

Chemical Microscopy

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The last review in this series appeared in April 1982 (B110); see below. The current review covers December 1981 to November 1983.

Since chemical microscopy touches on virtually every science and has made inroads into almost every technology, a review of the subject cannot be totally complete. As in previous years, there has been an increase in the amount of activity in this field over antecedent review periods. Hence a strictly comprehensive treatment would be even more difficult. Thus, it was necessary to be somewhat selective in the choices that were made for inclusion of citations in this work. A great effort was made to make the review fair, representative, and proportionate.

Some section categories in this review are almost empty except for some cross-references. The headings were kept because the lack of activity may be important to many readers. If important work was omitted it was accidental. I would greatly appreciate having such omissions brought to my attention for possible inclusion in the next review.

A positive effort has been made to include non-English references (noted in the bibliographic citation). The response in terms of reprint requests from outside the U.S. and the U.K. was almost overwhelming after the 1982 review, attesting to the popularity of this policy and to the truly international scope of this journal. Besides, a grossly distorted view of the work in many subject areas would result if other languages were discriminated against. Some areas simply are not dominated anymore by English speaking workers.

Only published works appearing in regularly abstracted proceedings and journals were considered for inclusion here except for a few highly relevant patents and government publications. The breadth of the horizon in chemical microscopy has made it necessary to use an alphanumeric numbering system for references. Sections range from A1 through Z1, A2 through Z2, and A3 to B3. Bibliographic citations are pointed to within a section by the rightmost one or two digits as in A17 or B211.

A1. Books of General Interest. A work on the use of microscope has appeared under the authorship of Marmasse (A11). Written for microscopists, it presupposes some knowledge of microscope design and applications. The book is a critique of many procedures, equipment, and accessories and it contains numerous savvy "tricks" and shortcuts. The author's approach is refreshingly unconventional yet logical and insightful. Spencer's (A12) "Fundamentals of Light Microscopy" was jam-packed by the author (in only 85 pages of text) with a distillation of important facts about microscopes and microscopy. It could make a useful reference book or a good textbook in the hands of a knowledgeable instructor but is not recommended for the lone beginner.

Volume 8 of "Advances in Optical and Electron Microscopy" edited by Barer and Cosslet (A13) appeared in 1982. It is divided about equally between optical and electron microscopy and the optical component is divided about equally between biological and physical applications. For instance, spacial filtering, photoelectric setting microscopes, polarized fluorescence, and linear dichroism microscopy were covered in articles by contributing authors.

A "no-nonsense practical working manual" that is a practical guide to basic microscopy and photomicrography was advanced by Smith (A14). As a first edition (spiral bound) it contains numerous small faults, errors, and omissions. Otherwise of good quality, the work could be a hindrance to the beginner and should not be used alone.

The "Handbook of Chemical Microscopy, Vol. 1", has appeared in a fourth edition by Mason (A15). Though it is only slightly changed from the third edition, it is still a fascinating manual of step-by-step procedures, experiments, and background material that should be indispensable to the practicing microscopist, "chemical" or not.

Mikroskopion (A16) is an irregularly published serial now available in four languages (English, French, German, and Spanish). Number 38, now current, features articles on photomacrography and micrography, a new generation of surgical operating microscopes, and applications of stereo-microscopes and macroscopes to problems in industry.

As a result of a merger *Micron* (A17) is now a very new journal first appearing in its new format in the fall of 1983 (Vol. 14, No. 3). It is the result of a combination of the old *Micron* and *Microscopica Acta*, now no longer in existence.

B1. Articles of General Interest. The popularity and importance of light microscopy are indicated by the amazing success of a new course in the "Techniques of Microscopy" offered by Michigan Technological University, Houghton, MI. Erbish (B11) provided a rationale for this course, an outline of course development, and a detailed description of course content; biological and nonbiological applications are emphasized equally. Educational benefits are identified and discussed, not the least of which was the salutary effect on enrollment statistics.

Beginning with Antoni Van Leeuwenhoek, microscopy has always been a scientific haven for the serious amateur, sharing this characteristic with astronomy. Myers and Duggan (B12) discussed their experiences in setting up a microscopy course for young people. The scene was Siena Heights College, Adrian, MI. The authors outline the philosophy and objectives of the course with allusions to prior work and precedent. They discuss planning, scheduling, recruitment of students, and teaching methods. Resources for contacting potential instructors are mentioned and a summary of course evaluations from both the students' and instructors' point of view was given.

One of the most fascinating modes of communication of the principles of chemical microscopy is that of case histories. Seemingly miraculous solutions to crucial, time-wasting, and expensive industrial problems is the forte of Coates (B13) who presents case histories from high technology at the Polaroid Corp.

Ford (B14-B16) scored a mighty coup with his discovery of intact specimens prepared by Van Leeuwenhoek himself. Nine small packets containing dried alga, diatoms, water fleas, and thin sections (<30 μ m) of cork and of bovine optic nerve plus many other specimens were uncovered in the archives of the Royal Society. The author supplies detailed background and descriptions of the specimens, including photomicrographs. The first of the cited references even contains pictures taken through one of Van Leeuwenhoek's own microscopes.

Some instructions for testing microscopes for chromatic aberrations and resolving power are given by Göke (B17, B18) with theoretical background and detailed explanations.

The attitude of the physicist toward the microscope is displayed, often with trenchant humor, by Dodd (B19). To him the optical components of the compound microscope include the specimen and what is *really* interesting is the manner in which the system reveals the astounding nature of light.

Kentgen (B110) reviewed chemical microscopy in 1982. That review covered the period from January 1978 to November 1981.

C1. Optics. A rotating mirror microscope was developed by Hara (C11). It was used to continuously record micro blood vessel diameters and the effect on them by catecholamine. Ormont and Maksimov (C12) perfected a scanning light microscope for dual mode study of surface structures of semiconductors in combination with a scanning electron microscope.

A stroboscopic microscope is described by Handschy and Clark (C13) who found it useful for observing fast electrooptic switching in ferroelectric smectic C liquid crystals. At the other end of the spectrum, Zeiss (C14) has produced a limited

edition replica of the Stativ VII, one of the first microscopes to have been designed according to the Abbe theory of optical imaging. This historic microscope was a model of craftsmanship as well as a revolutionary technological advance.

See also: *G12, W16, W17.*

D1. Instruments (including tools, accessories, components, etc.). A special microscope device for simultaneous reflected light intensity measurement and micro differential scanning calorimetry was designed by Haines and Skinner (*D11*). They used it to investigate heats of phase transition for a variety of substances including polymers and liquid crystals. Fu and Xu (*D12*) have designed a simplified microscopical melting point apparatus.

An unusual microminiature refrigerator is the heart of a cryostat for coldstage applications. Its use was envisioned by Yakushi et al. (*D13*) for microspectroreflectometry.

A miniature tensometer that fits on a polarized light microscope stage was designed by Polymer Laboratories Inc. (*D14*) for deformation, yield, and fracture studies on polymers and like substances. Control features allow video recording of the fracture or deformation process if desired. This can be done with simultaneous recording of a stress-strain curve. An environmental cell is available as an option giving temperature stasis between ambient and 100 °C. The drive is computer controlled allowing complete oversight by the operator and very complex loading patterns.

A new graticule for particle size determination of aerosols and liquid droplets, that is, for homogeneous (or nearly so) particulates, is advanced by Chea (*D15*). Metal/chromium oxide films were employed by Koike and Yamamoto (*D16*) to act as antireflection coatings for optical instrument diaphragms.

A slideless microscope was described by F. Deindorfer of International Remote Imaging Systems, Inc. (Chatsworth, CA) as reported by McCrone (*D17*). The system design allows specimens to be suspended between layers of flowing fluids. Shear induced orientation effects allow simplified, more rapid computer image analysis. The system lends itself to automated, high throughput, high accuracy microscopical analysis of blood cells, urine, and possibly many other suspended or suspendable materials.

See also: *U24, V12-5, W11-W12, W18.*

MICROSCOPICAL METHODS

E1. Polarized Light Microscopy. Because of the extensive, even pervasive, use of the polarizing light microscope (PLM) throughout most of the subject areas covered in this review, it is not possible to mention all references to its use under this heading. the PLM is the primary tool of the chemical microscopist and it really deserves a substantial section, perhaps a review, all its own. However, some typical references will have to suffice to give the reader an idea of the capabilities of this analytical tool. In order to apprise him of its usefulness, a complete set of cross-references to major PLM research cited in the rest of this review is provided here under "see also".

Bloss (*E11*) has written a book on the use of the single-axis rotation (spindle) stage. Crystal system, the positions of all three extinction positions, alpha, beta, and gamma refractive indices and their dispersion provide many unique characteristics. (Note to the reader: by comparison, for routine work, actually only three to six IR bands are usually very definitive and PLM can provide this many characters). The spindle stage is a convenient tool for rapid, accurate, and precise measurement of these intrinsic crystal properties. A computer program, EXCALIBUR, is presented which helps plot the data obtainable and refines it for even greater precision. The value of combined X-ray and optical study of crystals is described. Three appendices plus 91 references are included.

