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Color Phenomena in Polymer Fracture*

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Thin layers derived from the matrix are produced in the fracture plane of some polymers prior to ultimate failure. A certain amount of the evidence would indicate that these layers consist of an oriented array of molecules. Fracture isolates these molecular segments in a thin film with physical properties differing from that of the matrix in which they originated. These films produce interference phenomena which in some cases are useful in elucidating structure and origin.

1. Introduction

It is difficult to ascertain when the strong colors on the fracture surfaces of polymers were first observed. In 1956 Busse, Orowan, and Neimark [1] [1] exhibited some surfaces produced by fracturing large notched plates of polymethyl methacrylate. About a year before, somewhat more diffuse colors had been observed on tensile specimens broken under static conditions [2] at the National Bureau of Standards [3]. Although Wolock et al. [4] and Berry [5] both reported observing colors associated with specific fracture markings, the first detailed discussion of the phenomenon was published by Higuchi [6]. Many of the same observations were made independently and reported by Wolock et al. [7] and Berry [8].

It is entirely possible, of course, that colored fracture surfaces were observed earlier than the period mentioned. Higuchi, for example, indicates that the experiments of Benbow and Roesler [9] must have produced such surfaces. Thus far almost all colored surfaces that have been studied have been produced on polymethyl methacrylate.

2. Fracture Color Patterns

The colors arising from interference in subsurface cracks are not considered in this discussion. Although they produce an optical effect which often appears quite close to the surface its origin is obvious and easily ascertained.

There is considerable variety in the color patterns obtained by various fracturing techniques. Those found on statically tested tensile specimens are weak, usually located about the periphery of the mirror area and are found on relatively few specimens. They are not usually associated with an individual structure, such as parabolas, but cover an area which encompasses a number of figures or structures without any specificity of color or tint for each element. Fractures exhibited by Busse and his coworkers were more brilliant and formed bands transverse to the direction of fracture. Higuchi used notched sheets 3–5 mm thick and 40 mm wide. The exact conditions of test are not described. His figures show a variety of patterns. Most of the conic sections are differentiated in color from their surround and occasional transverse bands cause a reversal of colors in the banded area. He finds that the colors on the two fracture surfaces are generally complementary and that the red areas are raised and the green are depressed relative to the specimen matrix.

Wolock [7] produced similar colors on fracture surfaces of polymethyl methacrylate specimens which had been broken by rapid overstress. Films of diffuse color were formed on surfaces of cast polystyrene broken in the same fashion. Specific colors, however, were not related to the individual structures on the polystyrene fracture faces. Somewhat later, Wolock noted the intense differential color produced on the relatively smooth fracture surfaces of specimens of polymethyl methacrylate tested for resistance to crack propagation. Many of the observations to be discussed were made on fractures produced in these specimens (fig. 1), which were broken by loading through a cross head moving at 0.008 mm/sec (0.02 in./min). These contain four types of color patterns or characteristics (fig. 9). The first is observed in the slow-growth area and actually consists of two types of patterns, namely, multicolored areas (fig. 9b) and single-colored areas (fig. 9a). The next pattern (fig. 9c) is composed of irregularly shaped elongated figures. These figures develop, in the third pattern of this series (fig. 9d) into oriented forms that lead, in many cases, into parabolas. The last area (fig. 9e) is darker (less reflective) with little or no color and containing many crowded parabolas in which only the foci are very distinct. The significance of the geometric markings on the fracture surfaces have been dealt with extensively in the literature [10–12] and will not be discussed in this paper.

A characteristic blue color is also found on fracture surfaces of hot-stretched polymethyl methacrylate. These surfaces are produced by cleaving the stretched sheets through the thickness dimension. The colors are not associated with specific markings.

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[1] Figures in brackets indicate the literature references at the end of this paper.

3. Optics of Color Formation

It can be demonstrated that the strongly colored areas of the fracture surfaces produced in crack propagation specimens are covered with a soft thin film. The presence of this film is a prerequisite for the development of color. In figure 10 a soft swab has been wiped across a fracture surface with removal of essentially all of the tint. Under room conditions the color will disappear from crack propagation specimen surfaces over a period of months. The time for color disappearance varies greatly, however. Colors on ordinary tensile specimens and on cleaved, stretched material faded in the course of a few weeks, whereas those of Busse et al. [1] were still intense after almost a year. Storage at temperatures of 50 to 60 °C causes rapid loss of color in crack propagation specimens in well under an hour. Loss of color under these circumstances is not necessarily associated with complete loss of the film. Even when the colors have completely disappeared an oily film still occupies the surface. The disappearance of color by aging or heating takes place by fading of the original tint without any pronounced color shift [6, 7].

Concentrated vapors of a number of organic solvents such as toluene and methyl chloride will often eliminate colors in a matter of minutes. In this case a change in tint is involved as can be seen in figure 11. The first color changes seem concentrated along level difference lines and a few other structures on the fracture face.

Another method of affecting color intensity of parts of the film is by exposure to low pressure. This can be demonstrated by placing one of the matching surfaces of a crack propagation specimen in a container maintained at approximately $1 \cdot 10^{-5}$ mm Hg pressure. After removal it will be found that almost all of the color is removed from the slow growth area. The color intensity over the remainder of the fracture is apparently the same as that of the matching surface which had been stored outside of the vacuum chamber. Microscopic examination and micromanipulation of the film on the slow-growth area of the fracture surface indicates that this portion is thicker than the rest of the film.

The fact that much of the surface is little affected by high vacuum makes it possible to apply the methods of multiple-beam interference microscopy to the study of the color phenomenon. Figure 2 is a multiple-beam interferogram of a portion of a fracture produced in a crack propagation specimen. The field shows a portion of the first parabola-containing area. A layer of silver has been deposited over the surface film and the fringe displacement over the composite surface is shown. It would appear that the adjacent areas covered by the contribution of different secondary fractures are consistently displaced by about 1500 Å. When the color is removed by aging or heating before the specimen is prepared for interference microscopy this displacement remains about the same.

Assuming first-order interference Higuchi [6] has estimated a film thickness range of 400 to 700 mμ from the observed colors. Using a soft swab to remove the film produces a swath with a depth of less than 200 mμ in the surface as measured by multiple-beam techniques (fig. 3). This experiment was repeated by focusing the radiation from a zirconium arc lamp on a small area of the fracture surface until all the color had disappeared and continuing the treatment until the film was scarcely detectable in the irradiated portion by microscopical examination. The depth of the circular depression was again found to be somewhat less than 200 mμ. Although the film in the slow growth area may exceed this figure, its sensitivity to vacuum prevents direct measurement. Figure 3 also shows the increased reflectivity of the treated area. Apparently swabbing or heating tends to produce a surface that is smoother than the original face of the film.

The presence of the film led Higuchi [6], Wolock et al. [7], and Berry [5, 8] to postulate its formation by the pulling out of chain ends and the scission of chains. These segments would have been extended in the fracture process and thus could have a somewhat different density than the matrix. The chain segments in only a relatively small thickness of material would be extended during the fracture process. Oriented layer and matrix would differ sufficiently in refractive index to lend themselves to the development of thin film interference as shown figure 4. In any given region, an average height would predominate which would result in the color observed. As the chain segments composing the film relax, the distribution of heights remains the same but the number of segments remaining in the film decreases so that the same color is present but is diminished in intensity. This would account for the fading observed with no change in color. Solvent vapors produce different changes in the structure reflected by the change in color withdrawal. The derivation of the film from the matrix would lead to a structure whose properties would differ from the matrix to the greater degree at the exposed face and tend to blend into the matrix at the other limit. Although the blending section close to the matrix could contribute to the optical effect, it would be difficult to establish its boundary by the methods described. Hence failure to realize the theoretical film thickness is probably to be expected.

Tolansky's thin film technique [13] permits corroboration of this explanation of the origin of the color phenomenon with a simple model. A fracture surface with the colors removed by aging or some other method is covered with a totally reflected film of silver *in vacuo*. A drop of a dilute solution of Canada balsam in xylene is placed on the silvered surface and allowed to drain by tilting the specimen. The surface of the film of Canada balsam in contact with the fracture surface replicates the topography of the surface. The upper surface of the film, however, does not contour the fracture surface but stretches itself into a single plane for considerable distances. On illuminating such a surface two-beam interference fringes will be formed. Figure 12 illustrates a portion of a fracture colored by thin film interference. The wrinkling probably caused by solvent attack on the silver and plastic substrate is noticeable but otherwise there is considerable similarity to the fracture-produced colors. Even the slight coloring observed in the rough, low-reflecting zone is almost identical (fig. 13) with that observed on the original fracture surface.

Considerable fringe broadening is apparent in the interferogram in figure 2. This is at a minimum in the initial, smooth, slow growth area and increases toward the crowded parabola portion. An electron micrograph of these two areas, figures 5 and 6, shows the change in surface granularity responsible for the fringe broadening. Although the surface is sufficiently smooth up to the first parabolas to permit the use of multiple-beam interference, the final surface is so rough that the method fails. Figure 6 reveals this surface to be sculptured with thin leaf-like projections. It is doubtful that a fragile film in the millimicron thickness range could be formed there. Only isolated fragments of color are found in this area.

Replicas for electron microscopy were produced by allowing a dilute solution of methyl cellulose to dry on the fracture surface. Despite its limited resolution, which is well illustrated in figure 6, this method was chosen because it would impose the least abuse on the polymer matrix. On stripping the film from a surface originally bearing color, the surface was found to be colorless. A carbon replica was made from the negative and shadowed by vacuum deposition. The loss of color would be caused either by the stripping of all or part of the interference film with the cellulosic replica or by disorienting it with the replicating solution.

