

Mr. T.W. Barkley

# WATS ON

Attached is an extraordinary edition of 'WATS ON' consisting of the American Chemical Society's Biannual Review on recent advances in microscopy. The Editors feel that you may find it valuable to have this impressive list of references, in particular so that you can refer your customers to the original publications on any of the recent techniques which may be relevant to their work.

We do not expect you to look up all the references yourselves!!

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The American Society for Testing and Materials (ASTM) sponsored a Symposium on Interference Microscopy at its June 1962 meeting in Atlantic City. The chairman was P. Bartels. A year later, in June 1963, the ASTM sponsored a Symposium on Resinographic Methods which was organized by T. G. Rochow. Eighteen papers on various aspects of resinography were presented. As a result of the interest in resinography demonstrated at this symposium, the ASTM is taking steps to organize a new Committee E-23 on Resinography. An organizational meeting is planned during the national meeting to be held in Chicago, June 21-26, 1964.

The New York Microscopical Society has held two meetings concerned with chemical microscopy. The first was a symposium on Teaching Microscopy, held in New York City February 1 and 2, 1963, under the chairmanship of T. G. Rochow. Twelve papers were presented. On May 3, 1963, a Symposium on development of Textile Microscopy was held under the direction of F. Morehead.

A Colloque International sur les Processus de Nucleation sur les Reactions des Gaz sur les Metaux et Problemes Connexes was held in Paris in June 1963. This meeting was sponsored by the Centre National de la Recherche Scientifique and organized by M. J. Benard. The papers presented were of particular interest to those concerned with the corrosion of metals. The Service du Film de Recherche Scientifique of the Office National des Universites et Ecoles Francaise has released a number of motion pictures taken through the microscope. Titles included "Surface Oxidation in Copper", "A Microscopic Study of Electrolytic Polishing," "The Development of Magnetic Domains and the Purity of Iron," and "Electro-erosion." Arrangements for the use of these films can be made through Mr. Morot-Sir, Conseiller Culturel, Ambassade de France, 972 Fifth Ave., New York 21, N. Y.

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## BOOKS AND ARTICLES OF GENERAL INTEREST

Microscopists interested in the historical aspects of their field will enjoy reading Hooke's "Micrographia" (144), which has been reprinted by several publishers. Also of historical interest is the story of the Dollands as presented by Martin as part of his John Dolland Memorial Lecture (205). The other part of the lecture was concerned with the theory of the microscope image. Siegfried Czapski's contribution to the development of microscopy was discussed by Gause (118) in commemoration of Czapski's centenary. Booth (37) has published an unfinished manuscript written by E. M. Nelson on the subject of illuminators, in which the designs of substage condensers and vertical illuminators and the history of their development are discussed. Two new books on microscopy have been published: "Industrial Microscopy in Practice" by Burrels (54) and "Microscopy" by Hartley (137). The "Proceedings of the International Microscopy Symposium, Chicago 1960" (197) also became available. E. Leitz, Inc., New York, began publication of a Technical Information Bulletin in September 1960. Volumes 11 and 111 published in 1962 and 1963 contained several articles of general interest (180) - "Depth of Focus and Lateral Resolution," "Influence of Cover Glasses upon Microscopic Image Clarity," "Astigmatism and Curvature of Field," "Microspectrophotometry," "Measurement of Path Differences in a Polarizing Microscope," and "Photomicrography."

Causes and remedies of the 14 commonest errors in the practice of microscopy have been discussed by Furst (114). Rau (250) suggested a method for increasing efficiency in microscopical examinations. He defined an "image criterion" as a property of a microscope objective directly proportional to depth of field and inversely proportional to resolution. The image requirements of the sample are matched with the image criterion of the optical system to "ensure optimum results." Some simple chemical experiments which can be effectively carried out on the stage of a microscope have been described by McCrone (195).



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They involve analysis, use of heat, precipitation, electromotive force series, liquid crystals, polymorphism, and polarized light.

The continuing concern of the ASTM with the establishment of standards is demonstrated by publications of several specifications proposed by Subcommittee 28 of E-1: "Tentative Definitions of Terms Relating to Microscopy" (15), "Microscope Objective Thread" (13), and "Tentative Specifications and Methods of Test for Cover Slips and Glass Slides for use in Microscopy" (16).

## OPTICS

Several recent books on optics contain sections of interest to microscopists. These include a second edition of Ditchburn's "Light" (88), a book on "Instrumental Optics" by Boutry (41), and an "Atlas of Optical Phenomena" by Cagnet et al. (55). Ditchburn's book (88) contains chapters on optical instruments, defects in optical instruments, and the limitations of optical instruments, as well as polarized light, the interaction of radiation with matter, and general topics in optics. In addition to general optical principles Boutry (41) discusses vision and image formation in optical systems, simple magnifiers, eyepieces, and compound microscopes. The "Atlas of Optical Phenomena" (55) is composed of pictures demonstrating optical phenomena, many of which are of direct interest to microscopists.

