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<u>PHYSICS</u>	<u>PAGE</u>
Research on Luminescence and Related Phenomena in Berlin.....	130
Electron Emission of Fresh Surfaces.....	130
Piezoelectric Behavior of Cadmium Sulfide.....	130
Luminescence in Polysulfides.....	131
Scatter Fringe Interferometry.....	131
Novel Methane Analyzer.....	133
<u>CHEMISTRY</u>	
Surface Properties and Structures of Arsenic Trioxide.....	134
The Shape and Thermal Transitions of Macromole- cules.....	135
Oxygen Exchange Between Nitric Acid and Water..	135
<u>METALLURGY</u>	
Plastic Deformation of Zinc.....	137
Slip.....	137
Twinning.....	138
<u>BIOSCIENCES</u>	
Measurement of Intracellular pH by Means of a Microtungsten Electrode.....	138
The Concentration of Radioactive Alloxan in the Tissues of the Rat.....	139
<u>MISCELLANEOUS</u>	
Technical Reports of ONRL.....	139

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RESEARCH ON LUMINESCENCE AND RELATED PHENOMENA IN BERLIN

Professor H. Gobrecht (Second Physical Institute, Technical University, Berlin) is directing an active program in the field of luminescent solids and related phenomena. Some of their current work is described below.

Electron Emission of Fresh Surfaces

The radiation known to emanate from freshly evaporated or freshly deformed surfaces of metals or of salts is being studied using photographic plates (containing no gelatin) and, more recently, using counter tubes. The origin of the radiation is considered by Professor Gobrecht to be chemical reaction or chemisorption on the surface. This is strongly supported by his recent observations on antimony. The blackening of the photographic plate observed when Sb is reacted with hydrogen sulfide is very much faster and stronger than that in oxidation. Comparable blackenings are observed in periods of exposure of one day and one week respectively. Their work at present is directed towards elucidating the more detailed mechanism of this electron emission by determining the energy spectrum of the emitted electrons using retarding potentials. It should be remembered that previous workers who have observed and discussed these phenomena favored the explanation that the electron emission is associated with recrystallization (cf. Z.f. Phys., 132, 129 (1952)).

Piezoelectric Behavior of Cadmium Sulfide

It was recently discovered that cadmium sulfide single crystals show piezoelectric properties. Current work centers around the relationship between the piezoelectric behavior and irradiation by visible light. The edge of the absorption band under normal conditions was found to be 5025Å; upon prolonged irradiation with visible light this edge moves to lower frequencies.

Luminescence in Polysulfides

In agreement with earlier tentative conclusions, (cf. Z.f. Phys. 132, 111 (1952)) it has been definitely shown that the luminescence in alkali sulfides is not due to the presence of an activator but that one deals with a mixture of monosulfide and polysulfides. In the case of the potassium compounds, a binary mixture of K_2S_2 and K_2S_3 was shown to exhibit the same luminescent behavior as that reported for the polysulfides.

The alkaline earth polysulfides possess such luminescent properties to a very much lesser extent. While the phenomenon can be conveniently studied in the case of barium polysulfide, the corresponding strontium compound is barely within the range of experimentally accessible luminescent intensities.

SCATTER FRINGE INTERFEROMETRY

Dr. J.M. Burch (Bristol) showed a series of unusual interferometers at the recent Physical Society Exhibition in London. These interferometers can be understood by reference to Fig. 1. Part of a narrow beam of white light is scattered by a screen, A, and part of it continues undisturbed.

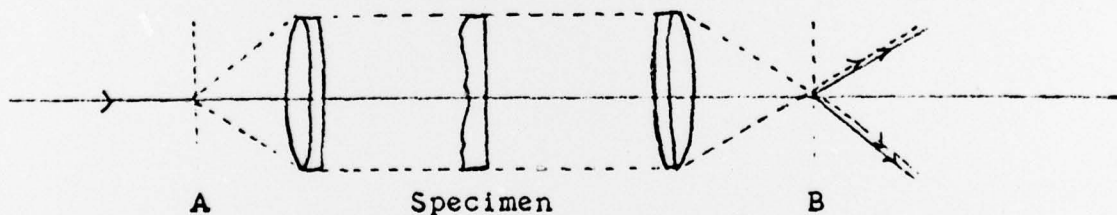


Fig. 1. Scatter Fringe Interferometry

The scattered light is collimated by a lens and illuminates the specimen whose retardation is to be observed. The scattered light is then brought to focus on an identical scattering screen, B. Part of this light is scattered a second time, and part passes through B undisturbed. This latter fraction is then capable of interfering with the portion of the original beam which has been scattered by

B but not scattered by A. These two interfering beams are indicated by dotted and solid lines respectively in the diagram. If the specimen has distorted the wave front of the primary scattered beam, this distortion will appear in the interference pattern.