Before replicating the surfaces in figures 5 and 6, an attempt was made to remove thoroughly the surface film by heating for 96 h at 50°C. For purposes of comparison replicas were also prepared from a surface bearing bright interference colors. In most cases the replicas from the unheated colored surfaces bore numerous droplets over a considerable portion of the fields examined, as in figure 7. It would seem that a portion of the film is removed and subsequently disrupted by the replicating process. These observations, moreover, pinpoint one of the difficulties encountered in preparing high resolution replicas of a polymer fracture.

This discussion has been limited almost entirely to polymethyl methacrylate. Attempts to produce color on a number of other polymers by varying loading rates and thermal conditions have been largely unsuccessful. However, polystyrene has occasionally yielded colored surfaces, as shown in figure 14.

4. Color Patterns in Crazing

Niegisch [14] has recently convincingly demonstrated the presence of oriented material in craze cracks produced in long-time tests of polycarbonates. Although this polymer did not exhibit brittle fracture under the test conditions, the ultimate elongation being about 100 percent, some evidence of similar "craze matter" in polystyrene and polymethyl methacrylate was also presented. From these data, Spurr and Niegisch [15] have offered an attractive theory for the development and structure of craze cracks in thermoplastics. Probably the most important contribution of their observations is the support they provide for the conclusion that the appearance of crazing does not mean the end of structural integrity or continuity in the stressed specimen.

The matter in polycarbonate cracks is visible by difference in refractive index probably coupled with some scattering from micropores. However, during some routine tests of standard tensile specimens [2] of acrylonitrile-styrene copolymer, a striking color phenomenon was also noted in the craze cracks of this material, as seen in figure 15. These specimens were injection molded and the stressed skin was apparently responsible for the formation of internal cracks which sometimes encompassed one-half or more of the specimen cross section. These tensile specimens, when loaded at a head speed of 0.008 mm/sec have the load-elongation characteristics shown in figure 8. The large internal cracks suddenly appear apparently full grown at the top of the straight line portion of the curve but without any discontinuities in loading. Ultimate failure occurs through one of these large cracks.

It was originally assumed that the optical effect was due to an interference system involving a void. On examining the fracture surface, however, it was found that a pattern similar to that noted in the intact craze cracks was found on one surface or parts of one surface, as shown in figure 16. The matching surface was devoid of color. Colors observed in the intact cracks are more intense than those found on fracture surfaces. Critical microscopical examination established the presence of a film on the color-bearing surface. Unlike the film found on polymethyl methacrylate fracture surfaces, the film is integral and shears cleanly from one of the crack faces. Edges of the film are sometimes curled free of the matrix on which they lie but mechanical removal of any significant amount of film appears impractical.

If the film-bearing surface of the fracture is examined at higher magnification, some additional details can be resolved. As in the intact cracks the peripheral area of the fractured craze is covered with fringes of equal thickness. The film in figure 17 bears first to fourth order colors which indicates a thickness of approximately 2 μ. Thicknesses of this magnitude are not uncommon although many cracks with much thinner films are found. In general, however, the film is much thicker than those found on the fracture surfaces discussed in the earlier section.

The center of the film-bearing area is likely to show more disorder. Colors are bright and the fringe pattern has contours that seem unrelated to film thickness. Such an area occupies about half the field in figure 18. The film is wrinkled and stretched, especially in the regions surrounding the small holes. Evidently it has been slightly dislodged from the substrate during fracturing.

In a few small areas the conic sections characteristic of brittle failure sculpture the face of the fractured craze crack as shown in figure 19. If the crack surfaces are connected by a thin layer of oriented molecules as originally suggested by Bessonov and Kuvshinskii [16] and developed by Spurr and Niegisch [15], the connection may not be as tenuous as might be suggested by a consideration of the properties of the film on polymethyl methacrylate surfaces. Besides its association with the brittle mode of failure, the crack material is much more resistant to heat, aging, and solvents. Exposure in an oven at 70 °C for 48 h produced little change in the appearance of the film on the acrylonitrile-styrene copolymer fracture surface. Prolonged heating at this temperature caused some fading of the colors and a great increase in the pitting, which is visible in the periphery of the film in figure 17. Colors in the intact cracks were unaffected by weeks of heating at 70 °C or exposure to solvent vapors that did not cause gross matrix deterioration.

Spurr and Niegisch [15] detected "craze matter" in cracks of polystyrene and polymethyl methacrylate. Its presence was much more difficult to demonstrate than in polycarbonate and it was possible to record photographically. Recently, however, the description of the work of Bessonov and Kuvshinskii [17] became available. They had observed phenomena closely related to those described in our acrylonitrile-styrene copolymer in specimens of "annealed polystyrene." Their specimens with a section of 2×2 mm and a free length of 15 mm were maintained at a temperature of 110 °C for 30 min and then cooled rapidly by plunging them into liquid nitrogen. These specimens were then loaded to failure at a rate of deformation equal to 0.04 mm/sec. The pretreatment resulted in the formation of a stressed skin and a 20 percent increase in tensile strength. Failure was preceded by the formation of large internal cracks as in the acrylonitrile-styrene copolymer. They report that the material filling these cracks remains on the fracture surface. Part of this forms interference fringes and clings to the matrix but part can be stripped. They suggested that the film consists of the bundles of linear polymers discussed by Hsiao and Sauer [18] and Kargin et al. [19].

Since certain details necessary for comparison with previous results were not given in this report, the work was repeated using standard tensile specimens [2]. The preliminary heating was extended to 2 hr in view of the larger cross section. These specimens were loaded to failure at a head speed of 0.008 mm/sec Large internal cracks formed shortly before failure and failure occurred through cracks encompassing one-third to one-half of the cross section. No color phenomenon was visible in any of the cracks on gross examination.

On microscopically examining the fracture surface a few faint interference bands could be observed. The film responsible for these adhered strongly to one of the surfaces. The remainder of the crack was partly covered on both faces by a transparent film. The film was partly detached and partly loosely attached to the surface. It was easily stripped and pieces 5 or 6 mm^2 were removed with forceps. The film was lower in refractive index than the matrix. It appears to have good physical properties and the relatively large areas available should permit the use of transmission electron microscopy, infrared spectroscopy, and other instrumental techniques in elucidating their structure and mode of formation.

Attempts to produce large cracks in specimens of polymethyl methacrylate by this technique have thus far been unsuccessful.

5. Summary

Thin layers derived from the matrix are produced in the fracture plane of some polymers prior to ultimate failure. A certain amount of the evidence would indicate that these layers consist of an oriented array of molecules. Fracture isolates these molecular segments in a thin film with physical properties differing from that of the matrix in which they originated. These films produce interference phenomena which in some cases are useful in elucidating structure and origin.

The suggestion has been made that these oriented layers provide structural continuity in polymers after optical inhomogeneities are produced by stress. This is supported, at least superficially, by observations on polycarbonate and acrylonitrile-styrene copolymer. In these materials intact craze cracks appear to be filled with homogeneous or oriented material. The film observed in intact cracks of "annealed" polystryene, however, shows some disorder. Folds and wrinkles are almost always present and it is difficult to attribute any load-bearing function to this structure.

It is possible that the two types of film formation may not be as closely related as has been assumed during much of this discussion. Further experimentation may provide the necessary data for supporting the assumption. If the mechanism proposed for the formation of these films is correct, we would expect to detect them on a wide range of polymers when failure is produced under suitable conditions. The search for, and study of, these films is continuing.

The authors acknowledge valuable discussions with Dr. Samuel G. Weissberg.

6. References

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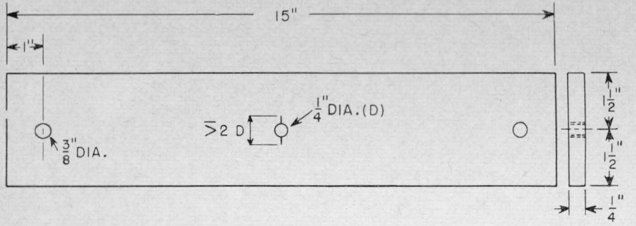

FIGURE 1. *Schematic diagram of crack propagation specimen.*

Small variations in overall dimensions and position of loading pin holes do not significantly affect fracture characteristics.

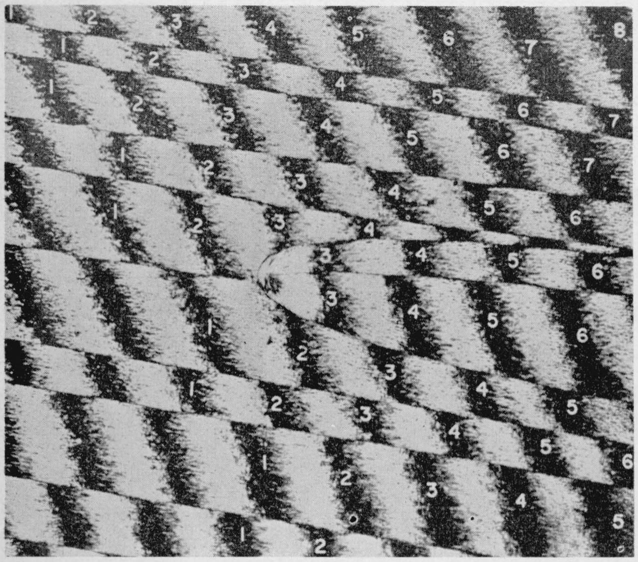

FIGURE 2. *Multiple-beam interferogram of parabola-bearing portion of a polymethyl methacrylate crack propagation specimen fracture surface.*

Numbers identify components of the same fringe. Source was the 5461 Å mercury line.