An "Elementary Theoretical Approach to the Abbe Theory of Optical Image Formation" has been presented by Gerrard (121), and Chaves (62) discussed the possibility of replacing the eye with a photoelectric receptor, which could then be used to produce a television type image. According to Ronchi's energetic theory of resolving power, this arrangement should increase the resolving power to an extent dependent on the characteristics of the photodetector and its related equipment. Charman (58, 59) reports experimental measurements of diffraction images in low and high resolution microscopy. For low resolution microscopy (58) the experimental results agree with theory. However, for circular objects, 0.1 to 0.3 micron, requiring high resolution microscopy, agreement between theory and experiment was not so close.(59).



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The influence of condenser aperture is also shown. De and Som (82) also discuss diffraction images of circular openings formed with partially coherent illumination. Equality of the numerical apertures of the condenser and objective does not ensure a state where incoherent illumination can be assumed. This affects measurements of the size of particles and may lead to misinterpretation of images.

Taylor (291) shows that many useful concepts regarding optical imaging can be taught visually without mathematics, using optical diffraction patterns (usually called optical transforms). The necessary apparatus is described and illustrations of the application of the method are presented. O'Neill (230) and Walther (308) are also concerned with optical transforms, as they discuss phase in image formation and phase retrieval in optics. Stokes (286) discusses the theory of optical properties of inhomogeneous materials. Langberg et al. (174) describe an image forming resonance scatter filter which may have applications to microscopy.

Hariharan and Sen (136) describe a radial shearing interferometer which can be used to measure the aberrations of microscope objectives. Hansen is concerned with the transmittance of optical instruments and describes research directed toward its measurement (135). He measures the transmittance of microscope objectives from 2100 to 6000 Å. (185). He also discusses the energy transmission limitations inherent in the Casperson microspectrophotometer as built by Zeiss (134).

## INSTRUMENTS

Microscopes. Montgomery and Hundley (216) describe a flying spot television microscope, of a design based on suggestions by Barer. The instrument is particularly useful for dry mass measurements. Barer and Wardley (22) discuss ultraviolet television microscopy which makes possible the measurement of the refractive index and absorption of living cells. With this type of microscope it is possible to vary image contrast at will. Lau (176, 177) describes the construction and testing of a "double microscope."

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Long et al. (184) have built a hypodermic microscope using fiber optics. Images of living tissue are transmitted through needles as small as 0.021 inch in inside diameter. A special ocular corrects for distortion caused by the necessary bevel at the outer end of the needle. Bayer (28) suggests an unusual shadow microscope which forms an enlarged image of a thin layer on the surface of a convex spherical mirror.

Wide-field, large-pupil binocular eyepiece designs were investigated by Spiro (282). Huygens-type oculars for apochromatic objectives are described by Biot (34, 35). Bordet (39) has built a special ocular for use with a polarizing microscope, with which one can see a conoscopic figure and the image of the crystal producing the figure simultaneously. Branemark (51) has modified a Berek condenser for remote control handling. This condenser also provides the possibility of working with relatively monochromatic light.

Stages. A cooled stage using semiconductor thermoelectric cooling has been described by Crawley (74). Sims (278) uses a warm plate for the microscopical observation of living cells. Peck (235) has built a stage with glass microscope slides and cover glasses, heated with a filament, which has low heat capacity and is useful up to 400°C. This stage is of such dimensions that it can be used with standard optics, including oil immersion optics. Mercer and Miller (208) have modified a previously described hot stage to allow continuous recording of cooling curves of melt droplets at cooling rates up to 20,000° per second. A low temperature vacuum stage, used for observation of the deposition of thin films (24) and a high vacuum stage ( $10^{-7}$  torr) (213) have also been described. A general description of high temperature metallographic equipment is contained in Lozinskii's book (185).

Recently microscopical observations have been extended to the field of ultra-high pressures by Van Valkenburg (304). With his high pressure optical cell, phase changes resulting from application of high pressures to solids and liquids have been observed in the polarizing microscope .



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Fisher (100) recommends the use of a temperature-controlled "spindle stage" in place of a universal stage for the measurement of index of refraction by the double variation method. Oppenheim (231) describes the construction of a spindle stage utilizing a hypodermic syringe. Kouhoubt (170) uses a motor-driven rotating stage for studying the rheological properties of fibrils 1 to 10 microns in diameter. A special stage for measurements of photographic records in nuclear emulsions has also been described (210).

Miscellaneous Accessories. Friday (110) describes a micromanipulation apparatus involving the sliding of glass plates lubricated with a viscous oil. An insect holder (200) for use under a stereomicroscope should prove useful for holding many types of specimens which need to be turned so that all sides can be observed. Devices for measuring physical properties such as tensile strength (181) and vectorial hardness of crystal faces (143) have also been constructed.

Light sources for microscopy are the subject of papers by several authors. Pijper (237) describes a heliostat used primarily for dark-field microscopy. He reviews work done with this heliostat and with an earlier model. Agroskin and Korolev (1) discuss monochromators for use with microspectrophotometers, and Couling and Young (72) describe a simple constant deviation ultraviolet microscope illuminator. Hughes et al. (145) have designed an illumination system for opaque objects which uses a circular reflector above the specimen and a substage light source.

A modification of the Hardy microtome using a micrometer barrel to advance the specimen is described by Mannering (201). Przybylska (246) has devised a simple means of cutting organic crystals.