The study of structure and morphology of crystals, especially of twinning in minerals, is reviewed by Uno (*E12*). Optical analysis of material failure, including the use of polarization and interference polarization microscopy, is discussed by Van Maaren (*E13*). Corrosion studies, resinography, the behavior of cutting tools, quality of welds, and other applications are described. PLM and microspectrophotometry are used by Vidal, Mello, and Pimental (*E14*) to study complexes of various dyes such as Chloramine Fast Red with collagen. Dye molecule orientations with respect to protein macromolecules are determined.

McCrone (*E15*) reiterated in the strongest possible language his firm conclusion that the so-called "Shroud" of Turin is a fake. He cited previously published work that showed the presence of typical pigment particles in "image" areas and their total absence in nonimage areas. He called for radiocarbon dating to settle the divergence of views caused by some key evidence that happens to be completely contradictory.

Fundamentals of PLM were presented by Matthiessen and Probst (*E16*) and by Kremer (*E17*) who emphasized the simplicity of the equipment and the ease with which teaching materials may be prepared. "Chemical Art" was demonstrated by Jung (*E18*) in the form of incredibly beautiful photomicrographs of frequently observed PLM epiphenomena.

See also: *A15, B11-3, D14, R12-4, W12, C26, D21, D23, D24, D27, D212, E23, E25, E28, E216, F25, F28, L21-3, L28, L214, L216, L217, L221-31, M21, N21, N24-6, O22, O28, O210, T21, A33-7.*

F1. Microphotometry and Microspectrophotometry. Raman and IR microprobes plus UV, visible, and fluorescence microspectroscopy are reviewed by Hirschfeld (*F11*). Microspectrophotometers for small sample analysis in forensics is discussed by Hausdorff and Coates (*F12*). The chemical microscopist, ever the consummate generalist, will be interested in a review of microspectrophotometry in plant biochemistry assembled by Dhillon, Miksche, and Cecich (*F13*).

Maestre and Katz (*F14*) describe a circular dichroism microspectrophotometer. The IKS-14 near-infrared instrument and its use for the measurement of optical absorption spectra of minerals are discussed by Taran and Nazarov (*F15*). The spectral range 750-2500 nm is covered for many small samples. Elsirafy (*F16*) describe a new method for signal enhancement in microspectrophotometric analysis of trace palladium and iron with Solochrome Red B as a reagent.

Fluorescence excitation spectra of biological materials have been recorded by Quaglia et al. (*F17*) who use a pulsed tunable dye laser microspectrofluorometer. Aoki (*F18*) describes his use of microspectrofluorometry in the study of colorless zircons and correlations with details of their origin.

Laser excitation microspectrometry was employed by Bajnoczi and Major (*F19*) to study homogeneity of metal alloys used in ancient coins.

Thermochromism and thermal decomposition of coordination compound microcrystals have been investigated by Fickel (*F110*). Absorption band assignment of transition metal complex ions in synthetic garnet group mineral microcrystals was performed by Frentrop and Langer (*F111*). They studied their specimens in the spectral range 4000-5000 cm⁻¹ and were easily able to distinguish the ligand field absorption patterns for ferric and ferrous iron and octahedral manganese(II).

Chromium oxide monolayers produced by a "molecular stratification" process on nickel, niobium, tantalum, tungsten, molybdenum, and titanium are studied microspectrophotometrically by Nechiporenko and company (*F112*).

Raman and IR microspectroscopy of iron oxide, olivines, pyroxenes and various silicates found in interstellar and interplanetary dust were the subject of investigation by Fraundorf and associates (*F113*). Fourier transform infrared emission microspectrophotometry of jet aircraft fuel deposits is pursued by Laner and Keller (*F114*). The mechanism of fading of disperse dyes in nylon fibers in the presence of ozone has been investigated by Kanrath, Reutsch, and Weigmann (*F115*) by means of microspectrophotometry.

The wavelength dependence of refractive index as a function of pressure in amorphous arsenic(III) sulfide is covered by Guo and Zha (*F116, F117*). They utilize a specially designed hydrostatically controlled high-pressure stage or cell. The same authors (*F118*) observed piezooptical properties and donor-acceptor luminescence of aluminum and copper activated cadmium zinc sulfide phosphor at high hydrostatic pressure by using the same apparatus.

Nickel complex formation in X-type zeolite catalyst was observed by Hass and Plath (*F119*) who used UV-visible transmission microspectrophotometry in Part I of a series.

Micro Raman spectroscopy, its instrumentation and applications in geology, biology, and chemistry, is reviewed by Dhamelincourt (*F120*). A new laser Raman microspectrometer called "Micromars" is described by Delhaye et al. (*F121*). By use of multichannel data acquisition, much faster operation is achieved. Spectra are typically obtained in seconds rather

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than minutes. However, some sacrifice in sensitivity and resolution is tolerated and, unlike MOLE, no Raman image can be obtained.

See also: *E14, H22, I22, I28, I29, N23, Q22, Q23, R23, Y21.*

G1. Infrared, Ultraviolet, and Raman Microscopy. Thermal imaging in microscopy for the nondestructive testing of materials is reviewed by Rosencwaig (*G11*). Nondestructive testing in general by use of thermal waves is covered. Temperature-dependent fluorescent thin films were employed as IR detectors in remote thermal imaging with 0.7 μm resolution by Kolodner and Tyson (*G12*).

Billingham and Calybert (*G13*) reviewed UV microscopy of polymers. These authors along with Ghaemy (*G14*) discuss UV microscopy in monitoring the curing of epoxy resins with amines. Also, in collaboration with Knoght (*G15*) and with Uzuner (*G16*) they studied localized oxidation of polymers such as polypropylene and the behavior of polypropylene antioxidants and polymer morphology plus inhomogeneities in epoxies.

Coherent Raman microscopy is the subject of a dissertation by Daigneault (*G17*). The determination of sulfate inclusions in halite, of ice in silicones, and of other inclusions in transparent material was achieved by Tallant, Higgins, and Stein (*G18*). They also studied chromium oxides in KCl/LiCl eutectics. Pineau et al. (*G19*) investigated atmospheric particles of respiratory health significance. They used Raman microscopy to determine the retention of airborne particulates in the respiratory tract by analyzing and comparing material found in ambient and expired air.

See also: *H21, Q23, Y21.*

H1. Fluorescence Microscopy. The orientational effect of drawing on amorphous isotactic polypropylene was studied by Pinaud and co-workers (*H11*). They used polarized fluorescence microscopy to register the ordering effect of strain on polymer chains. Dresden and associates (*H12*) describe an inverted microscope for automated measurement of proteinase activity with a fluorogenic substrate. Stanton (*H13*) used fluorescence microscopy to characterize organic matter dispersed in sedimentary rocks and to analyze the components of coal.

See also: *A13, F214, I26, L29, S22, S23, A39.*

I1. Laser and Holographic Microscopy. Marten and Ash (*I11*) successfully tested a laser photodisplacement microscope. A modulated semiconductor laser was used as a heating source and a HeNe laser served as an interferometer probe. Only single pass scans were reported, but by multiple pass scanning, a complete image correlated to thermal properties of materials near surfaces would be produced.

See also: *F11-3, F19, F113, F121, G17, G19.*

J1. Interference Microscopy. Schulze (*J11*) describe some possibilities for measurement of contact angle, hence surface tension, of a liquid by means of an interference microscope. The method amounts to a contactless determination of liquid surface properties. Much the same work is recounted by Schulze and Schoeppe (*J12*).

A simple method for studying the microtopography of crystal surfaces is set down by Prasad and Raja (*J13*). Interference contrast microscopy is utilized by Sato and Sasaki

(*J14*) to detect artificial color coatings on diamonds.

Quested and Raine (*J15*) use microscopy to examine deliberately introduced interference coatings for the differentiation of phases in nickel-based superalloys. A film of zinc sulfide is applied by reactive sputtering. The use of interference layer analysis by light microscopy for the characterization of brazed joints of nickel alloy and of titanium thermal coatings was discussed by Graef (*J16*). The interference layers here are sometimes deliberately and at other times incidentally produced.

See also: *E13, C22, E211, E214, F25, L212, R21.*

K1. Phase Contrast and Schlieren Microscopy. See (*B19*) for an interesting application of Schlieren microscopy in the study of films.

See also: *A32.*

L1. Dispersion Staining. No papers devoted to dispersion staining itself have appeared, but several emphasizing specific applications or the study of underlying phenomena were published during the review period. These have been placed in their appropriate subject categories, as indicated by the cross-references below.