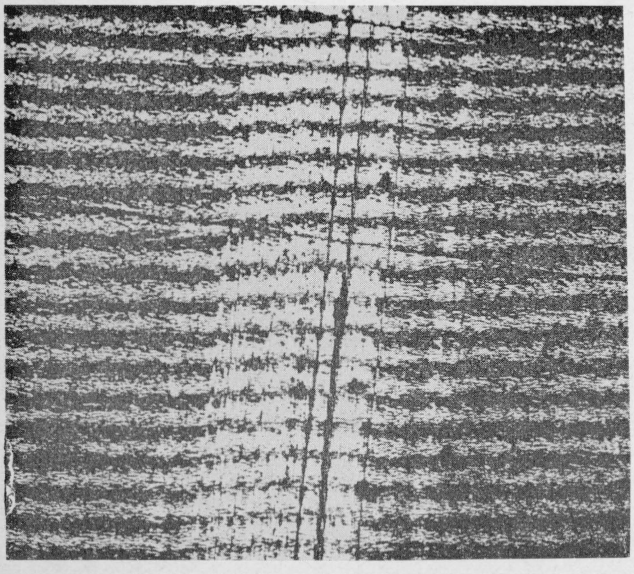

FIGURE 3. *Multiple-beam interferogram of the swabbed area of the fracture surface of a crack propagation specimen of polymethyl methacrylate.*

Source was the 5461 Å mercury line.

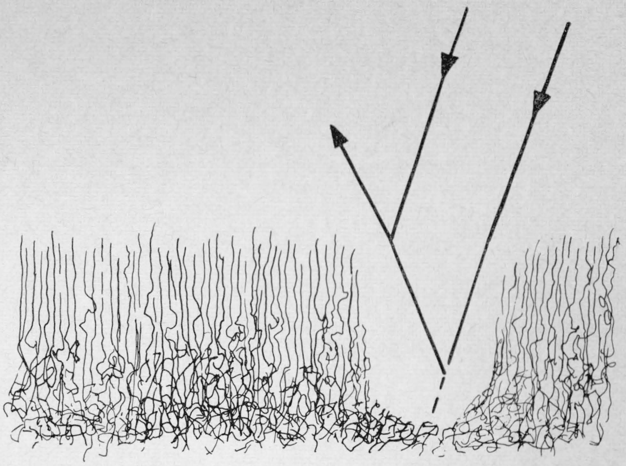

FIGURE 4. *Diagrammatic representation of interference in the oriented film of the fracture of polymethyl methacraylate.*

(Modified from Higuchi [6])

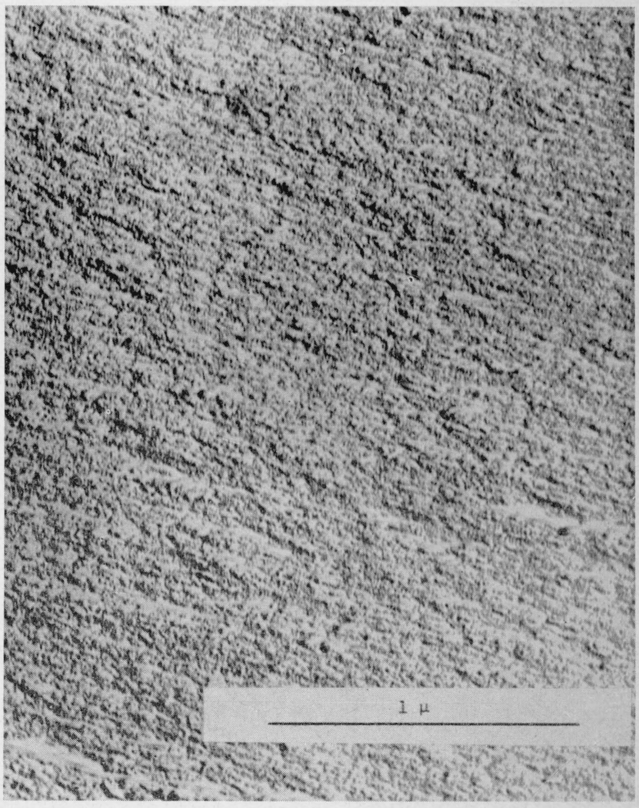

FIGURE 5. *Electron micrograph of a replica of the fracture surface of a crack propagation specimen of polymethly methacrylate.*

The area shown is from the slow-growth region.

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Figure 6. *Electron micrograph of a replica of the fracture surface of a crack propagation specimen of polymethyl methacrylate.*

The area shown is from the crowded parabola region.

Figure 7. *Electron micrograph of a replica of a fracture surface of polymethyl methacrylate broken by rapid overstress.*

The surface is covered by droplets derived from the surface film.

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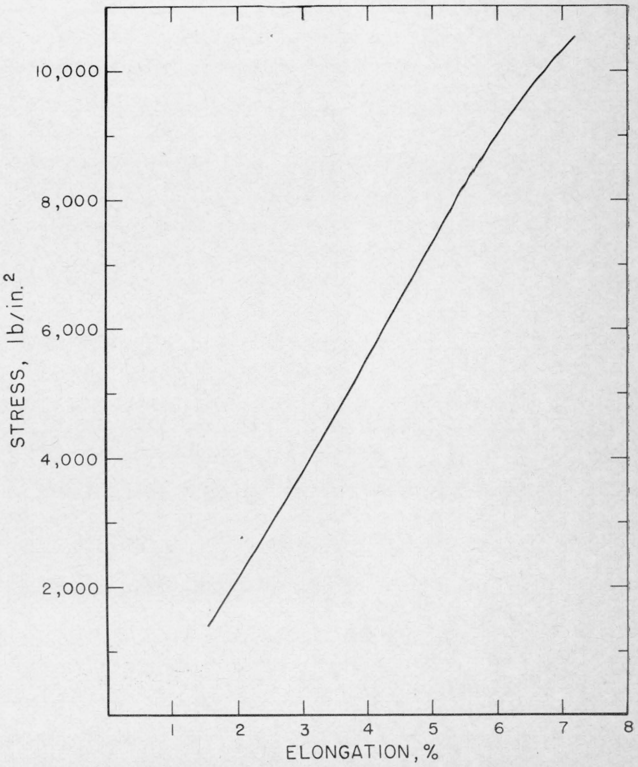

Figure 8. *Stress-elongation characteristics of acrylonitrile-styrene copolymer.*

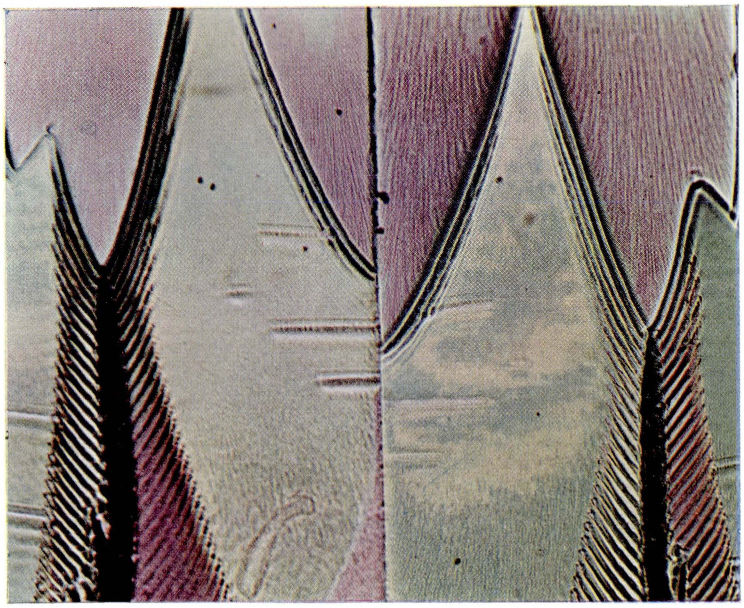

FIGURE 9a. *Single colored area in the slow-growth region of a fracture produced in a crack propagation specimen of polymethyl methacrylate.*

The matching faces are shown side by side.

FIGURE 9b. *Multicolored area in the slow-growth region of a fracture produced in a crack propagation specimen of polymethyl methacrylate.*

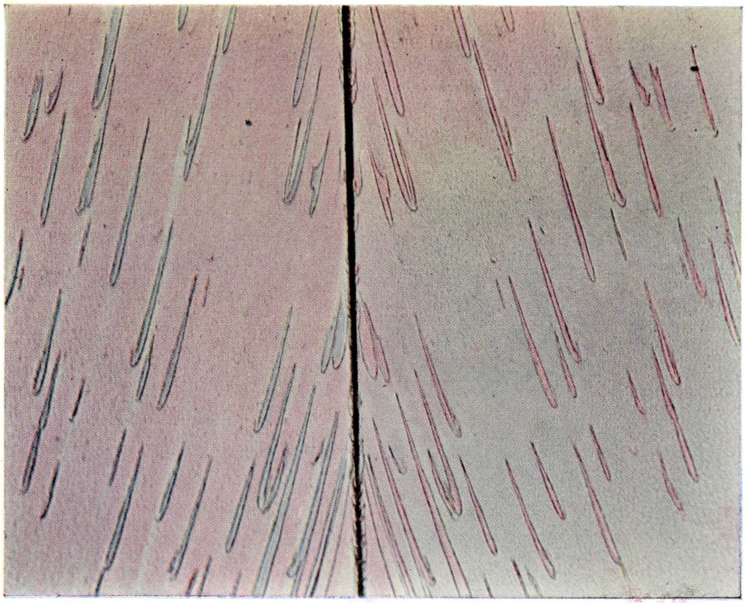

FIGURE 9c. *Area of irregularly shaped elongated figures on a fracture surface produced in a crack propagation specimen of polymethyl methacrylate.*

The matching faces are shown side by side.