Steel (283) shows that a visual method of focusing due to Simon can be adapted to photoelectric setting with an increase in precision. Leifer et al. (179) have designed a back-projection screen for microprojection work. This screen is grainless and has high definition and luminance. A cover-glass spring clip made from paper clips (69) appears to be a useful device. The apparatus necessary for the study of living human cells has been discussed by Dawson (81). Equipment for containing, nourishing, and maintaining sterile cultures at the proper temperature is described.

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Accessories for observing and photographing the cells -- namely, phase microscope, cinecamera, microflash unit, and time lapse unit -- are discussed.

Additional instruments are described under specific headings such as polarizing microscopes and interference microscopes.

## POLARIZED LIGHT MICROSCOPY

Shurcliff (276) has written a book on polarized light, which should prove of great interest to microscopists who use polarizing microscopes. Smakula (280) discusses the fundamental properties of single crystals and their application in polarizing radiation in the spectral range from x-rays to microwaves. Bartels (25) gives the principles of observation and measurement using a polarizing microscope. He discusses the theory of the interaction of polarized light with specimen materials and the interpretation of the image. Nahmmacher (219) gives a similar discussion of polarizing microscopy with emphasis on resinography. Schmidt (268) considers the relationships between the structure of Pleurosigma shells and the birefringence and visibility of pores and slits as observed in the polarizing microscope. Hartshorne (138) describes methods of illumination in polarization microscopy, and discusses the requirements for high resolution polarizing microscopy. Green et al. (131) gives the design of a polarizing microscope for observation of magnetic domains using the Kerr magneto-optic effect. The limit of resolution is 2.8 microns. Booth (38) proposes a simple system for viewing interference figures. The system is not a microscope, but is made from a microscope ocular. Allen and Rebhun (5) have devised an apparatus for the photoelectric measurement of small fluctuating retardations in weakly birefringent, light-scattering biological objects using a revolving tilted compensator. The smallest detectable retardation is 1 A. Grabar (129) proposes a method of measuring opticaxial angles on a Bxa figure. Nikitenko and Indenblom (223) discuss the polarizing infrared microscope and its application in the examination of silicon. Cameron in his book on "Ore Microscopy" (56) presents the theory of the reflection of polarized light from materials, describes the apparatus for observing the effects, and explains the interpretation of the image with regard to the examination of ores.



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## ULTRAVIOLET AND INFRARED MICROSCOPY

Barer's ultraviolet television microscope (22) has been mentioned. A high temperature ultraviolet microscope designed by Deacon and Barella (83) permits observation of sintering at magnifications up to 150X and at temperatures limited only by the stability of the heating filament which also supports the sample. Ruch (258) discusses ultraviolet microspectrography. Zaitseva et al. (319, 320) apply ultraviolet microscopy to the examination of metals in reflected light. Forty (106) uses transmitted ultraviolet light to examine the internal structure of alkali metals, silver, and silver-based alloys. Felton et al. (99) survey ultraviolet equipment and its use in resinography. Young and Edwards (318) use the ultraviolet microscope to detect formazan deposits in tissues. Infrared microscopy has been utilized in the examination of silicon (223) and thin sections of coal (188).

## INTERFERENCE MICROSCOPY

Barakat and Mokhtar (20) have extended the work of Tolansky to find the optimum conditions for maximum intensity of a multiple-beam Fizeau fringe. Schulz (271) describes a simple interference microscope for opaque objects, while Francon and Yamamoto (109) describe one for transmission examination of isotropic specimens. Rienitz et al. (254, 255) have designed a new transmitted light interference microscope utilizing two objectives. The flying-spot television microscope mentioned above (216) is also an interference microscope. McCartney and Ergun (191) use interferometry to measure the thickness and refractive index of ultrathin sections of coal.

Applications of interference microscopy to the study of metallographic specimens are described by Mehaute (206), and the technique has also been used to examine the surfaces of iron whiskers (300) and glass after exposure to alkalies (169). The interference microscope has been widely used in the quantitative examination of biological specimens and this application has been discussed by Walter (307). Evreinova and Kuznetsova (97) have used interference microscopy in a study of coacervates, and Wunderlich and Sullivan (314) applied this method to the determination of the thickness of single crystals of high polymers.

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## MICROSPECTROPHOTOMETRY AND MICROPHOTOMETRY

A discussion of instrumentation for microdensitometry and microspectrophotometry has been presented by Zieler (323) and Kruger (171) along with a discussion of problems encountered using these techniques. Wagener and Grand (305) describe an ultraviolet recording spectrophotometer, and Zanotti (321) and Sacchi (261) discuss auxiliary equipment, and the use of a comparator ocular, respectively. A scanning microscope for photoelectric data evaluation is described by Tove et al. (298). Image discrimination techniques for cytophotometry are presented by Mitrany et al. (212) and by Drets (91). Kelly and Carlson (159) suggest the use of protein droplets as microscopic standards for quantitation of cytochemical reactions. Applications of microspectrophotometry to the analysis of spots on paper (141) and to the colorimetric determination of a substance in a few drops of solution (53) have been described.

## FLUORESCENCE MICROSCOPY

No attempt has been made to review biological microscopy. At the present time fluorescence microscopy is most widely used in the biological sciences, but it deserves wider applications. A review of "Microfluoroscropy in Pathology" has been written by Gagne (116). Some quantitative methods of fluorescence microscopy are described by Donath (90) and by Ruch and Bosshard (259).