See also: *A33-7, R22, V19.*

M1. Ultramicroscopy. The formation of micelles from cetyltrimethylammonium bromide and sodium dodecyl sulfate solutions was watched by use of flow ultramicroscopy by Chernoberezhskii and company (*M11*) and critical micelle concentrations were determined. Particle size distributions of colloids in aggregated and nonaggregated dispersions were determined with a modified flow ultramicroscope by Cummins et al. (*M12*). A laser flow ultramicroscope for particle counting and size distribution analysis is described by Walsh and co-workers (*M13*) and by Babyuk, Lychnikov, and Babyuk (*M14*).

Krylov and associates (*M15*) studied suspended particles in superhigh purity liquids and determined dispersity of the particles. The laser ultramicroscope was also used by Balabanov et al. (*M16*) to determine dispersity of suspended particles in volatile inorganic hydrides. Some effects due to the laser beam are noted.

Magnetic moments of ferromagnetic particles were determined with the aid of an ultramicroscope by Mikhel'son (*M17*).

See also: *E21, G23, G24, Z22.*

N1. X-ray Microscopy. X-ray microscopy is reviewed by Schmahl (*N11*), including use of synchrotron radiation as an X-ray source. Bailey and associates (*N12*) evaluate the gas puff Z pinch as a source of soft X-rays for lithography and microscopy. X-ray and α particle coded imaging and microscopy using microstructures such as a coded aperture are discussed by Ceglie, Stone, and Hawryluk (*N13*). They described reactive ion etching of such apertures as gold micro-Fresnel zone plates.

Miyake et al. (*N14*) built a high-pressure device for X-ray diffraction and X-ray microscopy of phase transitions. The apparatus is based on a specially designed diamond anvil. Progress in the development of sputtered sliced zone plates for X-ray microscopy is outlined by Rudolph, Niemann, and Schmahl (*N15*).

O1. Acoustical Microscopy. A scanning acoustic microscope has been introduced by Olympus Corp. of American (*O11*).

TECHNIQUES FOR SPECIMEN PREPARATION

P1. General Techniques of Specimen Preparation. See: *E23, E212.*

Q1. Embedding and Mounting. Graham (*Q11*) report a new cellulose caprate modified diethylene glycol distearate embedding wax. Such waxes are of use for sectioning of plant tissues.

A search for new mounting media was conducted by Liva (*Q12*) who did not identify the thermoplastic, solvent based and cross-linkable resins he tested. The properties observed are referred only to a code number, the meaning of which is not revealed. However, the author promised to supply samples of these commercially available materials to any reader who requests them.

See also: *R23.*

R1. Microtomy. A method for determining nutrient gradients near the root-soil interface of growing plants was described by Kuchenbuch and Jungk (*R11*), who used thin

sectioning of rhizospheric soil. Afonskii, Zaitsev, and Puzanov (R12) patented an epoxy resin embedding technique for the preparation of thin soil sections for analysis by optical microscopy. Microtomy of peat for polarized light and scanning electron microscopy was developed by Raymond and Bardin (R13). They obtained both elemental and mineralogical analytical data this way. Rohner and Brunner (R14) describe a method for preparing thin sections of fibers for microscopical analysis. They use an epoxy resin embedding technique.

S1. Grinding, Publishing, and Etching. See: M24.

T1. Miscellaneous Specimen Preparation. Delignification of wood fiber cell walls and a sample preparation technique for the observation of lignin distribution by UV microspectrophotometry were advanced by Gadda and associates (T11).

TECHNIQUES FOR SPECIMEN EXAMINATION

U1. Photomicrography. The following citations represent only a sampling of the work that has been done by use of this most favored method microscopical examination and analysis results documentation. The comprehensive treatise on photomicrography by Loveland (U11) was reprinted. In two volumes, it remains the only comprehensive work available in English. Errors have been corrected, some parts have been expanded, and the bibliography and list of references has been updated.

Morinaga (U12) extracted more information from metallography photomicrographs in Part 2 of his work, which resolved details of copper and aluminum alloy structures. A new method for producing colored filters for photomicrography is explained in Part 2 of the accounts by Chappard et al. (U13). The use of filters for photographic recording of staining results is discussed. Storz, (U14) discourses on some of the more interesting aspects of fluorescence photomicrography.

Widefield photomicrography is discussed by Göke (U15), who described a new Pentacon adapter.

Fourier plane micrographs were demonstrated by Delly (U15) for those who wish to triple their pleasure. These were whimsically shaped "aperture diaphragms" whose image at the microscope objective back focal plane was photographed "conoscopically", that is, with the aid of a Bertrand lens. An Abbe diffraction plate (3200 lines/in.) was inserted in the object plane to yield fascinating zeroth and first order diffraction multiple images.

Practical applications of photomicrography are exemplified by Mitrevej and Hollenbeck (U16) in water vapor adsorption-desorption and swelling of super disintegrants used in medicinal tablets.

See also: A14, Z14, Z15, A21, C211, R217, R218, T22.

V1. Refractometry. Theoretical and empirical considerations of the energy level band gap in isotropic nonmetallic substances and correlations to refractive index are covered by Borets (V11). Perhaps this represents a step toward accurate and precise prediction of refractive indices and/or band structures in anisotropic nonionic crystalline substances.

A simple hand-held linear refractometer was designed by Geake and Smalley (V12) and a patent on an interfacial contact instrument was obtained by Bartky and Stackhouse (V13). A device for refractometric measurements on photo-dielectric layers was built by Gailis and Durandin (V14). A diffraction grating was employed by Altukhov and Yatskova (V15) for the determination of refractive index.

Refractometric determination of anisotropy in mesophases was advanced by Rachkevich and Yakovenko (V16). Liquid crystal properties as a function of alkyl group chain length in homologous series of cholesteryl alkyl carbonates was studied. Pritykin and Vakula (V17) used ellipsometric refractometry to study the properties of boundary layers of elastomers. Density and packing coefficients at nitrile rubber interfaces were calculated from refractometric data.

The effect of copper(II) on the conformation of lysozyme was investigated utilizing dilatometric and refractometric methods in work by Katime Amashta, Abad, and Compos (V18).

Refraction of chalcogenide glasses is reviewed by Chepur, Rosola, and Puga (V19). The refraction and dispersion data for the germanium-arsenic-sulfur system with substitution by antimony and/or selenium are evaluated. Sigelle et al.

(V110) present a method for real-time measurement of organic crystal growing solutions.

A study on the hardening kinetics of polymer resins was conducted by Bolotina, Badalov, and Chudov (V111). Cross-link rate in epoxy resins by polyethylene polyamine is the specific subject. Rimatori, Sperduto, and Iannaccone (V112) employed differential refractometry and UV spectrophotometry to determine aerosols of lubricating and cutting oils containing polycyclic hydrocarbons in work areas. Rapid quantitative assay of pharmaceuticals with a refractometer is outlined by Wang (V113).

See also: F116, F117.

W1. Hostage and Coldstage Techniques. A Japanese patent describes a thermal analytic microscope device for studying phase transitions (W11). A polarizing microscope hostage with a controller capable of millidegree stability, which was claimed to be particularly suitable for liquid crystal studies, was revealed by Yokoyama, Kobayashi, and Kamei (W12).

An investigation of the use of various oxides as fluxing additives to feldspar for use in ceramic glazes was conducted by Kozma and Bansagby (W13). They tested magnesia and calcium oxide. Kim, Sommerville, and McLean (W14) employed a high temperature microscope for the observation of binary eutectics of borax, calcium oxide, and alumina. The ternary mixture, which is used as a flux for continuous casting of steel, was also studied. Van den Berg and co-workers (W15) prepared potassium vanadium bronze (a solution of monopotassium monoxide in vanadium pentoxide), analyzed the evolved gas, and observed the gas evolution process at high temperatures.

A hotstage microscope capable of observations on the weld structure of martensitic age-hardening steels was described and the toughness loss in plasma welds of such maraging steels due to the formation of residual austenite was investigated by Rickel, Fleer, and Klingauf (W16, W17). A hotstage microscope with a device for deformation of specimens at high temperature was presented by Sveshnikova and Gulei (W18).

Valasyuk and Radomysel'skii (W19) watched diffusion phenomena in the process of iron-graphite powder sintering. Blistering of thick copper films on printed circuit boards during firing was studied by Pitkanen, Cummings, and Sartell (W110). Atmospheric factors, including the role of air oxidation, were addressed.

Cosier and Wood (W111) designed a simple continuous flow cryostat operable in the range 10–350 K for low temperature studies with the light microscope.

See also: D13, I212, O21, O215, W21, X22.

X1. Autoradiography. Sklarew (X11) recounted a method for simultaneous Feulgen densitometry and autoradiography for the estimation of nuclear DNA in tritium labeled thymidine treated cells. A computerized image analysis system was used (a Quantimet 720D) and a counting algorithm for the computer was discussed. Yamabayashi et al. (X12) utilized tritium labeled befunolol, a β blocking agent, to study its binding to melanin granules in the eyes of pigmented rabbits.

See also: Z111, T24.

Y1. Stereology. Microscopical stereological analysis of EPOM foam rubber uncovered thermal degradation and aging effects that had not been very well characterized before. In this work by Underwood (Y11), the microstructural changes associated with foam shrinkage and compression were noted.

