as was the previous one used with high-speed pulses. Directly connected to the cell is equipment for the purification of the hexane. Measurements are in progress.

TRANSITION FROM INSULATORS TO METALS

Photocurrent, space-charge build-up and field emission in alkali halide crystals (A. von Hippel, E. P. Gross, J. G. Jelatis and M. Geller)

Additively colored alkali-halide crystals represent, in a first approximation, a transparent solid with frozen-in electrons which can be mobilized by light absorption. When moving towards the anode, these electrons leave

a positive space charge behind, and an adjustable cathode fall results which can be steepened until electrons are released by field emission. The steady state and transient solutions for the charging and discharging cycle are calculated, the effects of light intensity, wavelength and field emission discussed, and KBr crystals investigated experimentally as function of time, voltage, light intensity and color-center density. Field emission has been produced with voltages as low as 1 volt, and a field-emission photocell has been realized.

This work has been described in Technical Report 59 and will be published in Physical Review.

Field emission into alkali halide crystals in its dependence on temperature, electrode metal and surface preparation (M. Geller)

In continuation of the preceding investigation, a detailed study has been started on the influence of temperature, electrode material, and types of surface contact on the electron transfer from metal to crystal.

Field emission into other types of crystals (D. A. Powers)

The studies on alkali halide crystals reported above are being extended to other types of dielectrics.

Electrical conductivity of Se, Te, and Se-Te alloys (F. R. Kotter)

The equipment for making a four-terminal resistance measurement with a single voltage pulse has been completed and room-temperature measurements of the resistance of several polycrystalline samples of selenium as a function of field strength have been made.

A block diagram of the equipment is shown in Fig. 11. Flat-topped voltage pulses, variable in amplitude from 50 to 750 v, and in duration from 10 to 200 micro seconds, are supplied by the manually triggered pulse gener-

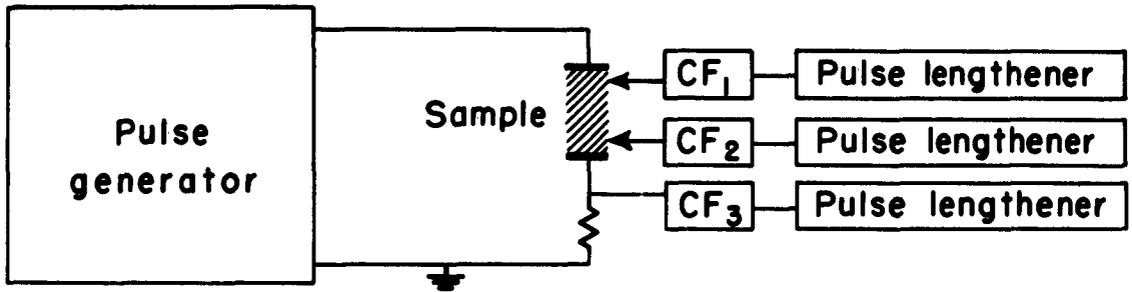


Fig. 11. Block diagram of test unit (CF = cathode followers).

ator. The cathode followers CF₁ and CF₂ are biased beyond the cut-off with precision voltage dividers. Thus the calibrated pulse lengtheners are required to indicate only the height of the pulses above the bias levels. The pulse lengtheners are very similar to that described by Elmore and Sands.⁵⁾

The measurements on several samples of polycrystalline selenium show that conductivity increases with increasing field strength.

The design of a suitable equipment for extending these measurements to much higher voltage gradients has been started.

FERROELECTRICS

Study of domain walls in BaTiO₃ (E. D. Alden)

Equipment has been assembled for studying the thermal, electrical, and optical properties of the domain wall. A considerable amount of time has been spent in determining the experimental techniques best suited for the study of small strain-free crystals. A single 90° wall has been isolated in a two-domain crystal. This can be moved at will by an electric field and observed in detail.

Experiments on the growing of large crystals from ternary melts

5) Elmore and Sands, "Electronics", McGraw-Hill Book Co., New York, 1949, p. 197.

are continued. The largest grown thus far has been a fairly perfect yellow triangular-plate crystal measuring about 0.6 cm on its hypotenuse.

Growing of single crystals of BaTiO₃ from the melt (J. Smiltens)

A new booster furnace has been built for somewhat higher temperature gradients. Very pure barium titanate has been prepared and experiments are in progress to check the existence and extent of a phase transition from hexagonal to cubic, as described by Roy⁶⁾ and co-workers. The single crystals grown to date give no conclusive evidence as to how far this transition interferes with our approach.

High pressure apparatus for ferroelectric and semiconductor studies
(P. W. Forsbergh, Jr.)

The hydraulic press for producing the proportional thrusts indicated in Fig. 12 has been completed. As mentioned in the previous Progress Report, the Bridgman externally-supported vessel for 30,000 atm. has been doubled as shown in Fig. 12. The increase in length will keep the piston and electrical-inlet seals at a sufficient distance from the heat of the furnace.

The inert gas argon will be used as the compression fluid. Since argon probably solidifies near 17,000 atm. at room temperature, it will freeze in the cool ends of the vessel. This, however, should not impede the motion of the piston, since Bridgman found at low temperatures that solid argon is plastic. At the other end of the vessel, freezing of the argon may cold-work the resistance coil pressure gauge. The coil can, however, be put just near enough to the furnace to prevent freezing around it, or it can be provided with a separate heater. A gold-chrome wire is reported

6) R. Roy, Quarterly Progress Rep., Signal Corp. Project Contract DA 36-039 sc- 5594, Penn. State College, Jan. 12, 1953.