## SPECIMEN PREPARATIVE TECHNIQUES

Embedding and Replication. Methods of embedding several types of samples have been reported: soil samples by Cornwall (71) and Wells (311), wood samples by Boutelje and Ishida (40), coals and cokes by Zeidler and Taylor (322), Luther et al. (188), and McCartney et al. (191, 192), and textiles by Vanderhoof and Borysko (302). The last authors also describe replication methods for textile materials.

Staining. A critical review of the use of dyes in starch microscopy is given by Seidemann (272). He reviews methods of identifying starches, detecting adulteration, and distinguishing among whole, damaged, and swollen starch grains. Marinozzi (203) describes a new technique for staining materials with silver.



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The method is applicable to fixed embedded specimens.

Microtomy. A new design for a fiber microtome is described by Mannering (201). Cloud (67) gives two methods of sharpening Rolls razor blades for microtomy. Francis and Groves (108) apply microtomy to the preparation of microscopical specimens of baked goods. Wright and Hall (313) use microtomy for the study of resins, and Tice and Lasko (295) have used ultramicrotomy to study the morphology of fine metallic filaments.

## TECHNIQUES FOR SPECIMEN EXAMINATION

Petrography. Cameron (56) has written a book entitled, "Ore Microscopy," which describes instruments, methods of specimen preparation, methods of measuring physical and optical properties of ores, microchemical techniques, and systems of mineral identification. Ore microscopy is primarily concerned with the examination of polished surfaces of ores using vertical illumination, and, therefore, the theory of the reflection of polarized light from materials is presented. "Determination Tables for Ore Microscopy" is the title of a book written by Schouten (270) which is a compilation of data needed for the practical application of the techniques given by Cameron. Luther et al. (188) have investigated thin layers of coal with visible and infrared light microscopy. They describe their methods and give examples of applications. The preparation of ultrathin sections of coal and their examination by transmitted light are described by McCartney et al. (191, 192). They measure thickness and refractive index of coal by interferometric methods (191). A method of preparing thin polished sections of clays and shales is given by Tourtelot (297). Stone (287) has built a simple device which can be used in conjunction with a cutoff wheel to make thin sections of rocks quickly.

Autoradiography. An inverted cover-slip method for the autoradiography of microscopical specimens has been worked out by Savage (263). Dawson and Field (80) describe a method of differential autoradiography of tritium and another beta-emitter using a double stripping film technique. Moyer and Ochs (217) have shown how additives are segregated when polystyrene, polypropylene, and polyethylene are crystallized in thin films. The additives were labeled with tritium, and autoradiographs were prepared and compared with the microstructures revealed by the polarizing microscope.

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Photomicrography. Two books on photomicrography, one by Brain and Toncate (47) entitled "Techniques in Photomicrography" and one by Schenk and Kistler (266) entitled "Photomicrography," have been written. A second edition of Michel's "Die Mikrophotographie" has been published (209). Ludwig has written an article on the practice of microscopy (186). Stereophotography is the subject of a book by Pietsch (236), which includes a discussion of stereomicrography. Platzer (238) also discusses the same subject. Pybus (247) describes the adaptation of an electrically driven (Vinten MK.1) 16-mm. camera for cinephotomicrography. He also describes the construction of a electromechanical timer and control unit for time lapse work. The use of 16-mm. cinecameras in cell research is the subject of an article written by Kuhl (172). Needham (220) gives hints on 35-mm. color photomicrography with emphasis on the use of the Leica camera.

Descriptions of several exposure meters designed for photomicrography have appeared. Elkan and Thrush (96) describe a home-built photo-multiplier exposure meter circuit and tell how to calibrate the meter. Barer and Underwood (21) use CdS photocells for exposure meters for microscopy. These cells are less sensitive than photomultipliers but are 10 times more sensitive than Se cells. Robust, portable, and inexpensive meters can be built using CdS cells. Kalin describes an exposure meter with sensitivity adequate for phase, dark-field, and polarizing microscopy (151). Stumbo (289) reviews several methods of determining exposure, and presents an equation for exposure evaluation, using a photometer, for 35-mm. photomicrography. This system is based on the use of the additive exposure value concept recently introduced into standard photography. Dobbins et al. (89) are concerned with wide-range photographic photometry and give techniques for developing film for high precision photographic photometry. The etching of microfine patterns in nylon photopolymer is the subject of an article by Lohse and Bruins (183). Sassoon and Parsons discuss Brownian movement in color photomicrography (262).

Refractometry. "Refractometry and Chemical Structure" by Batsanov (27) has been translated from the Russian by Sutton. This book should be of general interest to chemical microscopists. Luster (187) describes a phase microscopical technique for determining refractive indices of anisotropic particles at high magnification. Phase contrast microscopy can also be used to advantage for determining refractive index by the glass powder method of Kofler (49).



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The refractive index of coal has been determined using interferometric methods (191), and the thickness and refractive index of histological specimens have been determined using the method of de Chaulnes (260). Newman (14) has compiled considerable data on the refractive indices of resins and polymers as a result of his activities as chairman of an ASTM task group.