See also: J13.

Z1. Automatic Image Analysis. Four works of a general nature are of interest to the chemical microscopist. Varmuza (Z11) outlines pattern recognition methods in chemistry by reviewing the mathematics involved. Another such review was produced by Abe (Z12). Martens and Arndt (Z13) review image analysis, per se. Research applications of interactive image processing are discussed by Schrodt and Saunders (Z14) who describes a television-based photographic image analysis system. They determined bubble size and shape in a foam, for example.

A video digitizer and computer program for encoding chemical information inherent in photographs and even strip chart recordings is described by McCracken, Severance, and Jones (Z15).

Particle size distribution measurements by use of the Quantimet 720 image analyzer is featured by Augustin and Buerkholz (Z16). Image analysis for the determination of

respirable quartz particles in coal mine dust is detailed by Snyder and Huggins (Z17) who identified the target substance and measured particle size distributions. Spectrochemical analysis was combined with these methods for the characterization of particulate product from the PVC dry blending process in work by Burley and Hayashi (Z18).

The determination of nonmetallic inclusions in steel is advanced by Polzin, Kelly, and Kopp (Z19). They characterized oxide and sulfide particles by visually verifiable criteria. Borszecki and comrades (Z110) evaluated classification of ancient Roman coins on the basis of the mint of origin by pattern recognition applied to laser excitation microspectral analysis. Trakowski, Schoefer, and Heinrich (Z111) discuss pattern recognition for automated analysis of tracks in plastic nuclear track detectors.

See also: I29, I211, S22.

A2. Particle and Grain Size. Particle size distributions in disperse dyes were measured by Stavash et al. (A21), who used photomicrography as their main tool. In the preparation of magnesium hydroxide and magnesite by the electrolytic process, Chauhan and Datar (A22) employ light microscopy to determine transparency and particle size distribution.

See also: D15, M12-6, Z16-8, I213, J23, Z21-3.

B2. Miscellaneous Techniques for Specimen Examination. Microscopical and spectroscopic ellipsometry with a focus on applications is reviewed by Matsuda and Sugimoto (B21). They are especially interested in studies of iron passivation and of lead oxide.

APPLICATIONS OF CHEMICAL MICROSCOPY

C2. Crystallography. Crystal growth and surface structure of potassium hydrogen phthalate in aqueous solutions was observed by Jetten, Van der Hoek, and Van Enckvort (C21). They used reflection microscopy. Kinetics of crystal growth plus the convective and diffusional aspects of the development of prismatic faces by potassium dihydrogen phosphate and deuterium potassium phosphate single crystals were investigated by Zugrav and Itter (C22). They used reflection and interference microscopy.

Electron and optical microscopy were integrated by Stuckenschmidt and Granger (C23) who studied vapor deposited silver chloride whiskers. Chen et al. (C24) made detailed measurements of the distribution of crystal growth inclusions in lithium niobate. New crystal phases in alkali earth sulfites and their solid solutions with calcium hydroxide dihydrate were discovered by Cohen (C25). He used thermal methods in addition to microscopy to classify the phases.

Polarization microscopy with the application of an electric field was used by Borodina, Kuznetsov, and Borodin (C26) to image ferroelectric domains in plate-shaped crystals of barium titanate.

Crystal dislocation dipoles and loops in gold-doped magnesium oxide were examined by Rao and Parasnis (C27). Deformation techniques were used by Rao (C28) to condition crystals for microscopical study of similar dislocation loops in pure MgO.

X-ray diffraction intertwined with optical microscopy was used by Bochkarev and company (C29) to investigate the nature of barium fluoride inclusions and certain structure defects in lead tin telluride solid solutions. The same combination was used by Morlevat, Uny, and Jacquet-Francillon (C210) to determine the extent and rate of crystallization in borosilicate glass radioactive waste confinement matrixes. They compare the suitability of the two methods.

High-speed video microscopy was used by Tsukamoto, Abe, and Sunagawa (C211) to explore high-temperature crystal growth and dissolution processes.

A study of crystallization in gold-rare earth amorphous films was conducted by use of optical microscopy by Shikhmanter, Talianker, and Dariel (C212). Hashimoto and Kambe (C213) observed crystallization processes of amorphous antimony deposited on silver substrates. Along with Sugibucki they (C214) looked at the same phenomena on such films deposited on glass under ultrahigh vacuum.

Kibizov (C215) patented an optical microscopy technique for the determination of the evenness of crystal dendrite twinning planes produced during fracture of materials with the diamond structure. Observations on the metal-carbon film that forms from iron-manganese-nickel alloy during the growth of synthetic diamond were made by Feng (C216).

Clues were sought on the mechanism of action of the metal solvent-catalyst to produce this material.

See also: E11, E12, F18, F19, V16, E24-6, E29, E210, P21, Q22, Q25-7, X23-5, B31.

D2. Liquid Crystals. The application of polarized light microscopy to the study of liquid crystals in terms of their microstructure and supermolecular organization is reviewed by Nicastro (D21).

Styrene/poly(oxyethylene) graft copolymers with water, 2-propanol, and styrene form micellar systems. Lamellar liquid crystals form from such mixtures when styrene is replaced by toluene. Candau et al. (D22) use low angle X-ray diffraction and optical microscopy to study the structural and topological features of these swollen polymeric mesophases.

The surface structures of smectic B liquid crystals are studied with polarization microscopy by Warengem and Billard (D23). The same instrument is used by Gawrisch and colleagues (D24) for lyotropic liquid crystal phase transition investigations. They determine transition temperatures of dipalmitoylglycerophosphatidylcholine mesophases.

Deuterium NMR combined with microscopy is employed by Alcantara and associates (D25) to determine some properties of potassium laurate/decanol-water induced lyotropic cholesteric mesophase. They describe some texture effects. Potassium laurate/KCl/water transition between mesophase and micellar pseudophase are observed by Figuerredo and Amaral (D26) with combined XRD and optical microscopy.

Crystal growth of single cubic liquid crystals from cholesteric blue phases is studied by Onusseit and Stegemeyer (D27) utilizing polarization microscopy. Kinetic parameters for isothermal crystallization of cholesteryl nonanoate and caproate are determined by Adamski, Klimczyk, and Czyzewski (D28). Mesophase formation, development of spherulites, and surface free energies are observed or measured.

Smectic and nematic phase polymorphism of homologous series of bis(alkoxybenzylidene)phenylenediamine at high pressures was investigated by Hermann, Kleinhans, and Schneider (D29). Reentrant nematic octyloxycyanobiphenyl and cyanobenzylideneoctyloxylaniline were also studied. Hermann (D210) employed high-pressure microscopy to determine P-T phase relations in the above two cyano compounds and their mixtures. A hexyloxyphenyl decyloxybenzoate smectic A to smectic A phase transition was observed at high pressure by Shashidar, Hermann, and Kleinhans (D211).

The melting of *N*-(*p*-ethoxybenzylidene)-*p*-butylaniline mesophase was observed by Yasuniwa and Minato (D212) by polarized light microscopy. Phase transitions in hexyloxybenzylidene toluidine were studied by Miyajima, Nakamura, and Chihara (D213). They also used thermal analysis to observe the crystallization of dendrites from the liquid crystal phase. Smectic C phase transition and morphology were investigated by Kostromin and company (D214). Thermotropic and electrooptical effects on cyanoacrylate mesogen containing polymers were examined by means of microscopy, DSC, and XRD.

Lastly, Viney, Donald, and Windle (D215) detected banded structures in oriented thermotropic mesophases of polyesters and polyamides.

See also: C13, D11, M11, J24-7.

E2. Resins, Polymers, and Their Additives. Late-stage phase separation and droplet coalescence were studied by Nojima, Shiroshita, and Kenji (E21). They used optical microscopy with light scattering to observe critical solution state phenomena in polystyrene liquid mixtures containing silicones and diisodecyl phthalate. Possible mechanisms of phase separation were discussed. Phase separation and morphological characteristics such as segmental structure were related to one another in work by Camargo and associates (E22).

Sample preparation was the major topic in effort by Boehme (E23). Optical microscopy as applied to analysis of polyamides and poly(oxyethylene)s is the object of this discussion of specimen manipulation.

Paracrystal morphology in aramid fibers, their size distribution, and theory describing equilibrium paracrystal growth, number, and size characteristics were investigated by Hindeleh and Hosemann (E24). Polarized light microscopy and small angle light scattering were used by Pietzsch and Moritz (E25). They looked at the thermal behavior of

spherulites in polyethylenes. Melt-crystallized samples of polyethylene were obtained by Gedde, Eklund, and Jansson (E26). The microscopical aspects of fracture analysis relating morphological features to molecular weight segregation in the samples were then advanced.

Methods for determining the purity of melts for fiber spinning of polyester were set down by Schmidt, Halbmair, and Otto (E27). Polarization microscopy was used for the detection and characterization of inhomogeneities in high-pressure polyethylenes in a patented technique by Hirte et al. (E28).