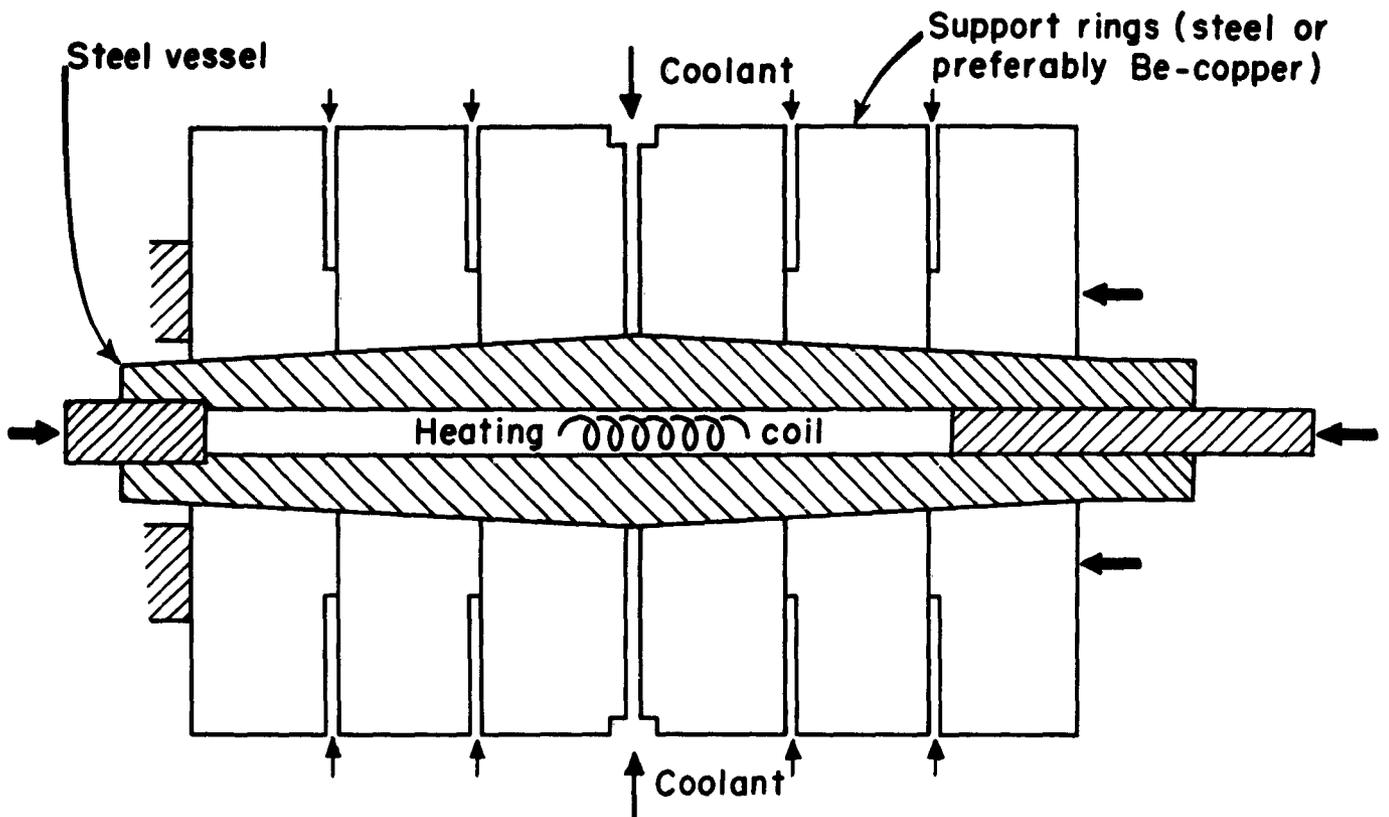


Fig. 12. Present adaptation of the Bridgman technique.

to be better than manganin under temperature changes. With a suitable calibration procedure, a thermocouple near the pressure gauge coil can be removed to avoid using any more of the electrical-inlet seals than requisite.

Reconsideration of the type of pressure vessel was made necessary by the recent discovery by Professor Bridgman that a beryllium-copper cylinder without external support will stand 18,000 atm. with very little internal stretch. This pressure is even higher than the tensile strength. A simple vessel of beryllium-copper would have greater mechanical simplicity, and the advantage of a thermal conductivity three times higher than that of steel. However, for sealing argon, the cylinder ought to be resistant to scoring by the compression piston, and beryllium-copper cannot be hardened

nearly as much as steel. Also, temperature creep under stress occurs at lower temperatures in copper alloys than in steels. Beryllium-copper cannot be exposed for any appreciable length of time to a temperature above the lower limit of the precipitation-hardening range starting at 250°C, and in fact creep occurs at 200°C. The recrystallization temperatures of copper and iron, which involve their atomic mobilities, are respectively 200° and 450°C. At temperatures below the onset of changes such as recovery, recrystallization, precipitation, etc., the creep resistance normally increases with tensile strength, which in turn increases with hardness. Even if the particular steel found most suitable mechanically should be better than beryllium-copper in creep resistance by only as little as 100°C, the three to one advantage in thermal conductivity of beryllium-copper would be half offset by the lower maximum vessel wall temperature. Since at the higher pressures the argon has to be kept from solidifying in the central portion of the vessel, the beryllium-copper would impose a much narrower temperature channel to work in.

FERROMAGNETIC SEMICONDUCTORS

Electric and magnetic properties of polycrystalline ferrites (D. J. Epstein)

An immediate objective of the Laboratory's applied research program is to determine how the magnetic properties of square-loop and related ferrites are influenced by ceramic preparation techniques. To this end, the Ceramics Group has fabricated approximately 500 toroidal specimens (of three different chemical compositions), which have been prepared under various well-defined conditions. These specimens are being subjected to the following tests, at room temperature: a) initial permeability and loss at 10 kc and 1 Mc; b) initial magnetization curve (static); and

c) hysteresis loop (static) at a peak magnetizing field of 25 oersteds. Data for determining complex permeability at 1 Mc have been obtained on approximately 25 percent of the samples. Permeability measurements at 10 kc and magnetization runs have been initiated. Analysis of our 1 Mc data is being undertaken.

In order to estimate the influence that internal stress may have on the magnetization curve, we have measured longitudinal magnetostriction for a number of polycrystalline ferrites (Fig. 13). In all cases, measurements were made on bars approximately $9 \times 2 \times 2$ mm. The experimental results must be regarded as only preliminary; to obtain more significant results it will be necessary to repeat the measurements using ellipsoidal specimens and plot the fractional change in length against the magnetization rather than against the applied magnetic field.

Specimen 3A15-1, referred to in Fig. 13, is a square-loop body belonging to the family characterized by the hysteresis loop shown in Progress Report XII.

In addition to the foregoing measurements, the magnetostriction of a commercial square-loop body (General Ceramics 1326 B) has been determined; at about 1500 oersteds $d\ell / \ell$ was -2.5×10^{-6} .

Measurements of d. c conductivity vs. temperature have been carried out on a specimen of sintered magnetite to obtain a check on sample stoichiometry. At the transition the conductivity drops by a factor of 25 indicating, on the basis of the data obtained by Verwey and Haayman,⁷⁾ that the ratio Fe_2O_3 to FeO is about 1.03.

7) E. J. W. Verwey and P. W. Haayman, *Physica* 8, 979 (1941).