It is necessary that the phase and intensity of the light scattered by the two screens be identical and this can only be achieved if the two screens are identical. This identity has been achieved by Burch in two ways: the same screen can be used twice if the optical system is folded upon itself, or the scattering screens can be plastic replicas of a rough surface.

Fig. 2 is an illustration of the first technique and serves as a method for testing microscope objectives.

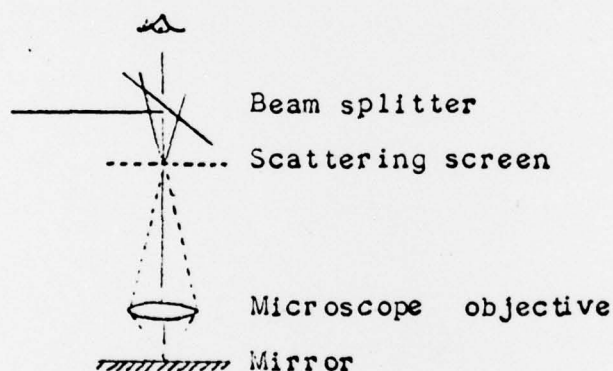


Fig. 2

An Interferometer for Testing Microscopes

In this case, where the same screen is imaged on to itself, all zonal aberrations and all even azimuthal errors are seen double while odd azimuthal errors such as coma and tilt are automatically annulled. This characteristic is shown in Fig. 3 where oblique incidence fringes can be seen on a flat metal surface while it is being turned in a lathe. The fringes are unaffected by vibration, decentering, or axial movement.

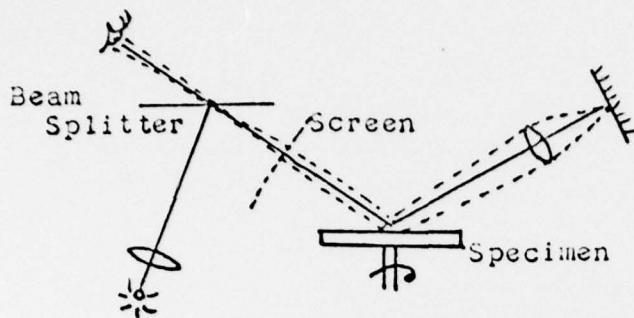


Fig. 3

A Test for a Surface Being Turned in a Lathe

It is possible to use scattering screens which scatter in one dimension only and under these circumstances the wave front errors in the other azimuth are of no consequence. This effect has been used by Burch in the examination of cylindrical surfaces and other astigmatic optical systems.

For best operation, the screen should scatter only a small fraction of the light so that the intensity of the two interfering beams will be substantially the same. It is convenient to achieve this by the replica technique using a rough metal surface which has been partially polished.

NOVEL METHANE ANALYZER

A particularly simple methane analyzer has been built by the National Coal Board and exhibited at the recent Physical Society Exhibition in London. A beam of infrared is allowed to pass through a rotating mica disc divided into segments, alternate ones of which are coated with a silicone resin the wave length of whose principal absorption band lies between 3.2 and 3.4 microns in the infrared. The result is that those wave lengths in the stated range are the only ones modulated in the near infrared spectrum. Methane has a strong absorption band at 3.3 microns and carbon dioxide, carbon monoxide, and water are substantially transparent in that region. Without further refinement the instrument is satisfactory for the control of ventilating equipment in coal mines. A more sensitive and stable model is

being built which will use two balanced beams.

The receiver is a lead selenide cell from the Services Electronics Research Laboratory. The peak of its photoconductive response lies at 3.4 microns.