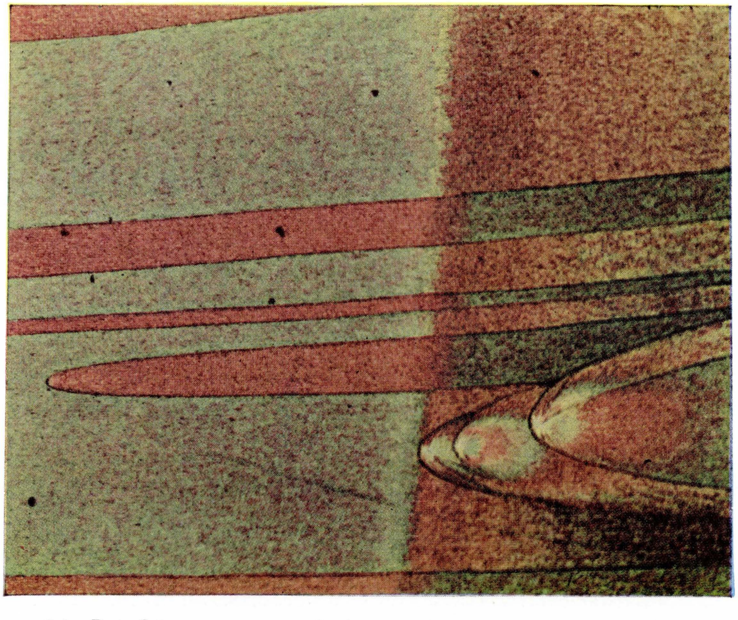

FIGURE 9d. *Detail from an area on the fracture face of a crack propagation specimen of polymethyl methacrylate in which the elongated figures merge into the parabola-containing region.*

Note the same structures bearing complementary colors on opposite sides of the transverse color interface.

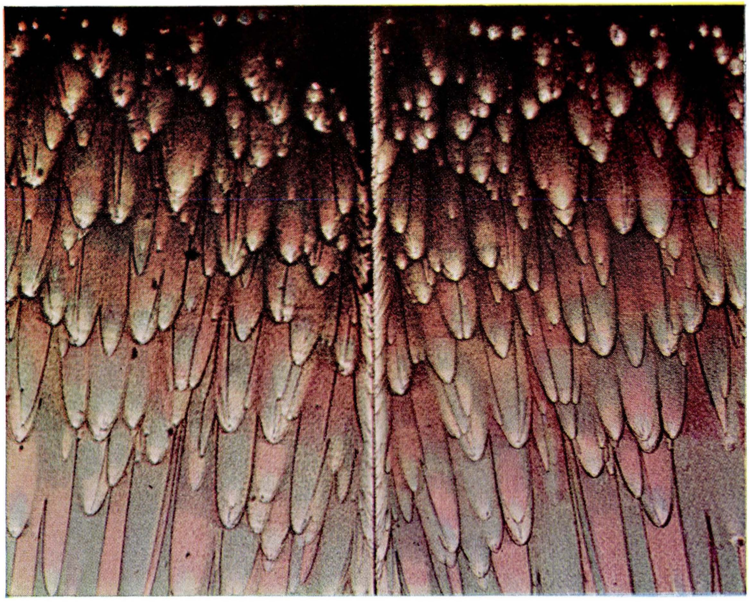

Figure 9c. *Area of crowded parabolas on a fracture surface produced in a crack propagation specimen of polymethyl methacrylate.*

The matching faces are side by side.

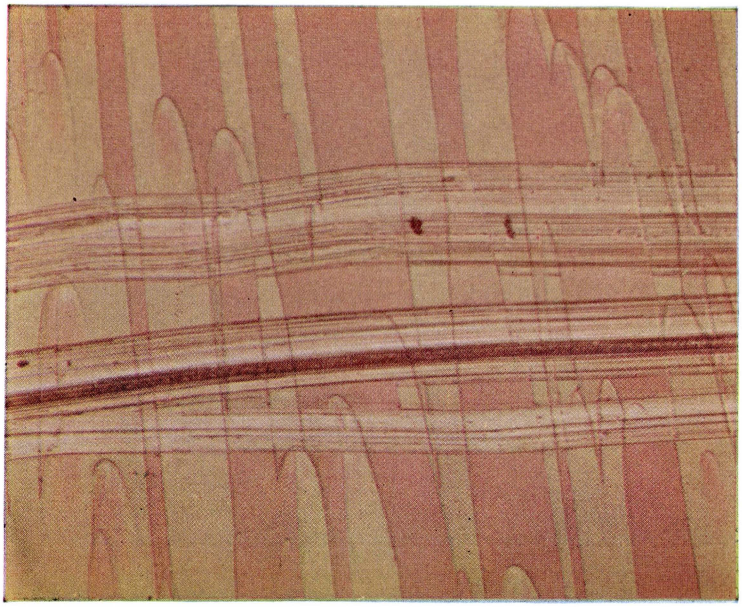

Figure 10. *Area of the parabola-bearing region on the surface of a fracture produced in a crack propagation specimen of polymethyl methacrylate.*

A soft swab has produced swaths through the surface film. Note the loss of color in the swaths which have not disturbed the geometric structures.

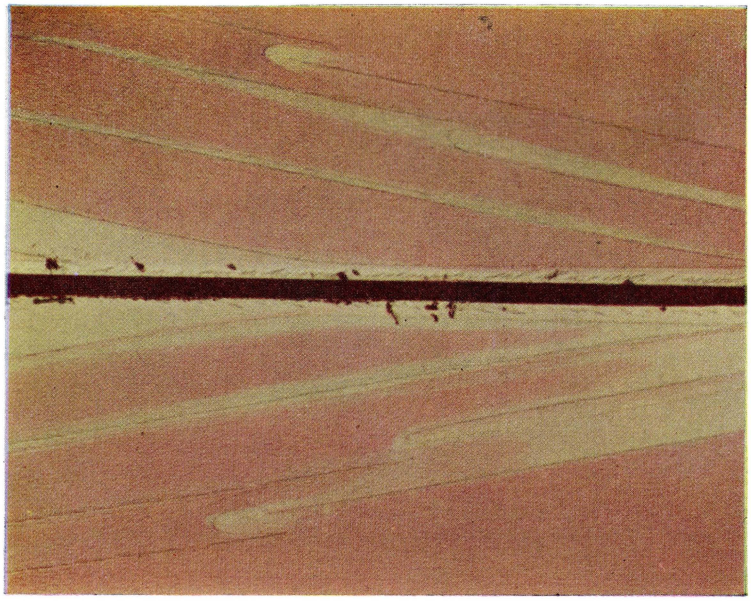

FIGURE 11. *Part of the surface of a fracture produced in a crack propagation specimen of polymethyl methacrylate.*

The surface shows the effect of exposure to vapors of methyl chloride (upper half). The lower section is the untreated matching surface.

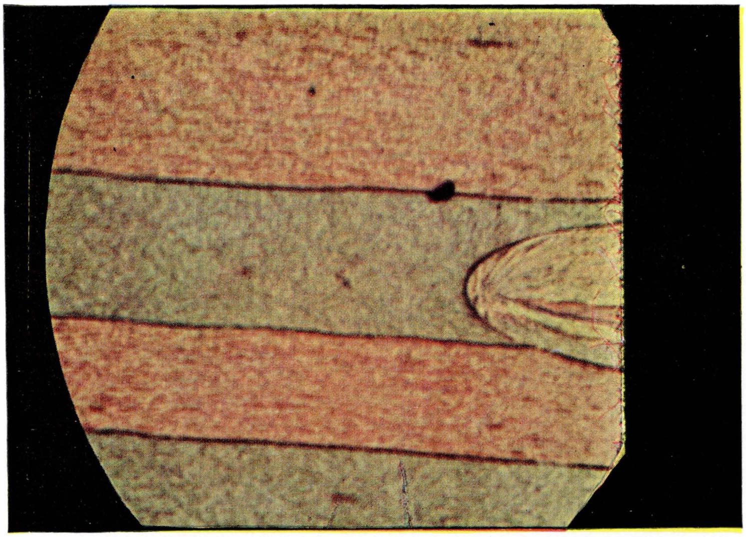

FIGURE 12. *Fracture surface colors produced by thin-film interference.*

A reflecting layer of silver was deposited on the fracture surface *in vacuo* and subsequently a thin solution of Canada balsam was deposited over the silver.