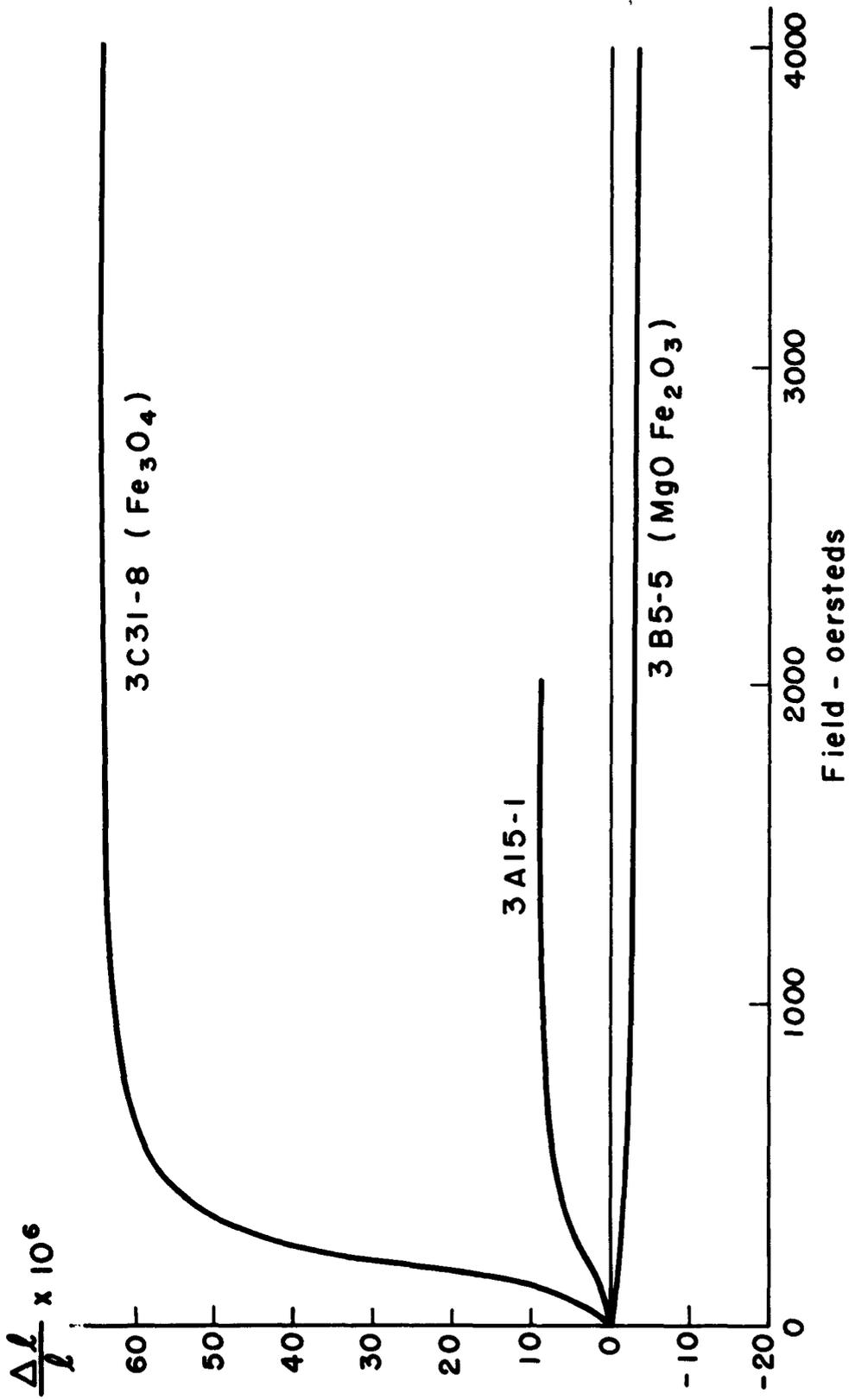


Fig. 13. Longitudinal magnetostriction for a number of polycrystalline ferrites.

The electric and magnetic properties of magnetite at low temperature

(B. A. Calhoun)

Verwey et al.⁸⁾ have suggested that the low temperature/^{transition}in magnetite is due to the ordering of the ferrous and ferric ions in the octahedral interstices of the spinel lattice and have proposed a specific ordered arrangement. An X-ray study of the crystal structure of magnetite below the transition has already been reported.⁹⁾ The c axis of the orthorhombic (ordered) structure is established along the cube edge closest to the direction of magnetization in each domain. An external magnetic field applied to a crystal of magnetite, as it is cooled through the transition, establishes a preferred orientation for the orthorhombic c axis by aligning the domains above the transition. The application of a magnetic field to the crystal below the transition can cause the c axis to switch to a new direction. This process involves a co-operative rearrangement of the ferrous ions in new sites. The equation $B_i = CT \exp(-u/kT)$, where B_i is the internal induction, C is a constant, and $u = 0.033$ ev, gives the relation between the internal magnetic induction and the temperature at which axis switching occurs. The axis-switching process is accompanied by relatively large changes in the dimensions of the sample.

The transition in stoichiometric, synthetic single crystals occurs at 119.4°K and is marked by an abrupt decrease in the conductivity by a factor of 90 in a temperature interval of 1° . The discontinuity in the conductivity occurs at the same temperature in both the warming and cooling cycles. The conductivity of a crystal cooled through the transition in zero magnetic field (Fig. 14) is isotropic below the transition. There is a marked change in the slope of the $\log \sigma$ vs. $1/T$ curve at ca. 53°K . When a crystal has been cooled in a magnetic

8) E. J. W. Verwey, P. W. Haayman, and F. C. Romeijn, J. Chem. Phys. 15, 181 (1947).

9) S. C. Abrahams and B. A. Calhoun, Tech. Rep. 61.