SURFACE PROPERTIES AND STRUCTURES OF ARSENIC TRIOXIDE

A number of contributions by Professor Stranski and collaborators (Berlin) have helped to elucidate the surface properties, accommodation coefficients and structures of the various modifications of arsenic trioxide, As_2O_3 . More recent results complete our knowledge of this substance and lead to a consistent picture regarding its vaporization and solution behavior. In addition to the two well-known modifications, the regular arsenolite and the monoclinic claudetite, a new modification is reported from Professor Stranski's laboratories. This can be considered as a sub-modification of the claudetite and can be obtained from the material produced during the "forced" condensation of arsenic trioxide vapor (cf. Z.f. Naturf. 4a, 21 (1949)). It has a crystal structure similar to that of claudetite, in which, however, the separation between the parallel chain arrays is somewhat reduced.

The vaporization coefficient (accommodation coefficient) for the two modifications is consistent with their envisaged structures; their structures also predict the inverse behavior of their rates of solubility correctly.

The accommodation coefficient of the monoclinic modification, claudetite, is extremely small, of the order of 10^{-6} . Since this modification is envisaged as being built up of essentially infinite chains of As_2O_3 molecules, it is evident that the probability of an As_4O_6 unit breaking away is very small. On the other hand the accommodation coefficient of arsenolite is not far from unity, although it shows some peculiarities (cf. Z.f. Elektrochem. 56, 476 (1952)). Since the crystal structure of this modification is such that As_4O_6 units are already present, this is the expected behavior.

Studies are in progress in which the rate of solution of these two modifications of arsenic oxide in water are investigated. As is well known, the dispersed units in aqueous solution contain only one As atom each (for example, HAsO_2). It is now found that the rate of solution of the monoclinic modification is considerably

faster than that of the regular modification, in good agreement with the model described.

A variety of other problems are being studied under Professor Stranski's direction both at the Technical University and at the Kaiser Wilhelm Institute for Physical Chemistry. These include nucleation, crystal growth, X-ray analysis, etc. Some of these are described in Technical Report ONRL-48-53, which is available from the Technical Publications Office, Office of Naval Research, Code 740, Washington 25, D.C.

THE SHAPE AND THERMAL TRANSITIONS OF MACROMOLECULES

The shape of macromolecules and their thermal transitions are being systematically investigated in the laboratories of Professor K. Überreiter (Kaiser Wilhelm Institute for Physical Chemistry, Berlin). One of the concepts guiding Professor Überreiter in this work is that polyethylene and other relatively unbranched molecules are considerably more ordered in the liquid and glassy state than has been assumed by most other investigators.

A valuable new approach to the study of the shape of macromolecules has been the study of the melting phenomena of rapidly frozen solutions of polyethylene in naphthalene. Volume measurements indicate that such systems undergo two distinct melting processes; these are due to the naphthalene and the polyethylene respectively. The results indicate that the macromolecules cannot be completely coiled, as they would not in this case (at the lower concentrations studied) be in mutual contact and consequently could not undergo a fusion process.

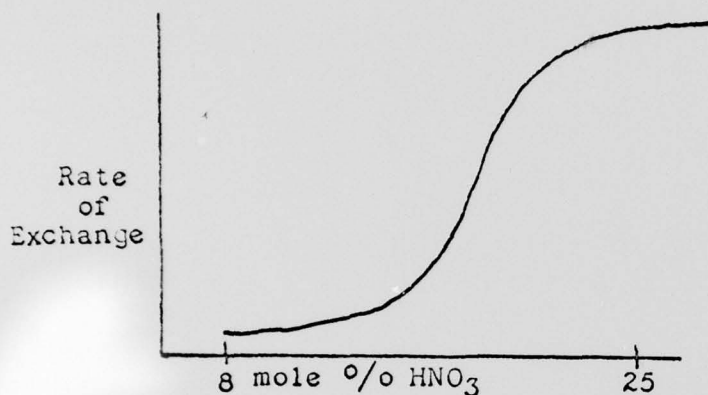
Specific heat measurements on polystyrene have been made for a number of different degrees of polymerization; it was observed that the lower molecular weight polymers (up to about a mol. wt. of 900) show a distinct λ point, but for the higher molecular weight systems this becomes a glassy transition.