Particle Size. Irani and Callis (146) have written a book on "Particle Size," in which they critically compare various techniques for measuring particles. They also give definitions of terms, methods of data presentation, statistical methods, and examples of applications. Charman (60) describes experiments in which the visual sizes of circular objects of known size were measured at various instrumental magnifications, retinal illumination intensities, and states of adaptation of the eye.

Gayle and Romine (119) studied the reliability of particulate contamination analyses by comparing counts made with stereo- and regular binocular microscopes. Jerusalem (148) compares a particle count procedure proposed by Henning with another in general use. The accuracy of the methods was the same, but Henning's method was much faster. Norris and Powell (229) describe a number of ways of improving total counts of bacteria. These include a simple interferometric method of measuring counting-cell thickness for every count.

Powell and Errington (240) use the Dyson image-splitting eyepiece to measure the dimensions of bacteria. They discuss the causes of variations in size counts. Dyson and Noble (94) have designed and built a variation of the image-splitting ocular in which the setting is varied over a selected range at a low frequency. Particles within the selected range blink, those large show permanent overlap of images, and those smaller are permanently separated. Deryaguin et al. (87) describe a flow ultramicroscope which automatically counts aerosol particles. A simple method of determining water drop sizes is given by Koenig and Spyers-Duran (167) in which they use photographic film, previously treated with developer and dried, to record droplet sizes. Barker et al. (23) used projection microscopy to measure the sizes of ascites tumor cells.

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Rambabu and Rao (249) have written an article on the micrometric analysis of micaceous mineral fractions.

Miscellaneous Techniques. The problem of the measurement of the position of a small image is shown by Dyson (93) to be equivalent to the measurement of path difference. He has built an experimental polarimetric microscope capable of measuring position to 1/900 of the conventionally defined resolving power. Cooke (70) has devised a Moire fringe technique for measurement of vibrations. Moncel and Guerin have shown how to determine the mechanism of a solid-state reaction by association of microscopical observation and electronic microsond analysis (215). Joebstl (149) describes the preparation of clean crystal surfaces by cleavage of the crystals in vacuo.

Leifer et al. (178) have measured the refractive indices of Aroclor 4465 at four wavelengths. Aroclor is a useful high index mounting medium. Epoxy resins can be used for oil immersion and for heavy mineral studies (175). Dispersion staining for identification has been discussed by several authors (52, 75). Milnes (211) describes the petrofabric analysis of quartz using a point count method. Bubble growth during nucleate boiling has been investigated by Streng et al. (288). A method of studying the three-dimensional structure of alloys is given by Shevchenko and Alpatov (274), and a method of examining the growth front of a crystal by decanting the remaining melt is described by Munson and Hellawell (218). Microfractography is the subject of another paper (232), and useful techniques for studying electrochemical processes microscopically are given by Simon (277). Schmatolla (267) discusses the practical application of microscopy in the graphic arts.

The art of making picked slides of diatoms is described by Fleming in two articles (102, 103). Wischnitzer (312) shows that the phase microscope is a valuable adjunct to the electron microscope for cytological studies. Reumuth and Loske (252) describe the numerous possibilities for the use of cuvettes in microscopical investigations of movements and changes in both biological and nonbiological objects. Wahls (306) describes a technique for producing paraffin rings for moist chambers using a mold. Swift (290) shows how to set up the necessary apparatus and to carry out biological and chemical investigations in a hanging drop.



Crossman (76) suggests optical methods for examining bacteria, yeast, and mold colonies and emphasizes the use of polarised light. Twomey (301) gives a simple method for the preparation of microscope slides of mold cultures growing on agar. Microstarch gel electrophoresis is discussed by Daams (79).

## APPLICATIONS OF CHEMICAL MICROSCOPY

Crystallography. A number of recent books on crystallography should be of interest to chemical microscopists. "The Art and Science of Growing Crystals," edited by Gilman (122), is not particularly concerned with microscopy, but there are several microscopical techniques described in it. Smakula (279) in his book "Einkristalle: Wachstum, Herstellung und Anwendung" discusses the growth and decomposition of crystals, their structure, and the theory of crystal growth as well as methods of growing crystals and their uses. "Kristallchemie" by Kleber (163) and "Crystallography" edited by Fox et al (107) discuss numerous aspects of crystallography. Terpstra and Codd (294) in their book "Crystallometry" promote the use of traditional methods, such as goniometry, for the identification and orientation of crystals. This book provides an excellent background for the chemical crystallographer. Gray (130) has written on the "Molecular Structure and the Properties of Liquid Crystals."

Arnold et al. (10), in the sixth of a series of articles on the isomorphism of liquid crystalline phases, discuss the textures of liquid crystalline phases. In a very interesting article (233) Palm and MacGillavry show that the modification of the habit of NaCl in the presence of urea is caused by the absorption of units of  $\text{NaCl} \cdot \text{urea} \cdot \text{H}_2\text{O}$  on the (111) faces of NaCl. They also find that the same complex is adsorbed on the (001) face of urea crystals, resulting in habit modification. Research on the etching behavior of nearly perfect crystals of high purity alpha-iron is described by Gorsuch and Alden (128). Bansigir and Schneider (19) have studied the substructure of pure and doped alkali halide crystals by optical and magnetic resonance methods. Albon (3) proposes a mechanism for the formation of dislocations during crystal growth. Brandstatter-Kuhnert (50) demonstrates again that crystal growth patterns alone are not reliable for identification of compounds, especially those which exhibit polymorphism. Crowle et al. (77) and Benas (29) discuss periodic (Liesegang) precipitation. Stein and Powers (285) provide a mathematical discussion of the factors involved in the microscopical measurement of the growth of spherulites.