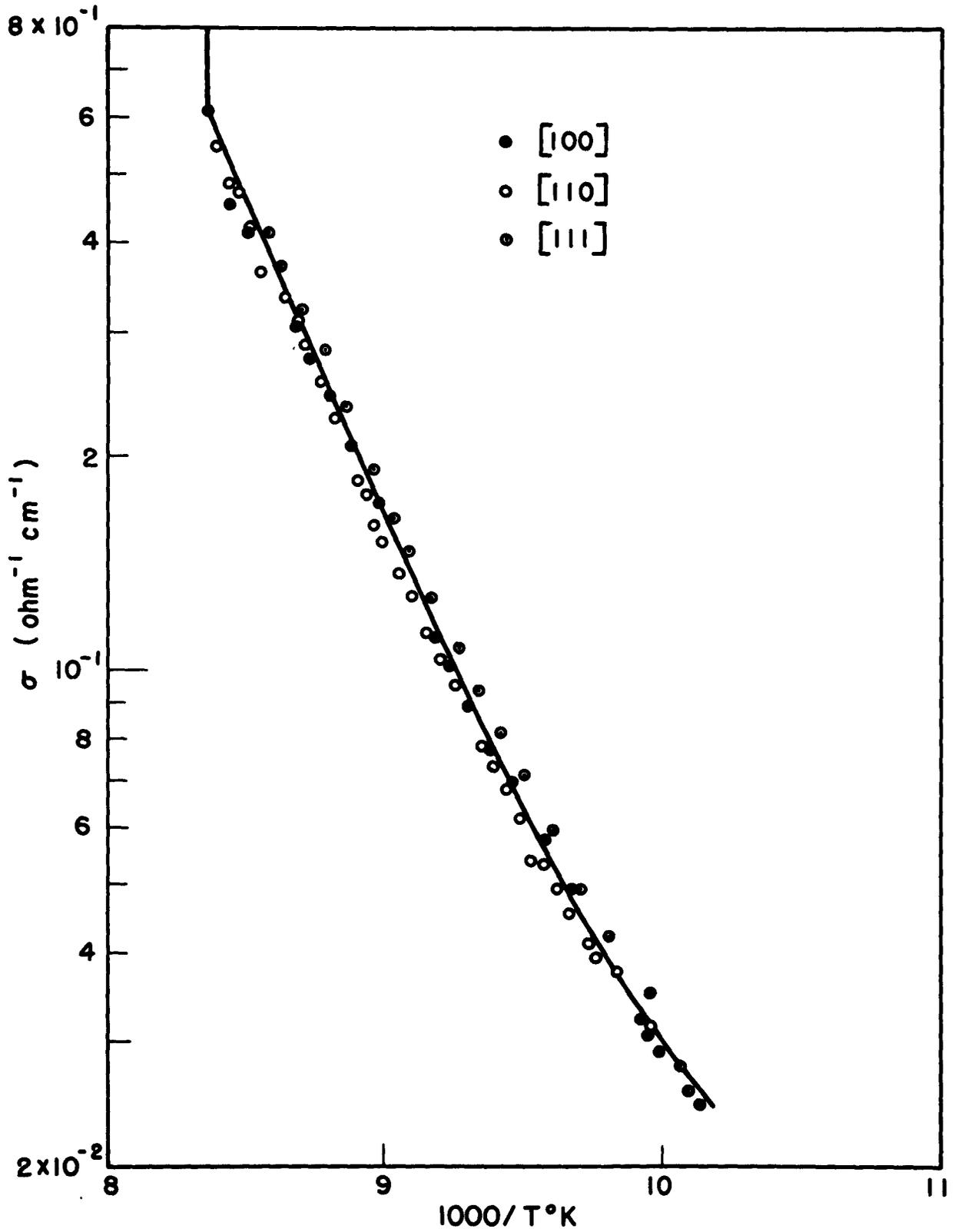


Fig. 14. Conductivity below the transition after cooling in zero magnetic field.

field to establish a preferred orientation for the orthorhombic axes, the conductivity below the transition is anisotropic (Fig. 15). It can be represented by the equation $\sigma = A + B(1 + \cos^2 \theta)$, where θ is the angle between the c axis and the direction of measurement. The ratio $B/(A + B)$ increases rapidly as the crystal is cooled from the transition to 90°K , indicating a progressive increase in the long-range order.

The anisotropy energy below the transition has the form

$$E_a = K_1 a_1^2 + K_2 a_2^2 + K_{11} a_1^4 + K_{12} a_1^2 a_2^2 + K_{22} a_2^4,$$

where the K's are the anisotropy constants and the a's the direction cosines of the magnetization. This form of the anisotropy energy is consistent with the orthorhombic holohedral symmetry of magnetite at low temperature. The anisotropy constants $K_1 = 4.0$, $K_2 = 7.0$, $K_{11} = 2.2$, $K_{12} = 13.0$, $K_{22} = 10.2 \times 10^5$ ergs/cc were determined at 85°K from measurements of the areas between the magnetization curves in different crystallographic directions. That the anisotropy energy is very much larger below the transition than above, can be seen from a comparison of the magnetization curves at 85°K (Fig. 16) with those at room temperature (cf. Fig. 20). The c axis is the direction of easy magnetization below the transition. The longitudinal magnetostriction in the c-axis direction is very small, indicating that the demagnetized state consists of an antiparallel array of domains, aligned along the c axis.

Adaptation of the magnetic loop tracer to measurements in external magnetic fields (B. A. Calhoun, D. J. Epstein, and B. Frackiewicz)

The quasi-static B-H loop tracer, initially intended for measurements of toroidal specimens, has been recently adapted to the tracing of magnetization curves for small samples in external magnetic fields up to 15000 gauss.

The sample is placed inside a tightly fitting coil in the gap of an iron

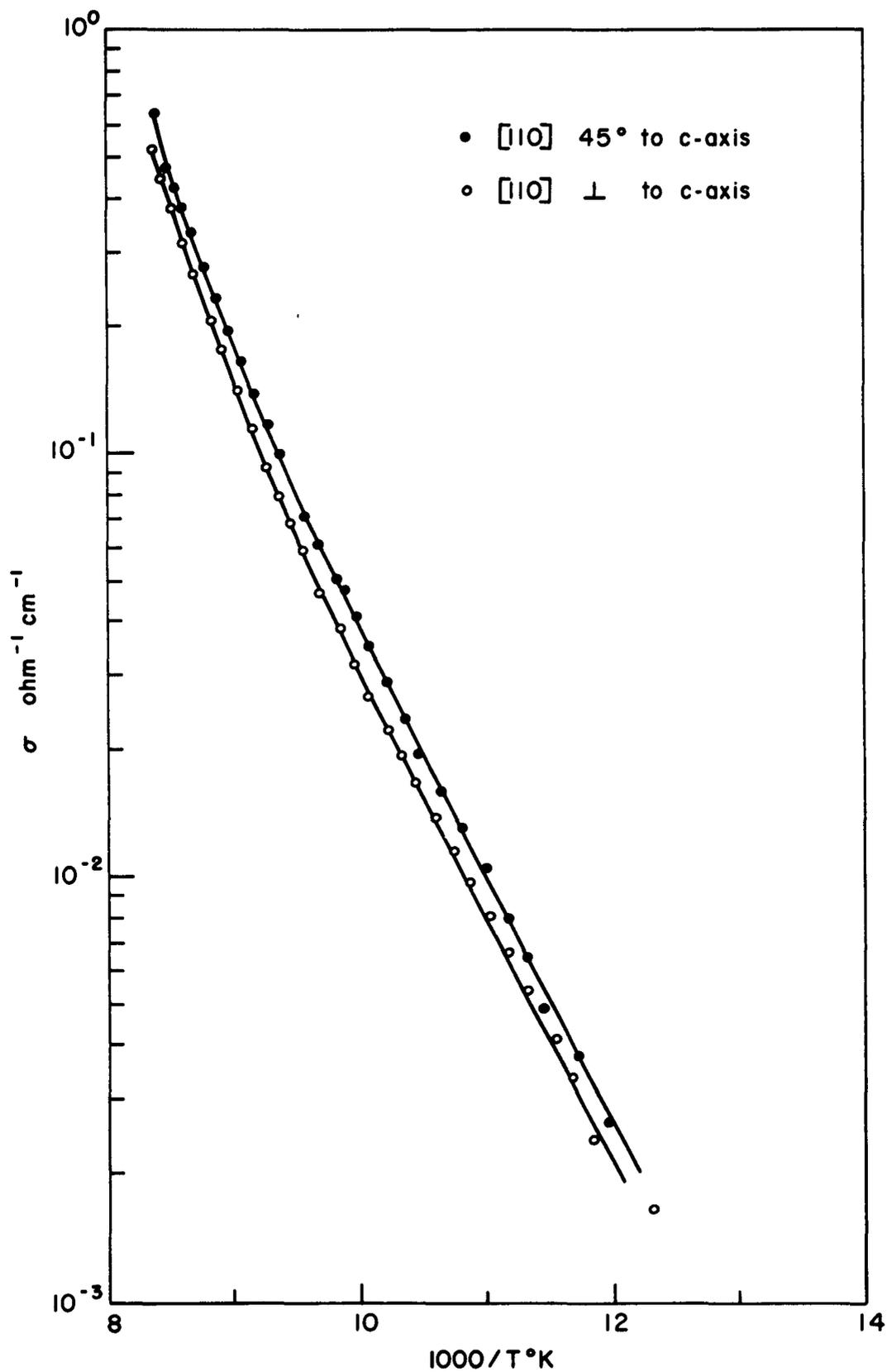


Fig. 15. Conductivity of a [110] bar below the transition after cooling in strong magnetic fields.