FIGURE 13. *Thin-film interference on the crowded parabola portion of the surface of a fracture produced in a crack propagation specimen of polymethyl methacrylate.*

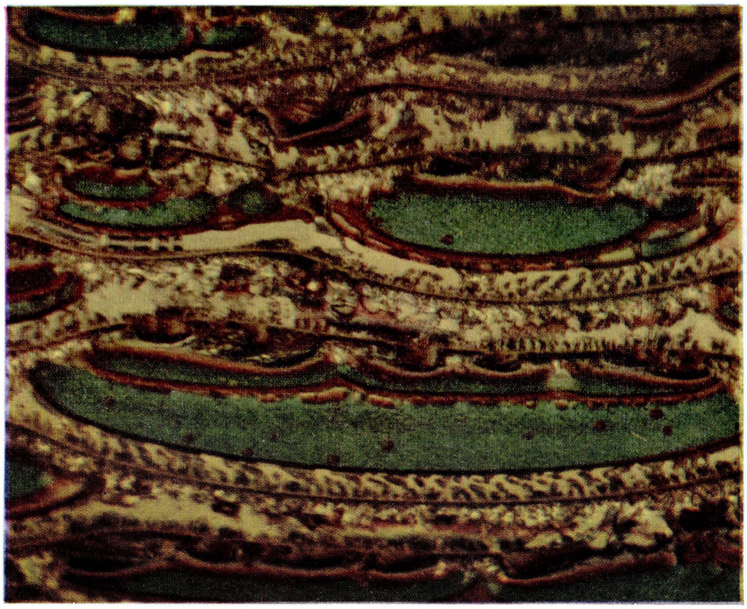

FIGURE 14. *Fracture surface of a tensile specimen of molded polystyrene.*

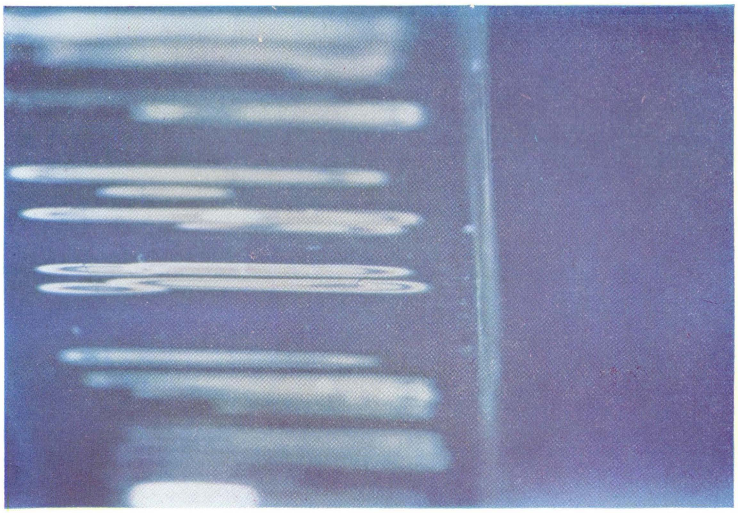

FIGURE 15. *Large internal craze cracks in a tensile specimen of molded acrylonitrile-styrene copolymer.*

The specimen was loaded to failure through a crosshead moving at 0.02 in./min.

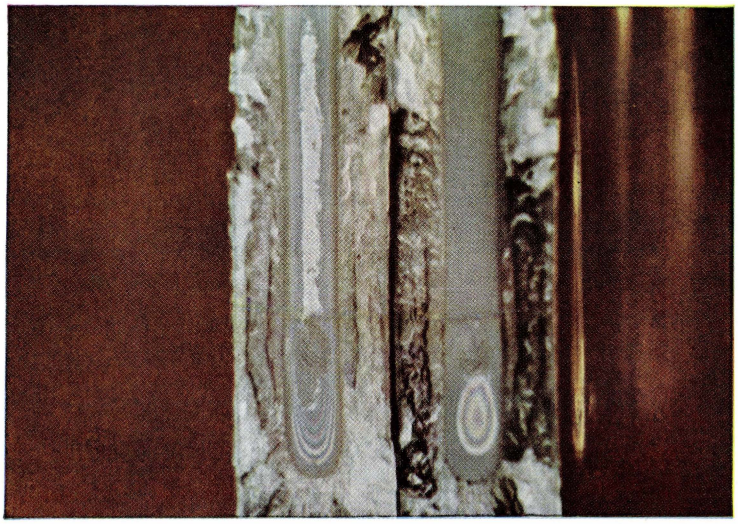

FIGURE 16. *Matching fracture faces of a tensile specimen of acrylonitrile-styrene copolymer.*

The specimen was loaded to failure through a crosshead moving at 0.02 in./min.

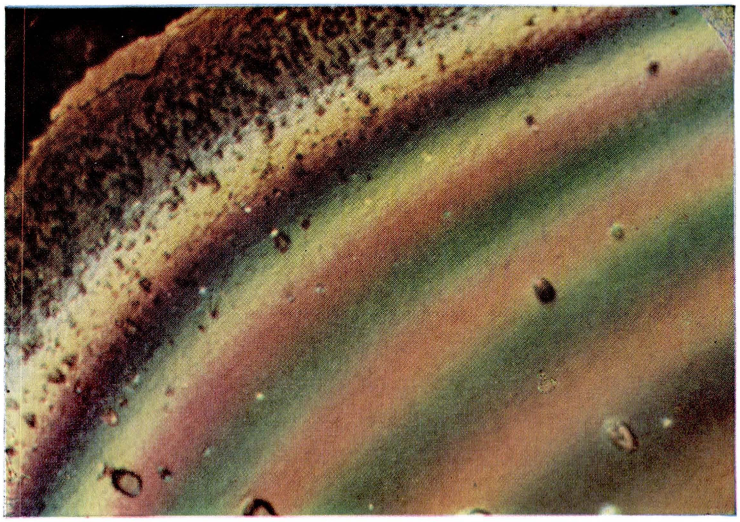

FIGURE 17. *Detail from a fractured craze crack of acrylonitrile-styrene copolymer.* The periphery of the crack is in the upper left-hand corner. The film displays fringes of at least four orders.

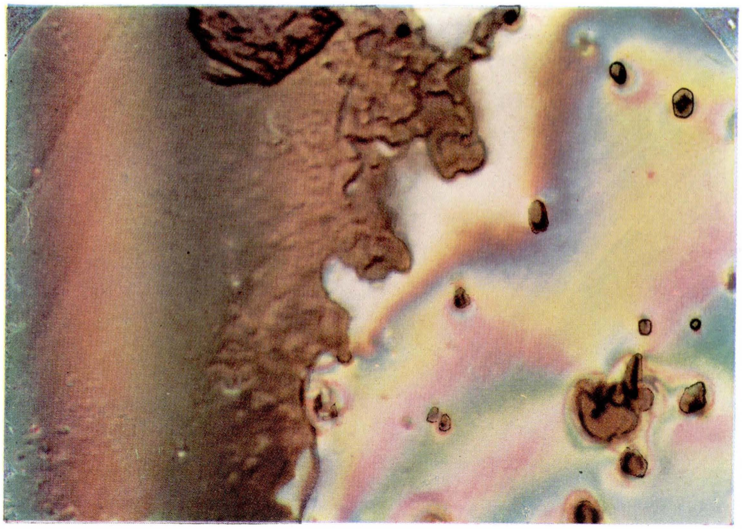

FIGURE 18. *Detail from a fractured craze on acrylonitrile-styrene copolymer.* The area bearing fringes of equal thickness is at the left. The bright fringes cover a portion of the film which is slightly wrinkled and also stretched in the vicinity of the holes.

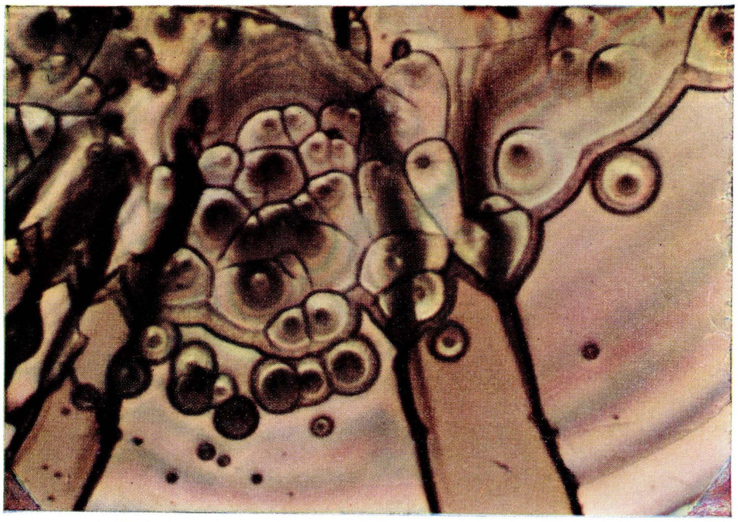

FIGURE 19. *Detail from the center of a fractured craze crack of acrylonitrile-styrene copolymer.*

Conic sections characteristic of brittle failure are associated with the surface film.

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Publications of the National Bureau of Standards*

Selected Abstracts

On the index of refraction of air, the absorption and dispersion of centimeter waves by gases, G. Boudouris. *Translated from French by G. Wm. Curtis, J. Res. NBS 67D (Radio Prop.), No. 6, (Nov.–Dec. 1963).*

The index of refraction, the absorption and dispersion are studied for several gases and vapors (pressure from 0 to 1 atm, temperature from 0 to 50° C, frequency from 7,000 to 12,000 Mc/s). The first part is devoted to a description of the microwave spectrometer used, while the second part is a survey of the several theories implied in the interpretation of the results.

In the third part, we present first the results concerning the index of refraction of atmospheric gases, of dry air, and of damp air. Then we study the absorption and dispersion by several vapors, and of ammonia and chloroform, principally. Finally, we indicate a new method making use of gaseous mixtures.

Our original results are discussed and presented within the frame of reference of works of other authors by means of comparison and intensive bibliographies.

Tensile and impact properties of selected materials from 20 to 300°K, K. A. Warren and R. P. Reed, *NBS Mono. 63 (June 28, 1963), 35 cents.*

The tensile and impact properties of structural materials were experimentally determined at temperatures from 20 to 300 °K. Tensile properties of a few materials were also determined at 4 °K. The materials included forty-two commercial alloys of iron, aluminum, titanium, copper, nickel, and cobalt, and two metal-bonded carbides. The properties experimentally determined were the yield strength, tensile strength, elongation, and reduction of area, the stress versus strain curve, and the impact energy. The test equipment and procedures are described. The individual data are presented in tables, and the average results are displayed in graphs.