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Berry and Skillman (31) discuss the ideal size distribution and growth in microcrystalline precipitates. Walton (309) shows by nephelometric methods that the dimensions of critical nuclei of sparingly soluble salts are close to unit cell size and therefore very few ions are involved in the nucleation of crystals of these materials. Kapustin's article on the effects of ultrasound on the kinetics of crystallization has been translated from the Russian (153).

Albon and Dunning (4) have determined the edge energy of growth steps on sucrose crystals. Powers (241, 242) describes microscopical studies of sucrose crystallization. Jones et al. (150) give optical and x-ray diffraction data on the sucrose-calcium chloride-tetrahydrate complex. McCrone (194) has published data on the polymorphism of acetanilide, TNT, RDX, DINA, TNR, HMX, and other compounds. Yamada and Miki (316) have studied the absorption of the red cupric complexes with succinimide using polarized light microscopy. Tolansky (296) describes the growth and etch phenomena which he has observed on synthetic diamonds. Yakel et al. (315) give optical and crystallographic data on the manganese (III) trioxides of the lanthanides and yttrium. Rose (257) has published a note on the crystallography of metahexamide, and Shead (273) has noted crystallographically shaped and oriented holes in thin platy crystals of  $\text{Ag}_2\text{Cr}_2\text{O}_7$ .

## Deformation and Defect Structure in Crystals.

A recent book, "Direct Observation of Imperfections in Crystals," edited by Newkirk (222) is not only of general interest but also includes sections on light microscopy. The "Course on Radiation Damage in Solids" edited by Billington (33) includes sections on crystalline defects and their detection by electron and light microscopy. Bradbury (42) has written on the structural changes produced in uranium oxide during irradiation and under the influence of high temperature gradients. Etch pits have been utilized by King (160) in his investigation of small cyclic strains. Parasnis (234) has decorated dislocation loops in AgCl by adding 0.1 mole % of  $\text{CuCl}_2$  to the crystal and subsequently annealing it at  $455^\circ\text{C}$ . These loops are visible in dark-field illumination. Nishijima and Oster (228) discuss the observation of diffusion effects using a microscope. The initiation and development of surface cracks, and the influence of the surrounding medium on these phenomena, have been investigated (161).



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The proper shape of an indenter for measuring vectorial hardness of a crystal face has been discussed by Hofer (143). Studies of the plastic deformation of crystals have been carried out on  $\text{Cu}_3\text{Au}$  by Kear (156), on  $\text{Tl}_2\text{O}_3$  by Ashbee and Smallman (11), and on  $\text{MgO}$  by Amateau and Spretnak (6). Mendelson (207) reports experiments on glide band formation and broadening in ionic crystals and gives a theoretical discussion of these phenomena. Klassen-Neklyudova (162) has written a long paper on the mechanical twinning of crystals.

Resins and Polymers. Part III of Volume XII on the "Analytical Chemistry of High Polymers" edited by Kline (164) has a section on microscopical methods written by Newman; Part II has a section on optical methods written by Forziati. Although Fisk's new book, "The Physical Chemistry of Paints," is not directly concerned with microscopy, it includes sections on crystallography, particle size, wetting, rheology, and colloids which are subjects of interest to microscopists (101). The spherulitic morphology of thin films of natural rubber has been described by Andrews (8). Lanceley et al. (173) report a wide range of crystalline forms observed when  $\omega$ -dicarboxylic acids crystallize in polymer films, and discuss the effects of these crystals. Bassett et al. (26) report microscopical observations of some new habit features in monolayer polyethylene crystals. A whisker-like growth of polyoxymethylene is described by van der Heijde (139). Hermans (140) describes the flow of cellulose microcrystals. Wunderlich and Sullivan (314) use interference microscopy to examine crystalline high polymers. Giuffria has made microscopical studies of the gloss and sheet morphology of polystyrene (125), the orientation of extruded polystyrene sheet (123), and the structure of rigid polyurethane foam (124). The influence of microstructure on the mechanical behaviour of crystalline polypropylene has been reported by Andrianova et al. (9), and Ward (310) discusses optical and mechanical anisotropy in crystalline polymers.

Textiles and Fibers. The "Microscopic and Chemical Testing of Textiles. A Practical Manual" by Koch (166) has been translated from the German by Hooper. Mogensen (214) has designed and built a glass cell which permits observation of a single filament of a fiber-forming material as it is extruded and coagulated in a liquid medium. Rollins et al. (256) review the use of light and electron microscopy in research on the finishing of cotton textiles.

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Prevorsek (245) describes a microscopic method for studying free thermal shrinkage. Bobeth and Vollrath (36) have observed differences in the physical state of polyacrylonitrile fibers by swelling them with  $\text{ZnCl}_2\text{-I}_2$  solutions. Berestnev et al. (30) have investigated the structural irregularities of viscose fibers using light microscopy. The surface characteristics of natural and synthetic fibers can be studied microscopically by treating them with Tollens reagent (126). Bradbury et al. have published a series of articles dealing with microscopical studies of wool (43-46). Inorganic materials on the surfaces of coarse vegetable fibers have been examined by replica methods (64), and the photodegradation of 6-nylon has been observed by Kato (155).