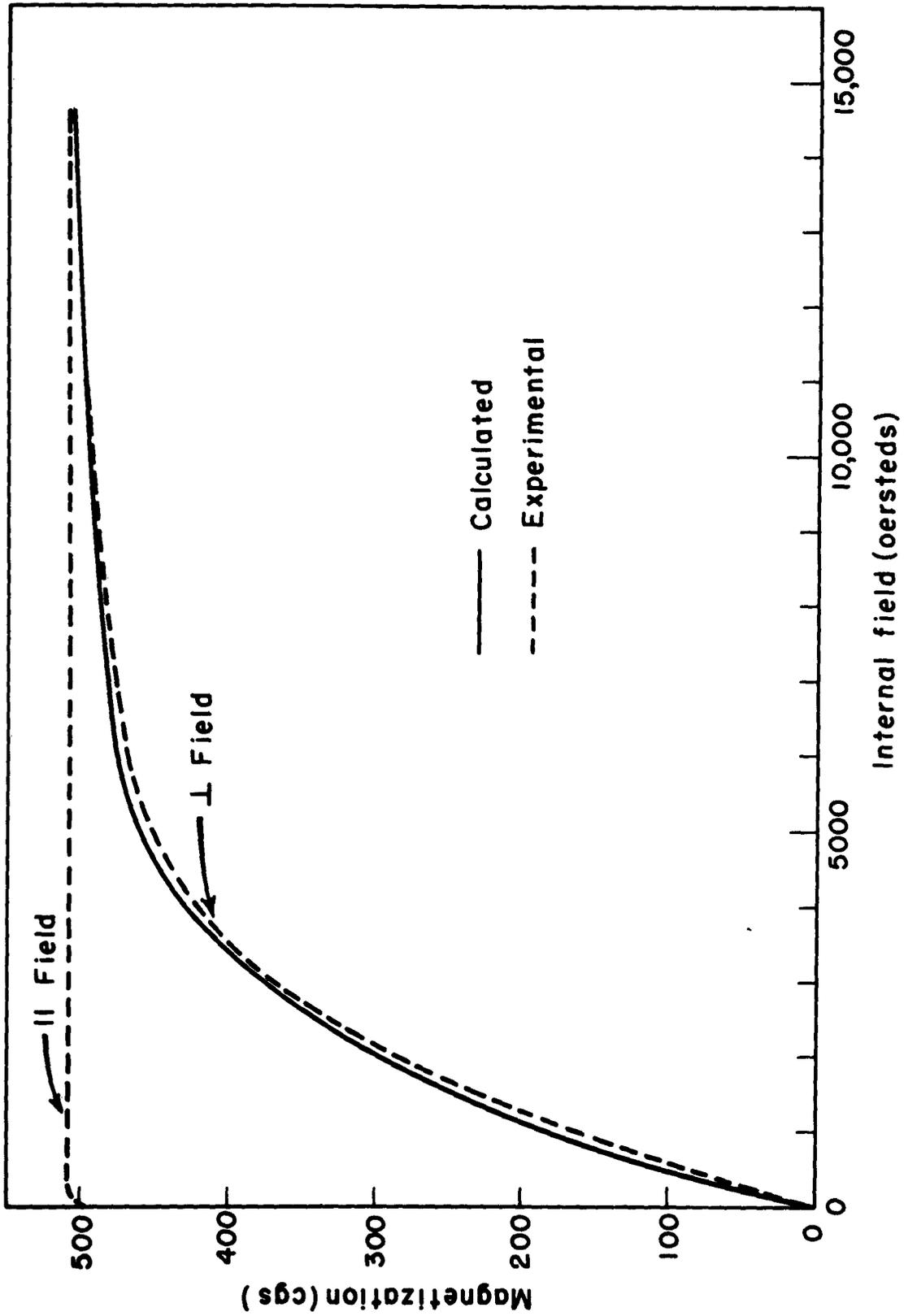


Fig. 16. Magnetization curves in the easy and hard directions below the transition. The calculated curve was obtained from the anisotropy constants.

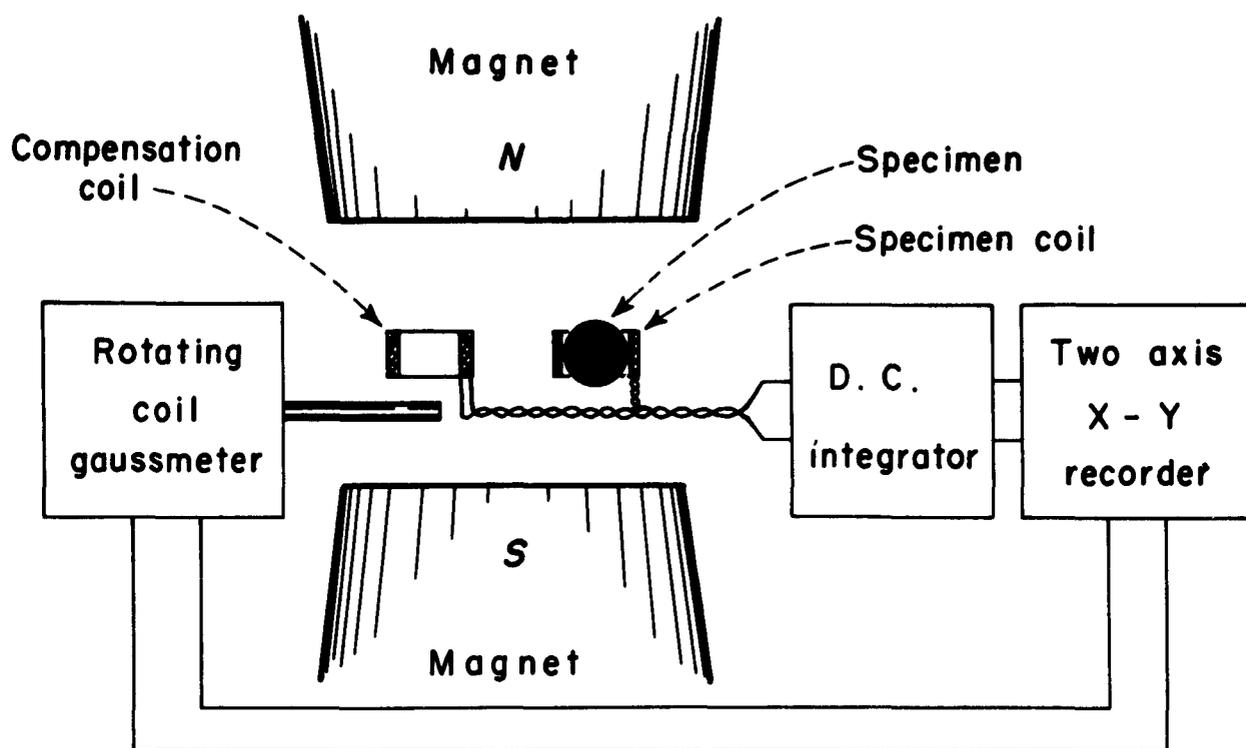


Fig. 17. Diagram of the magnetization curve tracing system.

core electromagnet. The sample coil is connected in series with a compensation coil and the two coils are so arranged that with the sample removed the combined output cancels (Fig. 17). The output voltage developed by the coils is integrated and displayed on one axis of an X-Y recorder, the H axis voltage being derived from a Rawson rotating coil gaussmeter. In Fig. 18 we show typical tracings obtained both with the sample in and with the sample removed. If the compensation were perfect, the latter curve would be a straight horizontal line. In practice, perfect compensation can not be obtained, because of slight field inhomogeneities which depend on the field strength. However, it is a simple matter to use the curve traced without sample to correct the one obtained with the sample in the gap.