OXYGEN EXCHANGE BETWEEN NITRIC ACID AND WATER

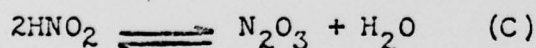
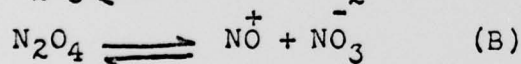
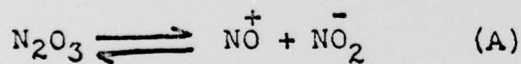
The mechanism of the exchange of oxygen between nitric acid and water has been clarified by Dr. C.A. Bunton and collaborators (University College, London). The exchange has been followed isotopically, as a function of nitric acid concentration, using O^{18} at $0^{\circ}C$. The observed behavior is radically different at low and at high nitric acid concentrations and the two regions are discussed separately below.

At nitric acid concentrations in the range 8 - 28 mole per cent, no exchange occurs if the system is rigorously free of the lower oxides of nitrogen. Very

small quantities, ca. 5×10^{-3} mole per cent of the lower oxides are found to bring the rate into a conveniently measurable range. These lower oxides on dilution in water analyze as nitrous acid. The dependence of the rate on nitric acid concentration in this range, in the presence of 5×10^{-3} mole per cent nitrous acid, is schematically shown in the figure below.

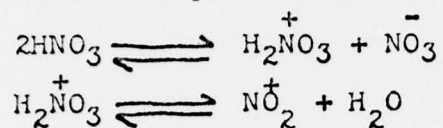


In this same range the order of the reaction, with respect to the analyzable nitrous acid, rises from 1 at the low end to nearly 2 at about 20 per cent nitric acid, and then drops fairly sharply to about 1.1. The results are interpreted in terms of a two stage mechanism involving exchange between nitrous acid and nitric acid, and between nitrous acid and water. This is shown by the following reactions:



At the low concentration end of this range the equilibrium (A) is established much more rapidly than reaction (B) and this relationship gets gradually inverted with increasing nitric acid concentration. This explains the shift from first to second order, and since both of these reactions are acid catalyzed, the rate gradually increases in this range. At about 20 mole per cent nitric acid, reaction (C) begins to be strongly displaced towards the anhydride: the order drops down towards unity and the rate no longer depends on the acid concentration.

Above 40 mole per cent nitric acid concentration, direct oxygen exchange occurs; the rate of exchange increases 500-fold in the range 40 - 60 mole per cent nitric acid. By comparison with aromatic nitration rates, the exchange was shown to involve the nitronium ion. The reactions postulated are:



PLASTIC DEFORMATION OF ZINC

Dr. R.W. Cahn (Birmingham University) has been studying the plastic deformation of zinc to observe the nature of slip produced during creep and in tensile test. He has shown for the first time that prismatic slip can occur in zinc.

Slip

In a first set of experiments, a study was made of the plastic deformation in polycrystalline zinc specimens during the progress of creep. Employing an X-ray back reflection technique, he used the beam to straddle two cells in a zinc grain. Thus he was able to determine the orientation relationship between the two cells and follow the change in this relationship during creep. At a temperature of 250°C it was found that deformation occurred entirely by basal slip; it was observed that all the slip roller axes lie in the basal plane. At 350°C, however, it was found that the roller axis was some axis between the c direction and the basal plane. Such an observation indicates that slip is not merely basal but includes some non-basal deformation such as prismatic slip and/or pyramidal.

To clarify the precise nature of the slip observed in creep at 350°C, some further experiments were conducted on single crystals of zinc. Specimens were oriented such that no basal slip could occur and were subjected to a tensile stress. Crystallographic analysis showed that at a temperature of 350°C prismatic slip definitely occurred. Further, the crystal was oriented such that if basal slip were to occur it would rotate the crystal one way, whereas if prismatic slip were to occur it would rotate the other. The observed rotation was in the direction compatible with prismatic slip. Further research is needed to determine whether pyramidal slip truly occurs. At a temperature of 250°C, a single crystal of zinc with its basal plane one or two degrees away from the non-basal slip orientation in tension,

produced a negligible amount of basal slip followed by fracture.