Solid polymer strength and aging relationships to crystal structure in emulsion PTFE were found by Qian and Li (E29). Combined methods for microstructural analysis of PTFE were evaluated by hornbogen and Wittkamp (E210). They demonstrated the usefulness of optical microscopy together with dilatometry and X-ray diffraction in a three-pronged attack on problems in polymer structure/properties relations.

Plastic zone sizes in crack propagation of PMMA under cyclic loading were measured by interference microscopy as reported by Doell, Koenczoel, and Schinker (E211). Metallography as it has been applied to characterize plastics and polymer structure features and trace components is comprehensively treated by Fazano (E212). He covers optical microscopy of plastics, surface preparation of specimens and interpretation of results.

Korab et al. (E213) and Koenczoel and associates (E214) examined welds in plastics. The former is a general treatment and the later focused on interference microscopy of welds in PMMA. Thermoplastic sliding surfaces undergo friction induced changes in morphology which Schaefer and Wittkamp (E215) observed in polyethylene using optical microscopy. Finally, internal stress, polymer chain orientation, and shape factors in injection molding were observed and controlled by Menges et al. (E216) using information provided by polarized light microscopy.

See also: D11, D14, E13, G13, G14, G16, H11, V17, V111, D22, F21-4, F28, F210-13, G25, M21, B31.

F2. Textiles, Fibers, and Films. The process and mechanism of yarn breaking were studied by Ulmann (F21) who watched the rending of polyamide, polypropylene, acrylic, rayon, and cotton yarns under the microscope. Gandurin and company (F22) examined sized synthetic yarns. Acetate and nylon reactions to isoprene/vinyl alcohol copolymer size were observed as a function of pH. Swelling was one of the effects noted as were size distribution in the yarns and solubility of size in the yarn fibers.

Polyester fiber undergoes structural changes during dyeing. These changes affect crystallinity and orientation of polymer chains. Schnaitmann, Fiebig, and Herlinger (F23) used interference microscopy to study these effects in polyester fiber treated with disperse dyes.

Fluorescence microscopy, enabled by fluorochrome staining of polymeric finishing agents, identified polyester, polyurethane, polysiloxanes, and hydrocarbon waxes on woolen textiles in work performed by Rothery and White (F24).

Polarizing interference microscopy was employed by Zurek and Zakrzewski (F25) to determine the optical properties of cotton fibers. Refractive indices, birefringence, and cross sectional area were measured. A method for preparing yarns for microscopical analysis of cross sections was described by Mavlyanov, Kungurtseva, and Pedozubova (F26) who used the technique to determine the degree of sizing in carboxymethylcellulose and starch-sized yarns.

Fraser, Courtney, and Harvey (F27) studied the use of the agrochemical bioregulator glyphosate for accelerated maturation of flax stem tissue. These light microscopical observations defined some of the effects of "preharvest retting" on stem fibers.

Polarized light microscopy determined degree of crystallinity and polymer morphology of stereoblock polybutadiene film in the work of Georgiev et al. (F28). Boni and co-workers (F29) made microscopical in situ observations of shear deformation in poly-1-butene. The effects of spherulite size on plastic deformation thin films were seen in relation to heat of fusion of polybutene paracrystals, degree of crystallinity, and various types of spherulitic transformations. Popova (F210) looked at morphology in polyethylene film by reflected light microscopy. New details of polymer structure in these crystallizable plastics were revealed. Morphological changes

on thermal degradation of fluoropolymer condensation and powder sinter films were noted by Bagmut and company (F211).

Microindentation hardness measurements were made on ethylene/vinyl alcohol copolymer coatings by Bickel, Jahn, and Mylius (F212) who applied an altered infinitesimal modulus method. Jahn and Mylius (F213) presented a sampling of their results on microindentation hardness measurement of plastic coatings. They included computer algorithms for handling raw test data. A patent for determining the cracking susceptibility of coatings was granted to Boxhammer, Mueller, and Borth (F214). They employed fluorescence microscopy and accelerated weathering under stress. They targeted laquers but the method could be used for other coatings.

See also: D16, F112, F115, V11, V12, J14-6, M21-4, O26, O212, P23, R22-5.

G2. Colloids, Gels, and Dispersions. The microstructure of sheared colloids was determined with light scattering analysis by Ackerson and Clark (G21). Oil droplet diameters produced by ultrasonic disruption were compared to those achieved by conventional methods (commonly used by the pharmaceutical industry to make emulsions) in work advanced by Eberth and Merry (G22).

Laser light scattering was utilized by Weiss (G23, G24) to analyze polyacrylamide gels under the microscope. Gel structure was related to dynamic light scattering functions.

Self-organization of highly charge latex particles to form coexistent ordered and disordered lattices was investigated by Ise et al. (G25) who studied potassium styrene sulfonate-styrene copolymer latexes. Novak (G26) discusses chromatography, spectrometry and microscopy as analytical tools useful to inkmakers.

See also: M11-7, P23, Q23, Q25, Q27, R215, B301.

H2. Wood and Paper. Saka and co-workers (H21) study the bromination reactivity of morphologically distinct regions of sprucewood. They employ energy dispersive X-ray analysis and UV microscopy and compare results obtained by both means. The lignin in the middle lamella fragments found in mechanical spruce pulp is measured by Bouteleje and Erickson (H22). They use UV microscopy and UV microspectrophotometry to determine lignin concentrations. The delignification of pine by the soda-oxygen pulping process is investigated by Gadda (H23). He looks at lignin distribution in cell walls after pulping.

See also: T11, R29.

I2. Coal. The role of residues in the agglomeration of vitrinite in short residence time catalytic hydrogenation and liquefaction of coals was studied by Bodily, Shibaoka, and Yoshida (I21). Microspectrofluorometry was used by Shibaoka and Russel (I22) to investigate coal hydrogenation residues. Microscopical observations of solvent swelling of coal were made "in situ" by Brenner (I23).

Rhoades, Gray, and Huntington (I24) review the microscopy of coal, coal components, carbonization, and coking. Metamorphism, coalification, and gelification of soft brown coals in relation to their low-rank vitrinite content was studied by Philip et al. (I25) who combined microscopy with pyrolysis and GC-MS. Teichmueller and Durand (I26) examine fluorescence properties of embedding resins for coal fluorescence microscopy in their investigation of rank classification of liptinites and vitrinites in peat and coals. They compare their results with those obtained by rock-eval pyrolysis. Vleeskens (I27) made an investigation of pit coal utilizing a technical classification system based on microscopy.

Microphotometric analysis of bitumens dispersed in sedimentary rock as it relates to metamorphism of fossil fuels is the subject of a special issue of the *Journal of the German Society for Mineral Science and Coal Chemistry* (I28). A Belgian patent (I29) describes an automated microphotometer that determines coal rank by reflectometric measurement. Timons (I210) as well as Shevchenko and Suslova (I211) determined the maceral composition of coal by reflectance microscopy coupled with automation techniques involving microcomputers and other methods.

Using transmitted light microscopy, Brenner (I212) studied the diffusion of oxygen into coal during burning. Points of penetration by oxygen in relation to coal components were identified by using thin sections at high temperatures. Flammability and thermal ignitability of coal and other dusts

is related to particle size distribution factors and microscopical ash analysis by Hertzberg and fellow workers (J213).

The role of a fluid mesophase in the removal of molecular and microstructural defects in coke was studied by Oya, Qian, and Marsh (J214, J215). They used XRD integrated with optical microscopy. The British Carbonization Research Association (J216) published a report on the microstructural correlations to many physical properties of electrodes containing carbon black, coke, and pitch.

See also: H13.

J2. Petroleum. Dow and O'Connor (J21) review kerogen microscopy as applied to petroleum prospecting with special reference to kerogen maturity and type determinations made by reflected light microscopy. The mechanism of the carbon dioxide flooding process in petroleum recovery is observed microscopically by Wang (J22) while Takamura (J23) characterizes porosity and particle size. He measures particle size distribution of Athabasca oil sand in order to describe its structure.

Gasparoux, Jaunait, and Garrigau-Lagrange (J24) used microscopy, IR spectroscopy, and magnetic measurements to study a carbonaceous nematic liquid crystal phase formed during pyrolysis of acenaphthalene. They also determine viscosity and other properties of the mesophase. Chwastiak, Lewis, and Ruggiero (J25) quantitatively determine mesophase content in pitch. Microscopical observations are made on the mesophase during the carbonization of coal tar and petroleum pitch as reported by Uemura et al. (J26). They use a hotstage. Perrotta, McCullough, and Beuther (J27) study petroleum pitch mesophase under pressure and high temperature.

See also: F114, H13.

K2. Lubricants. See J2, V112.

L2. Minerals. Feklichev (L21) outlined polarized light microscopy and other microscopical methods for the analysis of minerals. A book, "Microscopic Examination of Minerals", was authored by Inoue (L22).