Refractive indices and densities of aqueous solutions of invert sugar, C. F. Snyder and A. T. Hattenburg, *NBS Mono. 64 (June 7, 1963), 15 cents.*

The refractive indices and densities of aqueous solutions of invert sugar have been determined, at temperatures of 15, 20, 25, and 30 °C, for concentrations up to about 82 percent of invert sugar (by weight). From the results, equations have been derived which relate the refractive index and percent of invert sugar (weight in air) at each temperature. Other equations relate the absolute density and percent of invert sugar (weight in vacuum) at each temperature. Five-decimal tables giving the refractive indices and densities of invert sugar solutions containing 1 to 85 percent of sugar are given for each percentage of invert sugar at the four temperatures.

Reduction of data for piston gage pressure measurement, J. L. Cross, *NBS Mono. 65 (June 17, 1963), 15 cents.*

Pressure measurements made with piston gages are affected by gravity, temperature, pressure, and several other variables. For accurate determinations of pressure the calculations must take these variables into account. A general equation is developed and simplified procedures for calculating pressure are illustrated.

Tabulation of data on receiving tubes, C. P. Marsden and J. K. Moffitt, *NBS Handb. 83 (May 23, 1963), $1.25. (Supersedes Handb. 68.)*

A tabulation of Receiving-Type Electron Tubes with some characteristics of each type has been prepared in the form of two major listings, a Numerical Listing in which the tubes are arranged by type number, and a Characteristic Listing in which the tubes are arranged by tube type and further ordered on the basis of one or two important parameters. The tabulation is accompanied by a listing of similar tube types and basing connections for the listed tubes.

Impedance of commercial Leclanché dry cells and batteries, R. J. Brodd and H. J. Dewane, *NBS Tech, Note 190 (July 5, 1963), 40 cents.*

An extensive study of the impedance characteristics of the most commonly used sizes of commercial Leclanché type dry cells and batteries has been made through the frequency range of 50 to 50,000 cycles. Changes in impedance due to aging and use were determined. Open-circuit voltages and flash currents were measured, and capacities determined on standard tests in an effort to find a possible correlation between any of those three parameters and residual capacity. Data obtained are displayed in tabular form and on Argand diagrams.

Tables describing small-sample properties of the mean, median, standard deviation, and other statistics in sampling from various distributions, C. Eisenhart, L. S. Deming, and C. S. Martin, *NBS Tech. Note 191 (June 14, 1963), 20 cents.*

This note includes a collection of tables useful for study of the sampling distributions of some frequently-used statistics, with brief discussions of their construction and use. (1) The probability level $P(\epsilon,n)$ of any continuous parent distribution corresponding to level ϵ of the distribution of the median. (2) Probability points of certain sample statistics for samples from six distributions: normal and double-exponential (mean, median), rectangular (mean, median, midrange), Cauchy, Sech, Sech ^{2} (median). In all the above tables, the sample size $n=3(2)15(10)95$ and the probability levels are $\epsilon=.001$, .005, .01, .025, .05, .10, .20, .25. Together with the tables listed under (2) are given the values of certain ratios useful for comparing the various statistics. (3) Probability that the standard deviation of a normal distribution will be underestimated by the sample standard deviation s and by unbiased estimators of σ based on s, on the mean deviation, and on the sample range. Divisors are given for obtaining the corresponding "median unbiased" estimators.

Calculations of the potential and effective diffusion constant in a polyelectrolyte solution, S. R. Coriell and J. L. Jackson, *NBS Tech. Note 192 (June 28, 1963), 25 cents.*

The results of numerical computations of the electrostatic potential and the effective diffusion constant of counterions in a polyelectrolyte solution are given. The potentials for various polyion charge densities and polyion sizes are presented graphically. The calculated diffusion constants are compared with experimental data on the diffusion of labeled sodium ions in polyacrylis acid-sodium hydroxide solutions.

National standard reference data program, background information, *NBS Tech. Note 194 (June 1963), 25 cents.*

Plans are proposed for a National Standard Reference Data System that will provide critically evaluated data in the physical sciences on a national basis. It will be conducted as a decentralized operation across the country, with central coordination and administration by NBS. Data will be centrally stored at NBS and disseminated through a series of services tailored to user needs in science and industry.

Microwave spectrum of tertiary butyl chloride, A. Comparison of tertiary butyl structures, D. R. Lide, Jr., and M. Jen, *J. Chem. Phys.* **38,** *No. 7, 1504–1507 (Apr. 1, 1963).*

The rotational constants of $(C^{12}H_3)_3C^{12}Cl^{35}$, $(C^{12}H_3)_3C^{12}Cl^{37}$,

$C^{12}H_3)_3C^{13}Cl^{35}$, $(C^{12}H_3)_3C^{13}Cl^{37}$, and $(C^{13}H_3)(C^{12}H_3)_2(C^{12}Cl^{35})$ have been measured. The r_s structure of the *t*-butyl chloride skeleton calculated from these constants is: $r(CCl) = 1.803 \pm 0.002$ A, $r(CC) = 1.530 \pm 0.2002$ A, $\angle CCC = 110.9° \pm 0.1°$. The quadrupole coupling constant for Cl^{35} is -66.9 ± 1.5 Mc. The structures of a number of *t*-butyl compounds are compared. The *t*-butyl group is practically unaffected by substitution, while the CX bond in $(CH_3)_3CX$ is systematically longer than in CH_3X molecules. Conventional concepts of valence theory do not provide a satisfying explanation for this behavior.

Calorimetric calibration of the electrical energy measurement in an exploding wire experiment, D. H. Tsai and J. H. Park, *Exploding Wires* **2,** *27–107 (Plenum Press, Inc., New York, N.Y., 1962).*
A discussion is presented on the requirements and methods for measuring the current and voltage during the transient discharge of a capacitor bank being employed in an exploding wire experiment. A method is described for accurately calibrating the measured current, voltage, and electrical energy by comparing the calorimetric heating of a resistance element of essentially constant resistance with the electrical energy dissipated in the element. Results show that the accuracy of the energy measurement is about 1–2%.

Quasi-equilibrium theory of mass spectra, H. M. Rosenstock and M. Krauss, *Book, Mass Spectrometry of Organic Ions, pp. 1–64 (Academic Press, Inc., New York, N.Y., 1963).*
The essential features of the ionization and dissociation of diatomic molecules by electron impact are well understood. The process of ionization produces a vertical or Franck-Condon transition to one or another of the potential curves of the ion. Fragmentation occurs in that fraction of the ions initially formed above the dissociation limits of the potential curve.

Electrode potentials in fused systems. VI. Membrane potentials, K. H. Stern, *Phys. Chem.* **67,** *893–895 (1963).*
Membrane potentials have been measured for the cells Ag AgCl(X_1), NaCl$(1-X_1)$ vycor glass AgCl(X_2), NaCl$(1-X_2)$Ag. In contrast to the simple liquid junction whose potential is zero in this system (the transport number equals the mole fraction), most of the current is carried through the glass by the sodium ion and $t_{Na^+} = 0.95$, independent of melt composition. Cells in which $X_1 \cong 1$, i.e. reagent grade AgCl, also operate as concentration cells with a Na^+ impurity $(X_{Na^+} \cong 2 \times 10^{-4})$. The use of these cells for reference electrodes is discussed.

Electron spin resonance spectra of aged, γ-irradiated polystyrenes, R. E. Florin, L. A. Wall, and D. W. Brown, *J. Polymer Sci. Pt. A.* **1,** *1521–1529 (1963).*
The electron spin resonance spectrum of polystyrene which has been γ-irradiated at $-196°C$ changes gradually from a broad three-peaked structure with outer derivative peaks at ± 46 gauss to a much narrower structure with poorly separated outer derivative peake at ± 21 to 24 gauss. The deuterated polystyrenes behave likewise in general. The aged radicals of α,β,β-trideuterostyrene and β,β dideuterostyrene exhibit only a single peak with derivative maxima at $\pm$ 7 or 8 gauss from center. The aged spectra are all consistent with the hypothesis of major hyperfine interaction with β hydrogens. During the aging about 80% of the original radicals are lost, but the remainder are very long-lived. Poorly evacuated samples or those with added benzene decay much more rapidly.

Numerical computation of time-dependent properties of isotopically disordered one-dimensional harmonic crystal lattices, R. J. Rubin, *J. Phys. Soc. Japan* **18,** *Suppl. II, 63–69 (1963).*
The principal purpose of this work is to investigate numerically the statistical dynamical properties of isotopically disordered harmonic crystal lattices. Two properties are studied in detail: 1) The decay of initial periodic disturbances $\dot{Q}_k(\tau)$ which are normal modes of the same lattice structure when all particles have the same mass; and 2) the dipole moment correlation function $\langle \dot{M}(o)\ M(\tau) \rangle$. The numerical calculation of the time evolution of either $\dot{Q}_k(t)$ or $\langle \dot{M}(o) M(\tau) \rangle$ involves the solution of a single initial value problem for a given random distribution of the isotope masses on the lattice sites. The time evolution is investigated for values of the isotope mass ratio m_1/m_2 and of K for which the perturbation results of Maradudin, Weiss, and Jepsen are not applicable. A new formulation of the formal solution of the normal mode decay problem is proposed. This formulation should make it possible to calculate the initial stages of decay in an infinite lattice for the extreme values of m_1/m_2 and K studied in this paper.