Twinning

Dr. Cahn is also investigating twinning in single crystals of zinc at room temperature. He has observed that if stress is applied to a crystal oriented such that it will not slip, a twin does not form. The result of stressing is that instead of forming a twin the crystal fractures at a stress four or five times greater than the critical resolved shear stress. However, if some localized heavy slip is produced in the specimen by a pin prick, it is found that the stress necessary to produce a twin is markedly decreased. Cahn thus believes that the energy required to nucleate a twin is high, but that required for its growth is relatively low. In this connection it is interesting to cite the work of Galber (Russian Journal of Physics 2, 1 (1947)) on the formation of twins in calcite. Calcite does not slip, and thus provides an interesting material in which to study twinning. It is reported that the stress required to nucleate a twin is 3000 gm/mm^2 while that necessary to propagate it is only on the order of 200 to 300 gm/mm^2 . A further point of interest in Galber's work is that if a twin forms in calcite and does not progress all the way through the specimen to the opposite side, fracture initiates from the tip of the twin. This occurs because the material cannot slip and consequently is unable to form an accommodation kink at the end of the twin to relieve the intense shear stress at this point.

MEASUREMENT OF INTRACELLULAR pH BY MEANS OF A MICRO-TUNGSTEN ELECTRODE

P.C. Caldwell of the Biophysics Department, University College, London, has devised a tungsten micro-electrode of about 15μ which can be inserted into large muscle fibers such as those of the crab, Carcinus maenas, which are usually between 100 and 600μ in diameter.

The electrode potential of tungsten, in common with that of a number of metals, varies with pH (J.R. Baylis, J. Industr. Engng. Chem. 15, 852 (1953)), and the micro-electrode can therefore be used for measurements of intracellular pH. This is done by inserting a tungsten micro-electrode and a KCl micro-electrode into the same fiber and then noting the change in potential between them when both are withdrawn into the bathing fluid. Since the pH of the bathing fluid can be determined with a glass electrode, that of the inside of the fiber can be calculated from the change in potential.

Determination of the pH of a muscle fiber with both the glass and the tungsten electrodes gives the same value, and it is therefore unlikely that there is anything inside the muscle fibers which would cause the tungsten electrode to give an anomalous potential.

Twenty-eight determinations on fibers whose resting potentials lay between 50 and 70 mV gave a mean value for the intracellular pH of 6.75 (S.D. = 0.24). Eleven determinations of the pH of the plasma surrounding the fibers have given a mean value of 7.53 (S.D. = 0.37). Hence it appears that in the intact animal the distribution of hydrogen ions between the cell and plasma approaches that predicted by the Donnan equilibrium expression.

THE CONCENTRATION OF RADIOACTIVE ALLOXAN IN THE TISSUES OF THE RAT

R.G. Janes of St. Thomas's Hospital Medical School, London, has labeled alloxan monohydrate in either the 2- or 5- position with C¹⁴ and injected this material subcutaneously in adult rats. The concentration of the alloxan monohydrate in tissues was determined at intervals from five minutes to six hours, and the excretion rate was followed up to 22 hours.

The radioactivity of the kidneys and plasma was the highest and reached its peak in about 15 minutes. The other tissues studied, including the pancreas, had very low C¹⁴ concentrations. Approximately 95 per cent of the labeled alloxan was excreted in 22 hours. Only minute amounts of the radioactivity appeared in expired air or as urea.

Janes concludes from this study that the toxic effect of alloxan on the pancreatic islets is dependent on the specific sensitivity of beta cells, and not upon an excessive concentration of this material in the organ.

TECHNICAL REPORTS OF ONRL

The following reports have been forwarded to ONR, Washington, since the last issue of ESN. Copies may be obtained from the Technical Publications Office, Code 740, Office of Naval Research, Washington 25, D.C.

- ONRL-36-53 "A New Small Air Liquefier" by W.D. Hayes
ONRL-48-53 "Nucleation, Evaporation, and Related Problems in Crystal Chemistry" by G.J. Szasz

- CNRL-49-53 "Faraday Society Discussion on Solutions
of Non-Electrolytes" by G.J. Szasz
- CNRL-61-53 "The Phase Diagram and Properties of Titanium-
Silicon Alloys" by E. Epreman
- CNRL-62-53 "A New Design for a Hydraulic Fender" by
W.D. Hayes

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