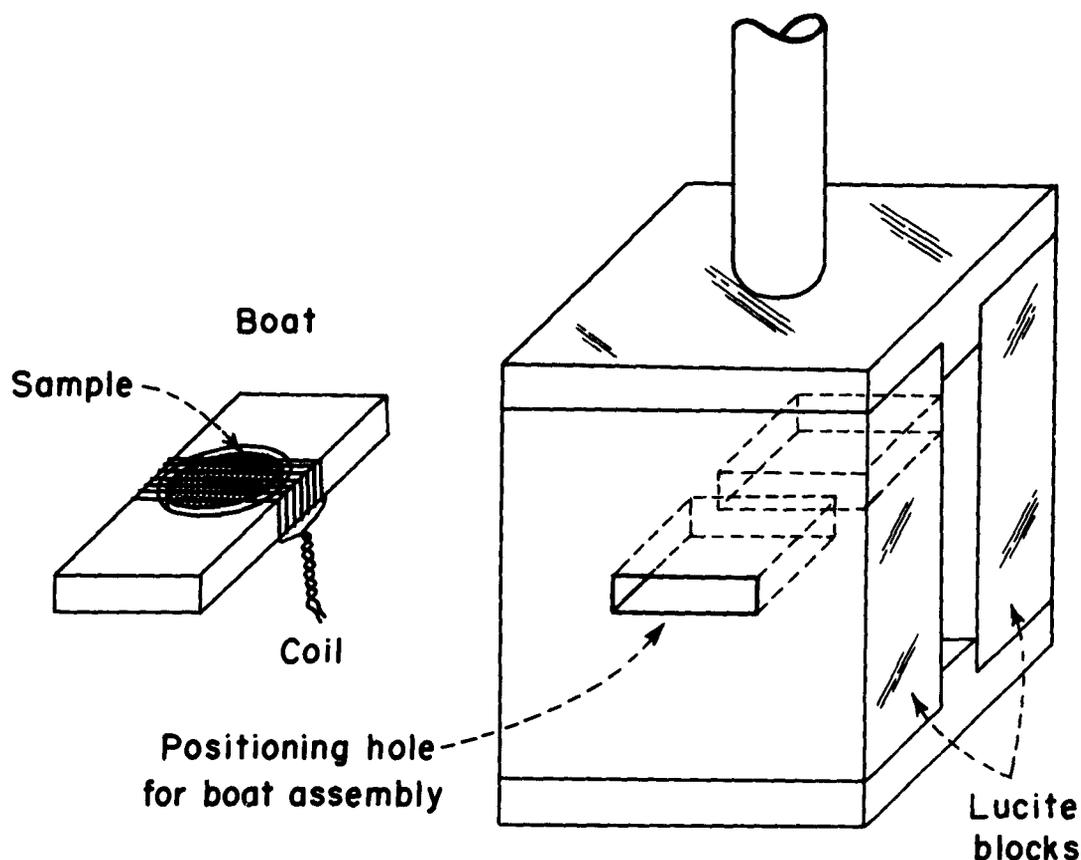


Fig. 19. Sample holder and coil.

ite grown in the Laboratory were obtained at room temperature and at temperatures below the transition.

The sample was an oblate spheroid cut in the (110) crystal plane. The axes of the spheroid were 3.84 and 0.97 mm, the calculated demagnetizing factor $N = 1.82 \pm 0.08$, while the demagnetizing factor from the [111] curve was $N = 1.75$.

The sample coil was wound on a thin polystyrene sheet and supported between two Lucite blocks (Fig. 19). It consisted of 80 turns of No. 40 enameled copper wire and had an effective area of 6.844 cm^2 . The sample fitted into a

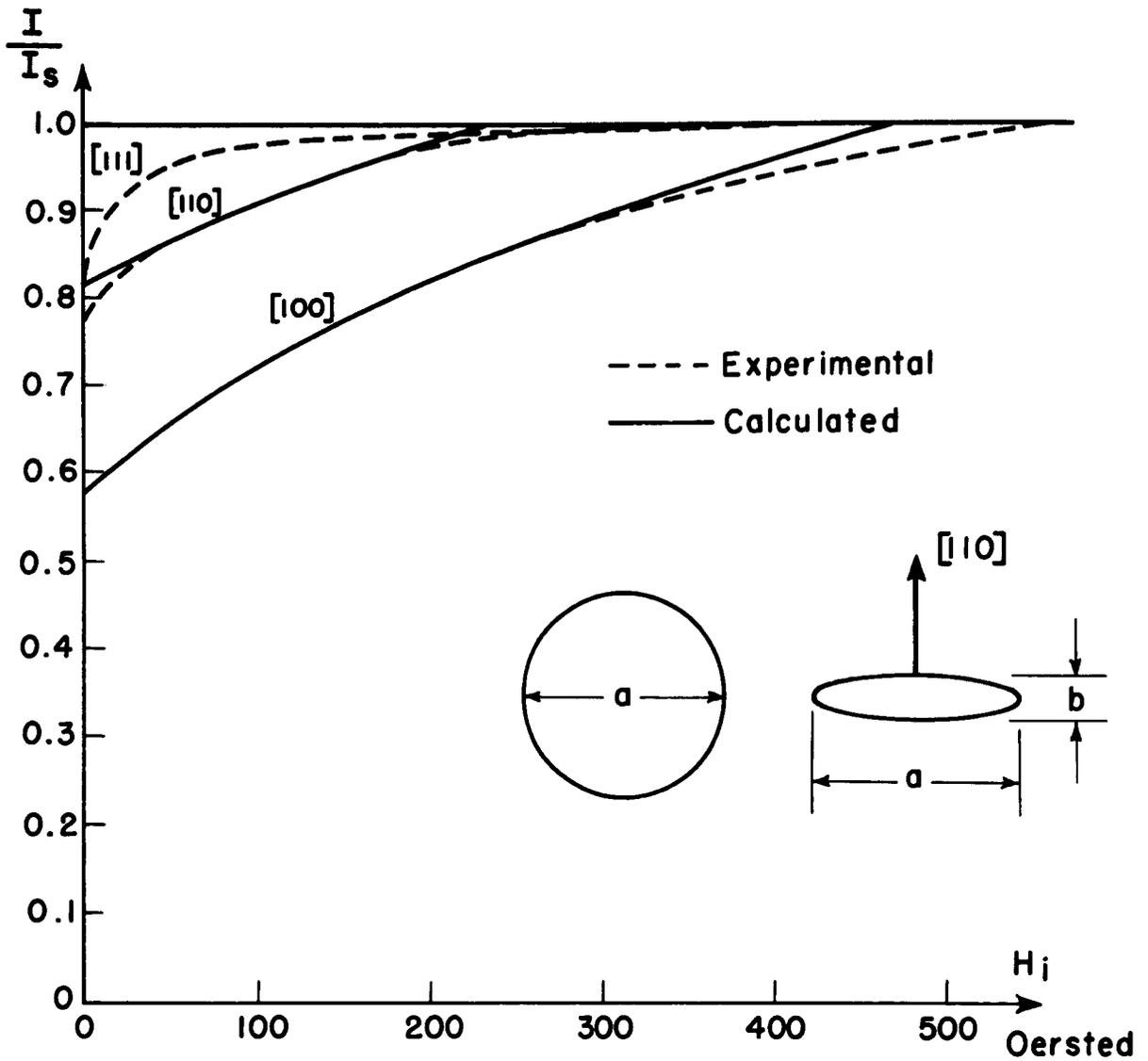


Fig. 20. Magnetization curves of magnetite at room temperature.

circular hole of a rectangular Lucite "boat" which slipped into slots in the Lucite coil-form, so that the sample was positioned in the center of the coil and yet could be easily removed and rotated in the "boat" to obtain alignment of the various crystallographic axes with the direction of the coil axis and with the field.