Symmetry conditions for internal friction caused by jumping of point defects in crystals, J. B. Wachtman, Jr., and H. S. Peiser, *J. Appl. Phys. Letter* **1,** *No. 1, 20–22 (Sept. 1962).*
Symmetry conditions for the existence of internal friction are given which require only a knowledge of the behavior of a defect-free crystal under strain and a knowledge of which sites the defect occupies. These conditions predict that in rutile under strain ϵ_{xx} oxygen vacancies should not and titanium interstitials should produce internal friction. An observed internal friction peak suggests the existence of titanium interstitials in lightly reduced rutile.

Measurement of the lattice constants of neon isotopes in the temperature range 4°–24°K, L. H. Bolz and F. A. Mauer, *Proc. 11th Annual Conf. Applications of X-ray Analysis, Denver Res. Inst. (Plenum Press, Inc., New York, N.Y., 1962).*
Using an X-ray diffractometer cryostat, the lattice constants of the isotopes ^{20}Ne and ^{22}Ne as well as the naturally occurring mixture have been measured throughout most of the temperature range in which they exist as solids (0° to approximately 24 °K). The heavier isotope has the smaller lattice constant, the values obtained at 4.2 °K being: ^{20}Ne—4.462_4 A, $^{22}Ne - 4.454_0$ A, and natural neon-4.462_2 A. The absolute error in these values is believed to be no greater than 0.001 A. The volume expansion coefficient, which does not appear to differ significantly in the three cases, increases to a value of $6.3 \times 10^{-3}/°K$ at 24 °K.

Design of an interferometric oil manometer for vacuum measurement, A. M. Thomas, D. P. Johnson, and J. W. Little, *1962 Trans. Ninth Natl. Vacuum Symp., Am. Vacuum Soc., pp. 468–473 (1962).*
The oil manometer under discussion is one which uses an interferometer to measure the changes in height of the oil surfaces as a change in pressure is applied. Interference fringes are developed between the lower surface of an optical flat and the oil surfaces of the manometer. Di-octyl sebacate is distilled into the manometer after outgassing the system and oil. A fringe shift of fifty-five fringes is produced by a pressure of 10^{-3} torr. A sensitivity of 2×10^{-6} torr can be attained by detecting a shift of one-tenth of a fringe.

Analysis of the third spectrum of praseodymium, J. Sugar, *J. Opt. Soc. Am.* **53,** *No. 7, 831–838 (July 1963).*
The spectrum of doubly-ionized praseodymium was recorded and analyzed, with the result that 118 energy levels of odd parity and 130 levels of even parity were derived. These include nearly all the levels of the $4f^3$, $4f^25d$, $4f^26s$, $4f^26p$ configurations as well as some low levels of the $4f5d^2$ and $4f^26d$ configurations. Electrostatic and spin-orbit parameters were determined for the $4f^26s$ configuration. From the *hfs* of the $4f^26s$ levels, a magnetic dipole interaction constant of 0.46 cm^{-1} was obtained. An ionization potential of 23.2 volts was calculated from levels of the $4f^25d$ and $4f^26d$ series.

Photodisintegration of light nuclei, E. Hayward, *Rev. Mod. Phys.* **35,** *No. 2, 324–331 (Apr. 1963).*
The data from some recent experiments on the photoeffect in light nuclei (Z= 6–20) are summarized. It is shown that the so-called giant resonance for these nuclei really consists of the superposition of many resonances, more than is consistent with a simple shell-model. The absorption cross section integrated over the giant resonance varies from 50 to 90 percent of the classical dipole sum, a large fraction of the nuclear oscillator strength being associated with the high energy quasideuteron effect.

Very low temperature data. Properties of paramagnetic salts, R. P. Hudson, *AIP Handb. 2d Ed. Sec. 5g, pp. 222–223 (1963).*
A revision of Section 4b of the American Institute of Physics Handbook, on the properties of paramagnetic salts at very low temperatures, has been made. The original discussion has been completely rewritten and brought up to date, and extended to other substances which have recently acquired importance.

Collective correlation of plasma, C. M. Tchen, *Czechoslovak J. Phys., Proc. 2d Conf. Electronics* **XXI,** *No. 5–6, 516–521 (Apr. 1962).*
The one-particle distribution function is described by the BBGKY equation, while the correlation function is determined by an equation of the Vlasov type (with self-consistent field, but without collision). Oscillations of small amplitudes are considered. The dispersion relation is obtained, and the Landau damping is calculated.

Pressure broadening as a prototype of relaxation, U. Fano, *Phys. Rev.* **131,** *No. 1, 259–268 (July 1, 1963).*
The theoretical results of Baranger, Kolb and Griem on pressure broadening are rederived by a more compact and flexible procedure directly applicable to other relaxation processes. Pressure broadening is worked out to first order in the pressure, including previously disregarded corrections. The procedure adapts the concepts and techniques of scattering theory to the Louville representation of density matrices. Its key quantity is a frequency dependent relaxation operator $<M_c(\omega)>$ introduced by Zwanzig.

The atomic spectra of the rare earths: their presence in the sun, C. E. Moore, *Appl. Opt.* **2,** *No. 7, 665–674 (July 1963).*
A summary of the present state of analysis of the first and second spectra of the rare-earth elements is presented with an extensive bibliography. The astrophysical importance of these spectra is stressed. Counts are given of the number of lines of each rare-earth spectrum identified at present in the accessible solar spectrum. Most of them appear only in the singly-ionized state; exceptions are Eu I, Tm I?, and Yb I. The possible presence of the strongest laboratory line of Co III in the solar spectrum is suggested. If correct, this is the first evidence of a third spectrum line in the accessible range of the solar spectrum ($\lambda > 3000$ Å).

The conductive-disk method of measuring the thermal conductivity of insulations, H. E. Robinson, *Suppl., Bull. Intern. Inst. Refrigeration, Annexe 1962–1, 43–50 (Aug. 1962).*
A comparative method of a new type for determining the thermal conductivity of isotropic insulations is described. It employs a circular disk of a material of suitable conductivity and thickness, which is sandwiched between two flat specimens of the material to be measured, the outer faces of which are put in contact with uniform-temperature cooling plates. The rim of the disk is heated uniformly, and as a result of heat flow from the disk through the specimens to the cooling plates, the steady-state temperature of the disk decreases toward its center. Measurements are made of the surface temperature of the disk at its center, and at a suitable radius, and also of that of the cooling plates. The conductivity of the specimen is determined from these data, together with the dimensions of the system and the conductivity of the disk material. If the conductivity of the disk material is not known, a calibration curve for the disk can easily be developed by means of a few tests made on specimens of known conductivity.

Electron affinity of atomic iodine, B. Steiner, M. L. Seman, and L. M. Branscomb, *J. Chem. Phys.* **37,** *No. 6, 1200–1202 (June 1962).*
Photodetachment of I^- ions has been observed in a crossed beam experiment. The apparatus used was essentially similar to that in previous photodetachment experiments. Use of four Sharp Cut filters provided good "effective" resolution close to threshold but did not permit an independent determination of the behavior of the cross section as a function of wavelength. A step function cross section, shown by Berry and co-workers to approximate closely the true cross section behavior, was used to provide an upper energy limit to the electron affinity shown to be close to the actual value. The value of the electron affinity thus determined, $3.076 \pm .005$ ev, is in excellent agreement with the shock wave determination of Berry, $3.074 \pm .003$. The absolute magnitude of the cross section in the region 0.3 ev above threshold was determined to be $1.5 \pm 0.8 \times 10^{-17}$ cm^2.

Field-emission study of carbon monoxide on tantalum, R. Klein and L. B. Leder, *J. Chem. Phys.* **38,** *No. 8, 1866–1872 (Apr. 15, 1963).*
Adsorbed carbon monoxide shows three states of binding on tantalum. The weakest of these is desorbed at temperatures above 125°K with no observable surface migration. The bonding with the surface is of the Van der Waal's type. This is in contrast with the second state, which is chemisorbed above 650°K. The third state is dissociated before desorption to give the oxygen on tantalum pattern. Surface migration involving the second adsorbed state occurs with an activation energy of 38 k cal. The work function of a carbon monoxide covered tantalum surface, obtained by spreading a CO shadowed tip at 40°K, is 0.8 ev greater than that of the corresponding clean tantalum. Fowler-Nordheim plots of clean and shadowed tips show that at least under these conditions the infinite field extrapolation of $\ln i/V^2$ is proportional to the emitting areas.

Field emission observations of carbon on tantalum, R. Klein and L. Leder, *J. Chem. Phys.* **38,** *No. 8, 1863–1866 (Apr. 1963).*
The carbon-tantalum system, observed with field emission, shows the temperature effects on the solubility and precipitation of the carbide phase. Carbon, once deposited on tantalum, cannot be removed by high temperature treatment, as in the case of tungsten. With low carbon contamination the 334 planes appear as dark areas, just as for carbon on tungsten. At temperatures in the region of 950°K, platelets, presumably Ta_2C, form and migrate to the [111] zones of the emitter single crystal tip. The energy of activation of this surface migration process is 54 k cal.