Carman (L23) produced a spindle stage study of the optical properties of a topaz and he described a technique that uses this tool for the analysis of inclusions in such minerals. Zhao (L24) reviewed the implications and significance of fluid inclusions, both gaseous and liquid, in minerals. Their meaning in geological research was discussed. Refractivity measurements were used by Yeh (L25) to determine molar volumes and microscopic densities of water structures found in various minerals. Harris (L26) used a very high temperature cryopumped vacuum hotstage to microscopically observe melting and degassing of volcanic glasses. Measurements of vapor pressures were performed between 77 and 273 K while degassing characteristics were observed up to 1280 °C as water, sulfur dioxide, and carbon dioxide were emitted from such glassy igneous minerals as pumice. These gases were quantitatively determined on a microscale.

Microchemical determinations of composition within a slate were related to cleavage lamellae microstructures by White and Johnston (L27). Twinning and other microstructural features of microcline found in some granitic rocks and pegmatites were recorded by Fitzgerald and McLaren (L28).

Gruber and Sternad (L29) reported UV fluorescence microscopical detection and identification of gypsum alteration products in calcareous stone used as building material. Spinel, chromspinel, clinoenstatite, magnesioferrite, monticellite, forsterite, periclase, and other mineral substances were detected in magnesian refractories by Perepelitsyn (L210), who used reflection microscopy.

Optical microscopy was used by Peres, Klinger, and Izmailova (L211) to detect and analyze for silica in limestones. The silica occurred as opal or cristobalite and was present in the insoluble residue remained after selective dissolution of the calcium carbonate matrix.

Anderson (L212) used Nomarski interference contrast microscopy to study periodic zoning in plagioclase etched and polished sections. The geochemical genesis of phosphate rock and phosphorite deposits in West African Precambrian strata of Upper Volta was studied from a microchemical and petrographic point of view by Lucas and Prevot (L213).

Petrographical and ore microscopical studies were conducted by Fasfous and Awad (L214). They were concerned with the formation of chromite deposits by metamorphic differentiation in shear zones of the Eastern Desert in Egypt.

Dry mounted and immersed specimens of lead chalcogenides were studied by reflectance microscopy using the Leitz MRV-2 microscope by Kulichikhina (L215). The ore microscopy of cuprotochilinite was discussed by Murumatsu and Nambu (L216) who obtained their specimens from the Kamaishi mine in Japan.

The ore microscopy of uranium minerals was reviewed by Hagni (L217). Similarly, Schidlowski (L218) reviewed the metallogenic implications of microscopical observations of the uraniferous components of conglomerates found in the South African Witwatersrand.

Friedrich (L219) identified sulfosalt minerals by microscopy and used his data for the improvement of mineral diagnosis. Over 25 different sulfo salts and related minerals were characterized. The morphology and origin of sulfide and wustite microscopic rods in silicate crystals of metallurgical slags were described by Wearing (L220).

The treatment and processing of ores and production of metals they form as they have been monitored by microscopy was reviewed by Haagensen and Martinez (L221) and also by Canepa and Bernuy (L222).

Ore separation and beneficiation by flotation and the use of the microscope in process control is discussed by Marley (L224). The treatment of zinc, lead, and silver ores was the focus of this work. Microscopy and its use in following the flotation separation of spodumene, a lithium ore, from pegmatite is covered by Hansen, Brown, and Atwood (L225). Quantitative microscopical characterization of a sulfide beneficiation process is outlined by Naruk (L226). The use of microscopy in gold recovery from pyritic ores is detailed by Hansen (L227).

Kamalov and company (L228) studied the microscopical characteristics of phosphogypsum crystals from the nitric-sulfuric acid attack on phosphate and phosphorite rock.

McKeague (L229) remarks on the synergism between light microscopy and SEM-EDXRA in analysis and characterization of undisturbed Canadian soils. Special emphasis is placed on soil genesis. Bisdom and colleagues (L230) used optical microscopy to interpret the result of weathering processes on biotite and its secondary products. Pinstaking microscopical analysis with preservation of structure of arenaceous saprolite derived from weathering of granite was conducted by Curmi (L231). Weathering characteristics in relation to composition of the plagioclase components was also studied.

The presence of salts in marine alluvial soils was studied by Kooistra (L232) while Rodriguez et al. (L233) focused on aridisols formed by the weathering of volcanic rock. The removal of iron oxides from soil thin sections by two different methods are compared by Pagliai (L234).

See also: E11, F15, F18, F113, F120, R12, R212, A31-9.

M2. Composites. We are opening this new category because we know that there is a great deal of light microscopy being done on composite materials. We wish to encourage its publication.

Composites are of inherent interest to the chemist because of their often unique composition and because of their multiphase nature. The category is of interest to chemical microscopists because of its growing importance and because of the challenge in applying their science and art to these fascinating materials.

For the purpose of this review, a composite is any multiphase material containing artificially imposed pattern or order for the purpose of enhancing the engineering properties of the product (often synergistically) above what would be achieved by mere dispersion or physical mixing of unformed or unshaped phases.

Sometimes composites are self-organizing to some extent, perhaps only under strain or stress during use. Hence, a paperlike material containing randomly oriented wood and polyester fiber constitutes a composite because the polyester fibers are shaped and controlled in length and because they are free to become partially aligned when the paper is strained, with a resulting enhancement of yield strength.

Metals and alloys, though multiphase, deserve a category of their own. However, metal alloys "grown" in space may have the order usually attributed to composites. Such materials would be placed in their obvious primary category with a cross-reference number under M2.

Only four publications have appeared in the open literature during this review period that we could find. Examination

of U.S. National Technical Information Service files shows several more could have been presented "publicly".

Kong, Pruneda, and Morgan (*M21*) used polarized light microscopy to study deformation and failure of epoxy/aramid-fiber composites. Janes and Majdic (*M22*) examined the physical and physicochemical interaction of silicon carbide and alumina fibers with the matrix in refractory concrete based on grog. Similarly, chemical reactions of aluminum oxide fibers with such matrixes were closely inspected in these reinforced magnesia grog materials.

Graphite fiber morphology as it relates to reinforcement properties when made into metal composites was investigated by Madronero (*M23*). This microscopical examination of the fibers themselves is Part II of a series on graphite fiber reinforced metals. Forrest and Marsh (*M24*) looked at the structure of carbon fiber reinforced carbon. The fibers were embedded in a carbon matrix and exposed for study by a chromic acid etchant.

N2. Cement and Concrete. Polarized light microscopy for field and process problem solving in the manufacture of portland cement and in technical service is advanced by Werner (*N21*). Accuracy and precision of methods for phase analysis of portland cement are discussed by Aldridge (*N22*). Bogue methods, X-ray diffraction, and light microscopy are critically compared.

Quantitative microphotometric determination of alite and belite in cement clinker was carried out by Chromy and Maki (*N23*). Polarized light microscopy for the measurement of birefringence, color, particle size, and phase composition in terms of alite and belite in cement clinker is described by Fundal (*N24*). Polarized light microscopy using the Ono method is discussed and reflected light microscopy was used for quantification of phases in cement clinker by Hargave and associates (*N25*). Major discrepancies were found in the Bogue approach to quantitation. Better methods were advanced. The Ono method was refined by Ono (*N26*) whose PLM technique was used to determine stage of cement burning, burning condition, and clinker quality as it relates to hydraulic strength. Alite size and birefringence and belite size and color are important determinants in the correlation.

Manufacturing problem diagnosis in cement clinker formation was investigated by Miller (*N27*) who applied microscopy to the mix as well as to the clinker. Burning anomalies caused by contaminant alkalis in cement clinker raw materials were studied by Demoulian and co-workers (*N28*). The effect of alite stability and clinker microstructure was determined. Sarkar (*N29*) used reflection microscopy for phase analysis of Nigerian cement clinker.

Calcium silicate polymorphic modifications in cement clinkers were identified by reflected light microscopy in a study conducted by Carin (*N210*). A method for cement plant process optimization that uses optical microscopy, particularly reflection microscopy, to determine phase composition in clinker during burning was outlined by Chromy and Weber (*N211*).

Microscopical determination of microstructure of cement used in oil wells and correlations to performance are assessed by Caveny, Weigand, and Bailey (*N212*). Makarov and Suchov (*N213*) report on the use of continuous microscopical observation of the microstructure formation resulting from dehydration of gypsum to give α -hemihydrate.

O2. Glass, Ceramics, and Abrasives. A high-temperature accessory for the microscopical study of glass devitrification was built by Shaina, Chernov, and Dorzhiev (*O21*). They investigated the crystallization capacities of magnesium aluminosilicate glasses with different thermal histories.

The thickness of the compression surface layer in oxide glass caused by stresses induced by cation exchange was determined by using polarized light microscopy and a special stratorefractometer. A measure of the induced stresses was provided by the data along with information on the degree of surface strengthening. Potassium and silver were exchanged for sodium in this work by Novotny (*O22*).