The design minimized the air gap between the coil and the sample, the

average width being ca. 0.006 in. With this arrangement the flux linkage between the sample and the coil was estimated at 70 percent.

The magnetization curves for the three principal directions of magnetite at room temperature as function of internal field are shown in Fig. 20 by dotted lines. The solid lines represent values computed on the basis of a simple model assuming the magnetic field orients the magnetization against the anisotropy force only.¹⁰⁾ The anisotropy constant used was -1.12×10^5 ergs/cc and the saturation moment was taken as 477 cgs units/cc.

The vibrating-coil magnetometer (D. O. Smith)

The development of the vibrating-coil magnetometer (Progress Report No. XI, p.53) is well advanced. The principal problem is to eliminate unwanted background signals arising from: 1) fluctuations in time of the main field, 2) inhomogeneities in space of the main field. To accomplish this we use a second coil (compensation coil) mounted to vibrate in phase with the first (sample coil) and connected in series opposition with it. The problem of making the two coils move in phase has finally been solved. If the compensation can be made good enough, the sensitivity should be sufficiently high to measure paramagnetic susceptibilities at a distance of 3/4 in. from a 1/4 in. specimen, which is far enough away to surround the specimen with a furnace and pressure bomb.

Preliminary tests determining the design of a pressure bomb for use with the vibrating-coil magnetometer have been completed. These tests indicate that it should be possible to reach 10,000 atm. at temperatures up to 1400°C. Construction will be started as soon as it is definitely established that the magnetometer will work.

10) F. Bitter, "Introduction to Ferromagnetism", McGraw-Hill Book Co., New York, 1937, Chap. 6.

The measurement of the magnetization curve of magnetite and, by extrapolation, the saturation magnetization at high pressure should be particularly interesting in the neighborhood of the Curie point. Alternative methods, if the vibrating-coil magnetometer should fail, are under consideration. A furnace for preliminary magneto-caloric measurements has been constructed. The molybdenum-wound tube-furnace has an inside working diameter of 1 1/4 in. and allows reaching temperatures up to 1500°C in magnetic fields of 15,000 gauss.

Regulated control system for the large electromagnet (L. E. Johnson)

The large electromagnet and its associated automatic current control have been relocated. The magnet and the current regulator circuits are installed in the Ferromagnetics section of the Laboratory, while the motor generators supplying the power for the magnet are operated by remote control and housed in another part of the building. This arrangement has greatly increased the operating convenience. The extensive shut-down period forced by the move was used advantageously to incorporate several modifications and improvements in the system before reassembly in the new location.

The magnet current-control system is an automatically self-correcting device that maintains the magnet current at any desired level; this level is continuously variable from a maximum negative to a maximum positive value. The system is a servomechanism which compares the magnet current with a specified value, measures the difference, and automatically reduces the error toward zero. Difference between the input command and the actual output may arise from two sources, namely, a sudden change in the input signal and extraneous current fluctuations. The latter may be caused by temperature changes, generator brush noise, generator structural asymmetries, and changes in generator speed caused by variations in line voltage. These fluc-

tuations are randomly distributed in time, ranging from occasional small shifts and slow drift in the d. c. value of the current to noise with appreciable components at frequencies up to 12 kc/sec; above 12 kc the noise amplitude is negligible due to the inherent filtering action of the magnet circuit. The present control system is designed to compensate for the drift and small shifts in the d. c. value of the current at any desired current level, but does not compensate for components of noise above ca. 1 cps. That is, it essentially determines the set point or d. c. value of the magnet current. Slow thermal drift and small shifts in the d. c. value are held to within one part in 10^5 . The amplitude of the remaining noise is less than 0.5 percent of the d. c. value.

Future refinement of the current regulating system calls for an additional channel whose function is to compensate for the remaining noise fluctuations. This might be accomplished by a dynamic electronic filter with a bandpass from 1 cps to 12 kc driving the magnet in parallel with the generator. Alternatively, small auxilliary coils at the magnet air gap could be driven electronically as demanded by an error-measuring device placed in the gap itself. The latter method lends itself to the use of a rotating-coil gaussmeter as the error sensing element, or to a nuclear resonance sensing element. Either method can be expected to achieve an overall precision of regulation of the order of one part in 10^5 when properly matched frequency-response-wise to the existing low-frequency channel. For present uses of the magnet the existing one-channel control system seems adequate.

Features of the present control system, as partly already mentioned above, are variability of the magnet current from maximum negative to maximum positive values with stabilization provided at any desired level of the current, quick reversibility of the polarity for convenience in demagnetization,