Paper. A new stain for detecting lignified cell walls has been proposed by Maacz and Vagas (190): a 0.5% aqueous solution of Astrazone Red F.G. Cloud (66) reports on a "Technique for the Preparation of Microscope Slides of Photographic Films, Papers, and Similar Materials." He gives methods of embedding, sectioning, and mounting. The ASTM has published a revised method for fiber analysis of paper and paperboard (12).

Minerals. Volume 3 of "The System Of Mineralogy by James Dwight Dana and Edward Salisbury Dana" has been revised by Frondel (111). This volume is on the silica minerals. Deer, Howie, and Zussman (84) have completed Volumes 2, 3, 4, and 5 of "Rock Forming Minerals." These volumes give information on the chemical, structural, and optical properties of minerals. Volume 2 is on chain silicates, Volume 3 on sheet silicates, Volume 4 on framework silicates, and Volume 5 on nonsilicates. Rehwald (251) has written on the development and future of petrography. Gazzi (120) has made a microscopical determination of the amphiboles in grains. Keil (158) reports a quantitative microscopical ore analysis of chondrites, and Knox (165) describes the microstructures of several stony meteorites. Microscopical and thermogravimetric studies of the thermal decomposition of coals have been reported by Luther and Bussmann (189). McCracken and Weed (193) describe the micromorphology and associated physical properties of southeastern soils.

Ceramics. Techniques for the microscopical examination of ceramic materials are given by Fryer and Roberts (113).



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Konovalov et al. (168) have compiled an atlas of micrographs showing the microscopic structure of cement clinkers, refractory materials, and slags, and Scholz (269) has written an article on the examination of cement clinker under the microscope. The microstructure of lime and its effect on the petrographic characteristics of portland cement clinker and on the strength of the cement have been reported by Fataliev (98). Bailey (18) discusses the microscopy of steam cured asbestos cement products. Three papers on microscopical examination of alumina ceramics have appeared: one on techniques by Taylor (292), one on the effect of microstructure on the permeability of sintered alumina materials to gases at high temperatures (112), and one on the contribution of optical microscopy and x-ray diffraction to the study of refractory alumina (32). High temperature microscopes were used by Kanclir and Demovic (152) to study kaolins, and by Levin and Nikulina (182) to study feldspar. Two papers (68, 264) are concerned with the microscopy of the strontium oxide-titanium dioxide system.

Temeshi (293) has described a high temperature microscopical method of determining the temperatures of melting and crystallization of slags. A comprehensive study of steelmaking slags from basic open-hearth furnaces, with particular emphasis on the role of MnO and MgO in the steelmaking process, has been made by Tromel and Gorl (299). Riegger et al. (253) describe the microstructures which develop in periclase when it is subjected to the conditions encountered in steelmaking.

A tentative explanation of the volume changes resulting from the growth of crystals during the hydration of  $\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O}$  is given by Chatterji and Jeffery (61). Eliseev (95) describes the development of the microstructures of some ferrites. Marley and MacAvoy (204) have investigated the growth of stannic oxide crystals from the vapor phase. They report three different crystal habits, depending on the temperature of growth. Petrographic methods were used by Yarmak (317) to investigate the formation of the microstructure of fired household porcelain. Amorphous and microcrystalline silicas were studied by Floerke (105), and a "Crystal Chemistry and Micro Morphological Study of Carbonate Apatites" has been reported by Newesely (221). The surface structure of water and ice is the subject of a paper by Fletcher (104).

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Metals. A second edition of "The Physical Examination of Metals" by Chalmers and Quarrell has been published (57). A method of studying the alloys is given by Shevchenko and Alpatov (274). The interface between a growing crystal face and the melt can be studied if the molten material is decanted (218). Lozinskii's book on "High Temperature Metallography" (185) has been translated by Herdan. This book discusses principles, apparatus, methods of examination, and interpretation of microstructures, and reports the results of studies of many materials. Mitsche and Jeglitsch (213) have studied quantitatively the selective evaporation of elements from the polished surfaces of alloys during heating at  $10^{-7}$  torr. Barras et al. (24) report a study of the phenomena observed at low temperatures during the deposition of thin metallic films in a vacuum. A study of the nucleation habits and branching phenomena of iron whiskers has been made with an interference microscope (300). Methods of studying electrochemical processes microscopically are described by Simon (277), and Jacquet (147) reports on the microstructures developed on annealing at electrolytic deposit of chromium at  $500^{\circ}$  to  $1000^{\circ}$ C. Hilliard (142) discusses the specification and measurement of microstructural anisotropy. The fractured surfaces of specimens of sintered iron have been studied by microfractographic methods (232). Some materials problems in electrical contacts and their solution by microscopical methods are presented by Keil (157). Aksenov and Drozdov (2) have investigated the microstructure of copper reduced from scale.