Quantitative microscopy as applied to ceramics is outlined by Kondo (*O23*). He reviews this subject and includes coverage of graphite inclusions in cast iron as quantified microscopically. Weidemann (*O24*) reviews reflection microscopy of ceramics and concentrated on specimen preparation techniques.

Schlueter, Elssner, and Ruehle (*O25*) presents a study of damage depth caused by penetrating radiation impinging on mechanically polished aluminum oxide ceramic sections. Gallant and Merigold (*O26*) review the microscopical characterization of ceramic fiber guides and other technical ceramics used in the synthetic fiber industry.

The characterization of the microchemistry and microstructure of sintered aluminosilicate refractory ceramics is detailed by Risbud and Zangvil (*O27*). Clark (*O28*) used polarized light microscopy to study refractories of many types. Numerous techniques are described.

Light microscopy integrated with scanning electron microscopy was used by Grabner (*O29*) to compare structures of plasma flame-fused magnesia refractories, chrome ore plasma fused materials, and chromite-magnesite plasma fused mixtures.

Efforts to determine the mechanism of wear in the refractories of steel making were conducted by Baker (*O210*). He used polarized light microscopy to study microtextural relations to the wear problem.

Microstructure of alumina fused-cast refractories was observed with quantitative microscopy by Carroll, Thornburg and Staten (*O211*). They looked at inhomogeneities in relation to molding of these materials. The microstructure of sintered silica ceramic fibers formed into thermal protection materials for the space shuttle is described by Newquist et al. (*O212*).

Phase composition and microstructure of synthetic abrasives is the subject of a book by Zaretskaya, Lavrov, and Filonenko (*O213*).

Intergranular phase analysis of corundum materials was performed by Kulig, Tomaszewski, and Torun (*O214*). The effect of various binder components on the wettability of corundum in fired tools, frits, and abrasives was studied by Nazimova (*O215*). A high-temperature microscope was used to investigate boron oxide, sodium tetraborate, lithium borate, and borosilicate glass binders.

See also: *W13, C210, L26, L210, M22-4, Q23, B301*.

P2. Metals. Quantitative crystallographic measurements by optical microscopy on the microstructure of some ferrous and nonferrous metals is described by Dunne and Kennon (*P21*). A review appeared under the authorship of Haemers and Bekaert (*P22*) who cover metallurgical surface analysis in materials research.

The prime importance of microscopy in the study of metal films in detailed by Rolf (*P23*) who concentrated on electrochemically formed metal coatings. Thickness determination of coatings and electroplates, etching as a tool in microscopical studies of plates, various types of coating materials, electrodeposits, and thin metal films are discussed.

Quantitative microscopy of dual-phase steels is treated by Marder and Benscoter (*P24*). They discuss ways of detecting a second phase presence by applying etching techniques. Quantitative microanalysis is covered. The importance of the light microscope is not emphasized, but their work seems of interest for horizon broadening and awareness.

Some observations using microscopy on weak acid bath bright zinc plating are made by Dumitru, Greenwald, and Varhelyi (*P25*). The theories and work of J. Henricks over the years should be kept in mind when contemplating their results.

See also: *F19, W16-9, Z19, Z110, C212-4, C216, M23, Q25, X21, X22*.

Q2. Semiconductors and Electronics. The microscopy of semiconducting materials is covered in a book by Cullis and Joy (*Q21*) as part of the Institute of Physics Conference Series.

Infrared microspectrophotometry for small-area analysis of materials encountered in semiconductor processing is described by Ramsey and Hausdorff (*Q22*). They were able to identify basic lead carbonate in soldered connections and phosphosilicate in oxide glasses, plant protein, silica, and silicon nitride films. Even interstitial oxygen could be detected. A general review of this type of analytical approach to electronics processing problems is presented by Elliot, Regnault, and Meggitt (*Q23*). They concentrated on polarized infrared microscopy of optoelectronic materials.

These references and others indicate that there will soon be an opportunity to use artificial intelligence in the expert systems sense to produce a MOLElike instrument (see *F1* and *G1*) that merges both IR and Raman spectral images into a single semiinterpreted image of great utility.

Atmani, Michon, Vautier (Q24) produced a study of crystallization phenomena in amorphous selenium thin films by optical microscopy. Likewise, general microscopy was applied to the study of the precipitation of copper from silicon on nuclei of silica. Copper was considered a metallic impurity in this work by Canepa, Manghi, and Walsoe de Rea (Q25).

To conclude, Valvian and company (Q26) used optical microscopy to study electrical properties relative to conditions of crystallization of TTF TCNQ films on glass. They also examined whiskers of this organic semiconductor.

See also: C12, C13, W110, B31.

R2. Criminalistics. Heuse and Adolf (R21) described interference microscopy for the identification of textile fibers. A large variety of natural fiber types were studied from cottons to keratins to regenerated proteins. Many synthetics were included from acrylics to urethanes to vinyl compositions. Even peanut protein fibers and spider web fibroins were included as were rubber and polyimide fibers.

Fong (R22) describes a rapid dispersion staining technique for the microscopical identification of synthetic fibers using only a single carefully chosen liquid mountant with well-characterized optical properties. Many permanent mounting media, available in Britain, are evaluated by Cook and Norton (R23) for use in forensic textile fiber examination.

The characterization of fibers and particularly of colorless fibers could be greatly extended toward near absolute certainty of uniqueness of identification if reflected light fluorescence microspectrofluorometry (RELMIF) were used to fingerprint fibers after Kubic, King, and Dubey (R24). The role of fibers in criminal investigations is discussed by Grieve (R25) who paid some attention to microscopical methods.

Strauss (R26) utilized light microscopical methods to further detail criteria for the characterization of human head hair. The actual research design and philosophy was revealed to properly apprise the reader of the researcher's inevitable biases. Conclusions of the study were moderately firm, very good hair differentiation was achieved. This study is commendable for its highly systematic approach to typing and for the high degree of success achieved toward the goal of establishing unique hair identity. A very detailed protocol for the investigation of hair as evidence is also presented by Schaffer (R27). This article is an easily understandable set of explicit instructions for the examination of hair by the criminalist. Reading it, one can almost sense an underlying, highly ordered flow chart behind the author's words.

Probabilistics and its impact on forensics, including the treatment of trace evidence studied by the chemical microscopist/criminalist, is discussed by Strauss (R28). Seven case examples are given showing how statistical principles are applied. The advantages and hazards of using probability arguments in court are discussed also.

Palenik (R29-R212) has published four sketches of famous microscopist/criminalists who used microchemical and microphysical trace evidence to solve near perfect crimes. Part I focuses on Albert Schneider M. D., Ph.D., whose reports to police officials seemed almost clairvoyant. Part II provides some clues to the thought processes behind the success of Dr. Max Frei-Sultzer, who could follow a trail more faithfully than a kennel full of police dogs. If Albert Schneider seemed clairvoyant, Edward O. Heinrich must have seemed virtually omniscient to those who benefited (or were stung) by his scientific and artful expertise, as detailed in Part III. Arthur Koehler, the wood detective, showed how the narrowly trained specialist can help solve the most important, sensational, and baffling of crimes. Koehler's evidence helped convict Richard B. Hauptmann of the Lindbergh kidnap/murder. Koehler's story is told in Part IV.

In a searing critique of the level of expertise in the use of the light microscope by chemists and microscopists in U.S. crime labs, McCrone (R213) evaluated an LEAA sponsored study. The competence of average crime lab personnel in trace evidence analysis was seriously questioned. Recent congressional hearings investigated the legal aspects of this problem, but the philosophical and moral implications of interest to every true scientist have not been addressed.

McCrone (R214) covered the comparison of soil samples by polarized light microscopy. Sample handling, fractionation methods, particle size distribution measurement, and component composition percentage estimation, among other topics, are included. The relevance of various microchemical,

microphysical, and microoptical characteristics for determining soil origin is discussed. A detailed classification technique for soil components is presented by Graves (R215).

A microcrystal test for the differentiation of *d*- and *l*-propoxyphene is advanced by Kubic and Klobus (R216). The method is based on the fact that bromauric acid forms characteristic racemic crystals from the racemate of propoxyphene. Identification of the racemate itself is straightforward. Determination of the active *d* form uses the *l* form as reagent and vice versa. Julian and Plein (R217, R218) detail microcrystal tests for a large number of illicit street drugs. Photomicrographs of positive test results are provided.

Corrigan (R219) used colloidal silica gradient separation of biological fluids to isolate sperm. Virtually isosmotic and chemically inert, poly(vinylpyrrolidone) coated silica can form gradient densities of up to 1.3 g/mL in water. Isopycnic banding is rapid under centrifugation and could be used by the criminalist for many other useful separations for subsequent microchemical or microphysical testing. A comparison to IR analysis is made.

Finally, identification of pigments found in paints and coatings for motor vehicles by transmitted light microscopy was the subject of Part III of a general work on forensic analysis of paints by Hamer (R220). Transmission and reflection microspectrophotometry is investigated by Laing and Isaacs (R221) for use in comparison of nanogram quantities of many ballpoint and fiber tip inks.