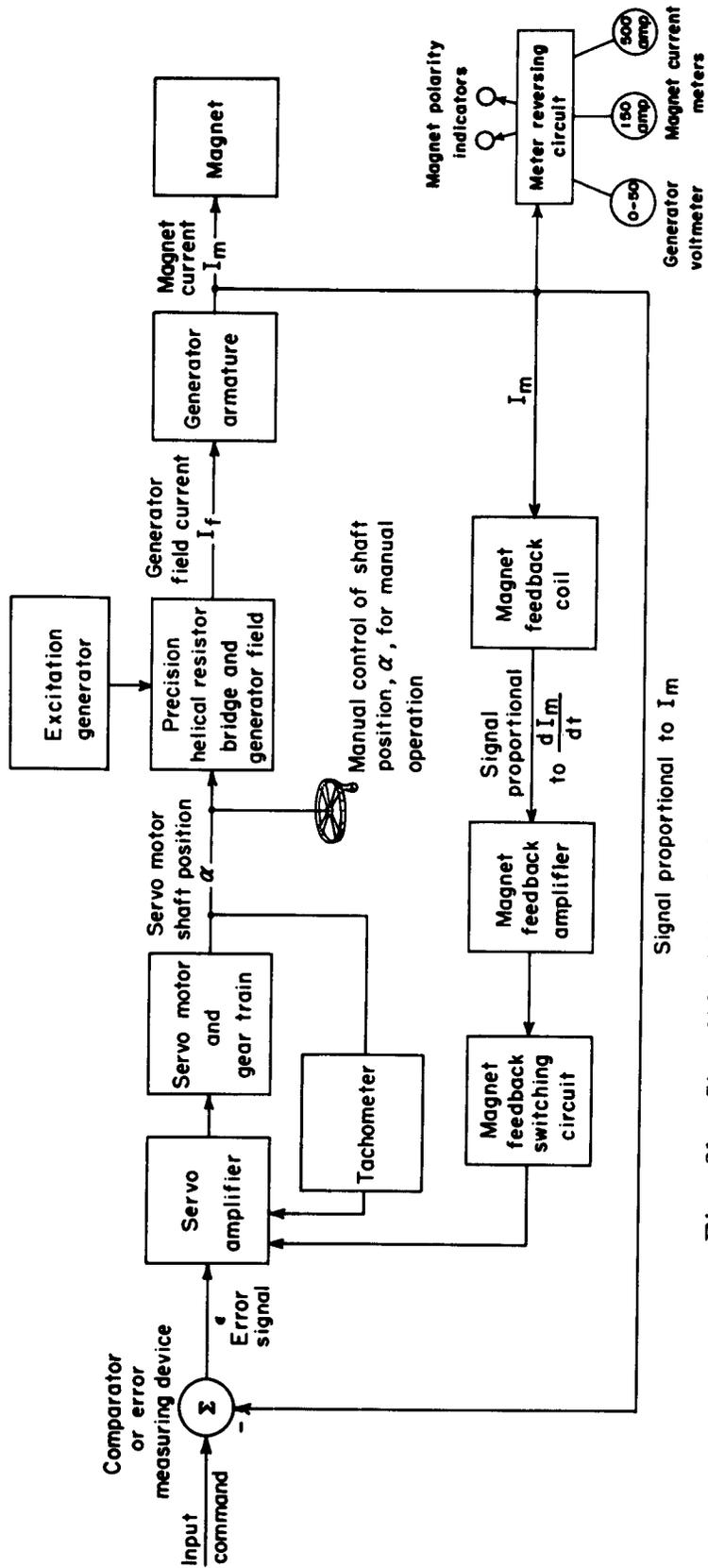


Fig. 21. Simplified block diagram of magnet control system.

and complete elimination of switching in the magnet circuit itself.

The operation of the control system is indicated schematically in the accompanying block diagram (Fig. 21).

CRYSTAL GROWING

(A. Smakula)

The laboratory for our Crystal Growing Section nears completion. The rebuilding of the rooms has been finished and the most important equipment (furnaces with temperature control units) are being installed. Our main objective is to produce crystals of improved quality for the various research projects of the laboratory. Furthermore, the study of crystal transitions and the development of new growing techniques will be emphasized. A variety of methods (optical, electrical, X-ray, and chemical) will be used to control the quality of the crystals, to study the removal of stains and to prepare samples from the crystals without the destruction of the crystal lattices.

Power control for high-temperature furnaces (W. B. Westphal and B. R. Moon)

A power controller has been developed according to the plans given in the last Progress Report except that the Variac and motor were found to have excessive time lags. The system was redesigned with thyratrons (Fig. 22) acting as a variable series control (impedance control circuit). The unit will maintain the power input at a 1500 watt load constant to 4 watts with a voltage change of 20 percent. The transformer for coupling the thyratrons to the load has a 3 kw rating and was rewound for the application. The unit is now installed for practice runs.

furnace will serve for the growing of crystals of other ferrites at temperatures up to 1900°C.

Growing of cesium halide crystals

(A. Smakula, J. Kalnajs, and V. Sils)

Cesium halide crystals are of special interest for several problems of the laboratory since they resemble in many respects the face-centered cubic alkali halides, but are body-centered cubic. CsCl transforms from face centered to body centered at 455°C. The question is whether it is possible to obtain large body-centered CsCl crystals by growing them from the melt. Cesium chloride crystals of about 2 cm dia. have been grown in our temporary equipment with a speed of 1 mm/hr in a temperature gradient of 15°C/cm. These crystals, while completely transparent, consist of sections oriented at random. The speed of 5 mm/hr led always to multiple crystallization. Whether or not it is possible to obtain a unidirectional transformation is being studied at present.

The melts were filtered through rolled Pt screens (40 mesh) to eliminate carbon particles stemming from organic impurities (Fig. 23). Vycor crucibles have been used.

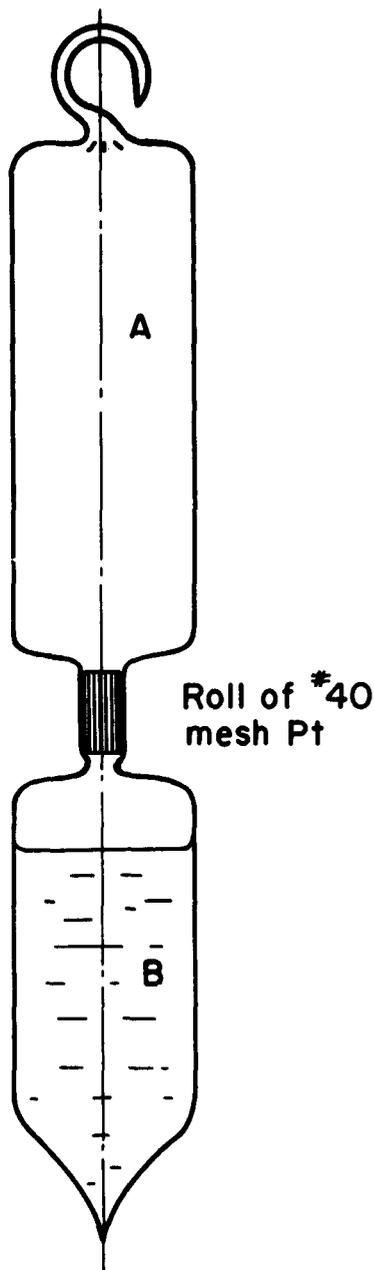


Fig. 23. Crucible with platinum filter.

CsBr and CsI crystals were perfect and very clear. However, we discovered a few weak but sharp absorption bands in the region 4.6 to 5.2 microns in both materials. CsI showed, in addition, another band system between 8.9 and 9.2 microns. The nature of these bands is under investigation.

CERAMICS

(G. Economos)

Ferrites

Ceramic toroids have been prepared from $Mg Fe_2O_4$ and of a composition producing square hysteresis loops. Various ceramic-fabricating procedures are being studied. The parameters under investigation are forming pressure, firing temperature, and firing time. In a few instances various firing atmospheres have been tried: flowing air, pure oxygen, low partial pressure of oxygen (ca. 10^{-6} atm.) and a "magnetite" atmosphere.*

Magnetite specimens have been prepared to correlate the behavior of ceramics and single crystals. Manganese ferrite poses a special problem. Slow cooling in air causes oxidation to a two-phase $Mn_2O_3-Fe_2O_3$ system. The "magnetite" atmosphere, on the other hand, appears to be too reducing, and MnO separates out.

Changes made in the square hysteresis loop composition mentioned in Progress Report XII have improved the squareness ratio (see Progress Report XII, p. 35) from 0.65 to 0.83. A closer study of these compositions is in progress supported by an extensive testing program.

* A charging ratio of $CO:CO_2$ gases which is in equilibrium with Fe_3O_4 at various temperatures (see J. Smiltens, Technical Report 49).