Electron energy levels and their relationship to lattice defects in reduced rutile, J. H. Becker and W. R. Hosler (*Proc. Intern. Conf. Crystal Lattice Defects*), *J. Phys. Soc. Japan* **18,** *Suppl. II, 152–160 (1963).*
Electrical conduction mechanisms and defect structure are studied by measurements of the Hall coefficient and electrical resistivity from 300°K to about 2°K, for samples of reduced rutile with $\rho \| c$ (300°K) ~ 1 to 5 ohm-cm. Virtually identical results are obtained for samples reduced in vacuum at 1000°C or hydrogen at 600°C. The anisotropic nature of the Hall coefficient and resistivity tensora (with two independent elements in each for this tetragonal system) is taken into account. The data are suggestive of conduction in impurity levels at very low temperatures ($< \sim 4$°K), a narrow Ti^{3+} conduction band (No. 1) at low temperatures ($\sim 7-30$°K) and additional contributions at intermediate temperatures ($\sim 30° - 300$°K) from another Ti^{3+} conduction band (No. 2). Recent evidence concerning the defects in reduced rutile is discussed; it appears that most of the data are more consistent with a model of Ti^{3+} ions at interstitial positions which act as donors than with a model of oxygen vacancies, as concluded previously by several authors.

New autoionizing atomic energy levels in He, Ne, and A, R. P. Madden and K. Codling, *Phys. Rev. Letters* **10,** *No. 12. 516–518 (June 15, 1963).*
The NBS 180 MeV electron synchrotron has been used as a continuum light source for absorption spectroscopy in the λ180–470Å region. Two-electron transitions to states which autoionize have been observed in He; transitions to autoionizing states have also been observed in Ne and A.

A modification of the Born-Mayer potential function as applied to the crystalline alkali halides, T. B. Douglas, *J. Chem. Phys.* **38,** *No. 10, 2461–2466 (May 15, 1963).*

In an attempt to improve the applicability of the Born-Mayer equation to more extreme conditions than those in the pure alkali halides, the repulsion constant ρ is assigned values different for like and unlike ion pairs in such a way that large deviations from the observed compressibilities of the lithium halides attributable to marked anion-anion repulsion are removed. It is then found that the "basic radii" can be assigned values which give lattice parameters in excellent agreement with the experimental ones, and that the calculated electron affinities still agree approximately with the most precise directly observed values. A general condition imposed by the Born-Mayer equation is pointed out which is not satisfied by the data for several of the alkali halides.

Microbalance techniques for high temperature applications—further developments, N. J. Carrera and R. F. Walker, *Book, Vacuum Microbalance Techniques, 3, 153–177 (Plenum Press, Inc., New York, N.Y., 1963).*

A description is given of recent improvements to and plans for a microbalance technique for studying the rates of vaporization of refractory substances, generally in the temperature range of 1600–2500° C. An ion pumping system is described which permits the attainment of 1×10^{-9} torr without bakeout. The apparatus, apart from the pump, can be completely enclosed in a bakeout oven to attain still lower residual pressures. The design of a variable-capacitance recording system for the Gulbransen-type microbalance has commenced, and preliminary features of this system are presented. It has been found possible to include a black body hole in the small samples suspended from the microbalance, thus improving the precision of temperature measurements. Some preliminary work on the application of reimaging techniques for the heating of samples is also described.

High resolution spectra in the region from 2 to 6μ, E. K. Plyler and E. D. Tidwell (*Proc. Intern. Molecular Conf.*), *Book, Advances in Molecular Spectroscopy, pp. 1336–1342 (Pergamon Press Inc., New York, N.Y., 1962).*

A grating spectrometer has been built which has hlgh resolution in the region from 2 to 6μ. Three gratings are used to cover the region and they have 7500. 10,000, and 15,000 lines/in. and their ruled area is about 5×8 in. The instrument can be used single pass or double pass. Under the most favorable conditions partial resolution is obtained of lines separated by 0.025 cm^{-1}.

The absorption spectra of several polyatomic molecules have been measured and molecular constants calculated. This report discusses the spectra of the $2\nu_4$ bands of fluoroform and chloroform. Molecular constants of fluoroform have been calculated from the spectra and have the following values, $\nu_0=2710.25_5$ cm^{-1}, $B''=.34521_5$, $\mathrm{B}'=.34452_6$, $D=3.97_5\times10^{-7}$

Theory of the electronic polarizabilities of ions in crystals: Application to the alkali halide crystals, A. R. Ruffa, *Phys. Rev.* **130,** *No. 4, 1412–1423* (*May 15, 1963*).

A theoretical estimate of the polarizabilities of ions in the alkali halides is made by means of a procedure based upon the product approximation which distinguishes the ionic constituents in the crystal but allows for their overlap and mutual interaction. Analysis by means of the Thomas-Kuhn sum rule is used allowing the sum representing the ionic polarizability to be represented by one effective parameter, which is calculated for the free ions from the Pauling theoretical values. The change in this parameter when the ion is transported into the crystalline environment is estimated by an energy level analysis used previously by Seitz.

It is shown that this procedure accounts for most of the quantitative features of the polarizabilities of the alkali halides. Moreover, the implications of these results are strongly at variance with the usual view concerning the polarizabilities of ionic crystals, since the additivity rule appears as an accidental result of the qualitative similarily of the interactions in many crystals, and the possibility of large fluctuations in the polarizability of a given ion in different crystalline environments is made evident.

Atomic flame reactions involving N-atoms, H-atoms and ozone, D. Garvin and H. P. Broida, *9th Intern. Combustion Symp., pp. 678–688 (1963).*

The low pressure reaction between H, N and O_3 has been studied by emission and absorption spectroscopy. When all three of these reactants are present together two types of flames are formed. One of these, a pink brush-like reaction zone represents a low temperature regime, while the other, a well formed white flame is probably a high temperature phenomenon. Electronic spectra in emission of OH, NH, NO and N_2 (1st positive) are found in the former. The latter has the same bands plus emission from NH_2 and the OH vibration-rotation bands.

The reaction mechanism is discussed. It is shown that H-atoms serve as a catalyst and suggested that vibrationally excited OH is responsible for the formation of NH and excited nitrogen molecules ($B^3\pi$)

Infrared spectrum of acetylene-d$_1$, W. J. Lafferty, E. K. Plyler, and E. D. Tidwell, *J. Chem. Phys.* **37,** *No. 9, 1981–1988 (Nov. 1962).*

The high resolution absorption spectrum of C_2HD has been studied in the 1900 to 3400 cm^{-1} region. Several bands at lower frequencies have been examined with medium resolution. Nineteen bands were analyzed for their rotational constants. Combination differences of four transitions from the ground state were averaged and the rotational constants $B_0=0.99156\pm.00004$ cm^{-1} and $D_o=1.17\pm.07\times10^{-6}$ cm^{-1} obtained. This B_o value together with those recently obtained for C_2H_2 and C_2D_2 have been used to calculate the ground state and equilibrium state bond distances of the acetylene molecule. The l-doubling constants for the degenerate modes were determined to be $q_4=4.4_2\times10^{-3}$ cm^{-1} and $q_5=3._6\times10^{-3}$ cm^{-1}. The frequencies of the bending modes have been calculated by use of difference bands to be $\nu^1{}_4=518.38$ cm^{-1} and $\nu^1{}_5=677.77$ cm^{-1}.

Audiofrequency dispersion effects in lanthanide salts at low temperatures, R. P. Hudson and B. W. Mangum, *Book, Magnetic and Electric Resonance and Relaxation, Ed. J. Smidt, pp. 135–146 (North-Holland Publ. Co., Amsterdam, The Netherlands, 1963).*

Measurements have been made of the quadrature components of the audiofrequency susceptibility of three rare-earth paramagnetic salts in the liquid helium region and in the presence of an external magnetic field. The susceptibilities and derived values of the spin-lattice relaxation time are compared with theory and suggestions put forward to explain major anomalies in the case of dysprosium ethylsulfate.

An LCAO-MO-SCF study of the structure of HO_2, M. E. Boyd, *Program Comm. Intern. Symp. on Molecular Structure and Spectroscopy, pp. B404–1–B404–4 (Science Council of Japan, Tokyo, 1962).*

A priori computations by the self-consistent field molecular orbital method have been performed on the radical HO_2 in an effort to determine its geometry. For a linear configuration the energy was computed as a function of internuclear distances. Fixing these at the value giving the lowest energy, calcuiations were performed of energy as a function of the HOO angle, and gave an energy minimum for an isosceles triangle geometry having $D(O_2\text{–}H)$ 4 e.v. and an ionization potential of 10.95 e.v. in reasonable agreement with mass spectrometer data.

Dependence of the electrical conductivity and thermoelectric power of pure and aluminum-doped rutile on equilibrium oxygen pressure and temperature, J. Yahia, *Phys. Rev.* **130,** *No. 5, 1711–1719* (*June 1, 1963*).

Several defect models for the production of charged carriers in pure and Al-doped rutile are discussed in this paper. For the case of pure rutile, it is shown that the anion-vacancy model does not lead to the observed pressure dependence of the conductivity whereas the titanium interstitials model does lead to the proper result. For the case of Al-doped rutile, the anion vacancy model combined with a certain choice of reaction constants gives a pressure dependence of the density of electrons (and holes) that is close to that observed. The titanium interstitials model is not solved explicitly for this case (as for the anion vacancy calculation) but it is shown that it may give rise to the right pressure dependence of the electron and hole concentration. The two-band formalism of Becker and Frederikse is applied to an analysis of our data and shown to be in reasonable conformity with the observations. There are some indications that the hole mobilities ~ 180 cm^2/volt-sec at 1000°K. As a consequence of this, an effective mass for the hole ~ 10^{-2}

m_0 is deduced, assuming high temperature polar scattering to take place. These hole mobilities are considerably larger than those expected for electrons at those temperatures.

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Limitations on electron beam density of unipotential electron guns at low voltages. J. A. Simpson and C. E. Kuyatt.

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