Miscellaneous Materials. Microscopical methods for the study of deposit formation in sunflower oil are reported by Ciocanelea and Rosu (65), and the solid phases in lubricating oils have been examined microscopically by Dudin and Mazharov (92). A book, edited by Ramachandran, entitled "Aspects of Protein Structure" (248), contains discussions of x-ray, chemical, and microscopical methods. Dempsey et al. (85, 86) have discussed the microscopy of leather. A review of present knowledge of the formation and distribution of amylose and amylopectin in the starch granule has been presented by Badenhuizen (17). The importance of starch in the microscopical identification of cereal grains in feed is brought out by McMasters and Waggle (199), and Smith (281) gives a general discussion of feed microscopy. Czaja (78) describes microscopical methods, using polarized and natural light, for the detection of ergot and cockle in flour.



# WATS ON

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## ANALYTICAL MICROSCOPY

One of the important reasons for the widespread use of chemical microscopy is that very small amounts of material can be examined, identified, and in some cases quantitatively determined. Some of the philosophy and problems connected with the identification of small quantities of material as well as papers on specialized topics are gathered into a book on "Submicrogram Experimentation" (63) edited by Cheronis. One of the sections of this book, dealing with the identification of single particles, was written by McCrone (196). McCrone and Salzenstein (198) describe work done in preparation of an atlas of atmospheric particulates, intended as an aid in the identification of particles collected from the atmosphere. The "Identification of Atmospheric Dust by the Use of the Microscope" is the title of a book written by Hamilton and Jarvis (133).

Brandstatter-Kuhnert has presented three articles of analytical interest. Two of these deal with methods of identification worked out by Kofler. One (48) is a review with 12 references, and the other (49) is concerned with an investigation of the use of phase contrast microscopy with the glass powder method of measuring refractive index. The third article, written in collaboration with Junger (50), shows that "crystal patterns" are not reliable for identification in pharmaceutical and forensic work. This is especially true with barbiturates, which exhibit polymorphism and isomorphism.

A recently developed method of identifying materials is dispersion staining. Brown and McCrone (52) discuss the theory, methods, and apparatus, and Crossman (75) describes some variations of the method which are referred to as "central screening" and "apertural screening".

A number of papers which describe microscopical methods of identifying elements and compounds have appeared recently. Heyndryckx (141) has used "ultramicrospectrophotometry" to identify spots on paper containing Co, Cu, or Zn. Gordon (127) has given techniques for the separation and identification of certain minerals and compounds in feeds: Limestone, orthophosphates, bentonite, sucrose, urea, S,  $MgSO_4$  and NaCl. Van Ligten and Cool (303) have discovered a new microscopical crystal test for Be using hexaminocobalt (111) chloride. Schaeffer (265) describes the reactions of the platinum metals with certain pyridine derivatives.

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2- Acetylpyridine can be used to detect 16 g. of Au or Pt chloride in a drop volume of 1/30 ml.; 2, 2'- bipyridine can be used to detect 33 g. of Os, 8 g. of Pt, 615 g. of Au, and 5 g. of Pd. Manolov (202) identifies Ag by means of a crystalline derivative, silver diamminithiocyanate. Gamble (117) describes a method of identifying isoparaffins by the formation of adducts of these compounds with urea and thiourea. In an article on the "Organic Chemical Microscopy and Thixotropic Behavior of Phenyl and Naphthyl Urethanes" Shulman et al. (275) give photomicrographs of derivatives of a series of alcohols. These derivatives, usually esters, were used to characterize the alcohols. Kartnig (154) describes a rapid method for determining hexachlorocyclohexane in the presence of DDT using the microrefractive index determination devised by Kofler.

For many years chemical microscopy has been used to identify drugs and pharmaceuticals. Amelink (7) has written a book on "Rapid Microchemical Identification Methods in Pharmacy and Toxicology." Nikolics (224-227) has written a series of articles generally concerned with rapid "microcrystalloscopic" methods for detecting and assaying drugs and pharmaceuticals. Steimetz (284) presents a systematic scheme for microscopical identification of organic and inorganic compounds. Twenty-five standard reagents are used. He illustrates the method using reactions for identifying aspirin, antipyrine,  $\text{CaBr}_2 \cdot 2\text{H}_2\text{O}$ , and  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ .

Five articles describe methods for the identification of various drugs and pharmaceuticals. Gupta and Kofoed (132) describe the separation, purification, and identification of some common barbiturates. Pligin (239) uses "phenyl stibine acid" and its derivatives for the microcrystalloscopic analysis of alkaloids and their synthetic substitutes. The microcrystalloscopic reactions of tekodine are described by Pozdnyakova and Bukanova (244). Pozdnyakova (243) also describes the microcrystalloscopic reactions of homoatropine, platiphylline, dicaine, and putrescine, and gives their optical constants. Reinecke salt,  $\text{K}_4\text{Fe}(\text{CN})_6$ , styphnic acid, and Dragendorff reagent are used as reagents. Fulton (115) describes tests for O<sup>3</sup>-monoacetylmorphine, and codeine. Minute amounts of these materials eluted from a paper chromatogram are treated with 1-KI reagent, HCl, and  $\text{H}_3\text{PO}_4$  or with  $\text{HAuBr}_4$  in  $2\text{H}_3\text{PO}_4$ -1HBr. Distinctive crystals, identifiable microscopically, are formed.



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