See also: F12, W21, W22.

S2. Food and Feeds. Staining of gluteins by Fast Green FCF was used by Moss, Pointing, and Berry (S21) to detect additives and adulterants in dough and other foodstuffs containing different proteins. They used light microscopy to follow the incorporation of glutenous components into food products. Fluorescence microscopy of cereals is reviewed by Fulcher (S22) with particular attention to the effects of sodium nitrite fluorescence. An "annotated bibliography" of food products microscopy was compiled by White and Shenton (S23). This is part III of a long series.

See also: Q23, A31.

T2. Biology and Medicine. Chayen (T21) wrote a general article on polarized light microscopy for the rheumatologist. Video time lapse microscopy was used by Evans, Davies, and Costa (T22) to study CHO cell phagocytosis, especially that of crystalline nickel sulfide particles in mammalian cultures.

Using both light and electron microscopy, Reginato, Schumacher, and Brighton (T23) characterized the deposition of hydroxylapatite in synovial membranes and the calcification of articular cartilage. They induced these pathological conditions with hydrotachysterol and ferrous chloride. In what may be a related development, Cipriano and colleagues (T24) studied the tendency of prosthetic heart valve materials to gather calcified deposits. They used light microscopical and radiographic methods on valves and materials tested in pigs.

See also: E14, F13, F17, F120, V13, V18, F27, R21, R214.

U2. Miscellaneous Applications. The microscopy of electrodes operating in molten salt environments is treated by Fischer and Vissers (U21). They observed and characterized surface and other phenomena associated with aluminum-lithium, silicon-lithium, and other lithium alloy electrodes in molten electrolytes.

Surface wear characterization and metal wear debris especially from steels and copper were studied by Ruff, Ives, and Glaser (U22). Wear phenomena in relation to plastic deformation of the substrate were also emphasized.

Das and fellow workers (U23) discuss microscopical and physicochemical studies on leather liming and its subsequent effects on tanning process parameters and results.

Howes (U24) details continuous microscopical observation of deformation in transparent materials by use of a specially designed microindenter.

ANALYTICAL MICROSCOPY AND MICROCHEMISTRY

V2. Elemental and Inorganic Analysis. "Microcrystal" reagents for iron, copper, and cobalt were put forth by Wills (V21). A careful distinction should be made between chemical microscopical reagents used in the manner of Chamot and Mason (V22) and microchemical reagents such as might be used to render a test compound volatile for gas chromatographic trace analysis.

Five specific microcrystal reagents for iron were evaluated by Wills (V21) along with reagents for copper and cobalt. Their suitability for determination of nanogram and picogram quantities of these ions by microcrystal methods is described. Bathophenanthroline was found to be advantageous for Fe(II), Neocuprine for Cu(I) and Nitroso-R-salt for Co(II). See the Merck Index for structures of these compounds.

A manual of microchemical methods of analysis by Stolyarov (V23) has appeared discussing broad ranges of inorganic microanalytical techniques, including chemical microscopy.

Walker (V24) has briefly reviewed the microscopy of consumer complaints involving water quality. This article concerned differential staining techniques for metal determination in dirty and discolored water. He concentrated on observations that could be made with a stereomicroscope.

See also: D12, E11, F16, F110, F119-21, G18, V18, V19, W15, L24, Q22, P22, P24, Q23, Q26, R215, X23.

W2. Organic Analysis. Thermomicroscopy and thermoanalysis of pharmaceuticals for the characterization and identification of specific drugs were the subject of Parts 15 and 16 of the continuing opus by Kuhnert-Brandstaetter's group (W21, W22). They used exceedingly finely developed light microscopical techniques including fusion methods as well as UV spectroscopy as an integrated complementary technology. Over 30 drug substances were added to the huge compendium of data they have already amassed.

Two references to microchemical analysis, per se, may be of interest to chemical microscopists. Yalcindag (W23, W24) completed Parts 4 and 5 of his work on the microchemical identification of drugs with specific functional groups. Part 4 covered guanoxine hydrochloride as an archetype while Part 5 studied amicarbalide.

See also: D12, E11, F120, F121, V18, V110, V112, V113, E27, E28, Q23, R213, R215.

X2. Structure and Phase Analysis. A review has appeared covering the study of phase diagrams by optical microscopy. Metallic systems were emphasized by Alekseenko and Aleksandrova (X21).

The lime-alumina-borax-flux for continuous casting of steel was studied by Kim, Sommerville, and McClean (X22) who used a high-temperature microscope to study phase relations in this system. Eutectic properties and structures were determined for the binary subsystems.

Tropochemical aspects of phase nonequilibrium leading to twinning in lead and bismuth sulfide as well as lead bismuth sulfide were studied by Prodan et al. (X23). Microscopic complex diamond crystal boundary structures and their properties were studied by Shen and co-workers (X24). They investigated synthetic diamonds. Poroschina and Nikonov (X25) used optical microscopy in conjunction with X-ray diffraction to determine structural characteristics of graphite intercalation compounds. Chlorine and bromine trifluorides were the intercalators. Interplanar spacings are given and intercalate phase growth models are proposed.

See also: E11, C25, D21-26, D28-15, E21, E22, B301.

Y2. Particle Identification. Supremely suited for single small particle analysis, the Raman microprobe (MOLE for molecular optics laser examiner) is both a microscope that gives Raman shift images at selectable wavelengths and a laser Raman spectrometer. Its use for the analysis of paint pigments of artistic, forensic, and industrial significance was delineated by Andersen (Y21). He also discussed X-ray dispersive analysis and SEM of particles such as pigments. He focused on examples such as the determination of rutile in delustered Spandex fiber, the occurrence of phthalocyanine pigments, many types of mineral pigments of interest to the art conservator (such as those found in Naples yellow) and pigment particulate forms and compositions of significance to the criminalist.

Tipton and Crutcher (Y22) used optical microscopy to examine particulate contaminants in transformer insulating oils. Particulate and other emissions found in waste gas from vehicle air-bag inflators were analysed by Joshi et al. (Y23). They used microscopical methods thoroughly integrated with chromatography and spectroscopy.

See also: E15-8, G19, V12, E27, E28, Q23, Q26.

Z2. Particle Properties. Part 3 of his review of particle size and distribution analysis was presented by Gahm (Z21). Many kinds of microscopy are treated. Shurov and company (Z22) analyzed suspended particles' dispersity in liquids. They

used a range of techniques including microscopy, ultramicroscopy, and X-ray diffractometry. Some chemical and physical effects by X-rays were noted whereas the optical methods produced no complications. Insoluble microscopic particles in pharmaceutical infusions were examined by Mochida, Izaka, and Tsuji (Z23). They suggest the use of reference standards for microscopical examination of such particulate contaminants and they advocate methods codes.

See also: A21, A22.

A3. Asbestos Analysis. The great concern over health hazards of asbestos continues and grows. It has been estimated that over 10 million analyses for asbestos must be performed over the next decade or so. This figure may prove conservative if current U.S. government efforts to develop inexpensive, rapid, and simple field analysis techniques are successful.

The following references highlight the chemical microscopists' response to old and new problems in asbestos detection and quantitation.

Millette, Rosenthal, and Feldman (A31) review microscopical means of detection and quantitative analysis for asbestos in drinking water. A discussion of phase contrast microscopy of the technically more important asbestiform minerals is presented by Hahne (A32). The approach is marred by a complete neglect of other complementary light microscopical techniques and a heavy tone of advocacy. Goldblatt (A33) describes rapid and reliable asbestos testing techniques using polarized light microscopy and dispersion staining. Many useful details are itemized and described in full while some tricks-of-the-trade are revealed.

Government concern over the hazards of asbestos in the environment are exemplified by three key publications. Lentzen et al. (A34) detail the U.S. Environmental Protection Agency's interim method for asbestos analysis of bulk insulation. Two methods seem destined for final approval. The first and most important is polarized light microscopy. Standard petrographic methodology is applied and the use of dispersion staining is permitted, if not encouraged, especially for less experienced analysts. The dispersion staining technique should be of value whenever speed and fatigue reduction are important. The second method X-ray diffraction (XRD) uses electronic detection of diffraction spacings and intensities. This method is meant to be used to verify PLM results, if and when necessary. It is much too complex and time consuming for first-line analysis on a routine basis. In addition, it is acknowledged that XRD alone is subject to severe interferences and ambiguities that can best be removed by prior examination with the PLM.

A technical evaluation of the interim method was published for the U.S. EPA by Brantly and associates (A35). Statistical analysis of accuracy and precision of the above methods is presented. The third member of this triad of publications is represented by Virta, Shedd, and Campbell's (A36) discourse on the need for standards for PLM analysis of asbestos. They claim there is a demand for standard reference materials representative of typical asbestos containing construction materials and of the airborne pollutants resulting from degradation of, and trauma to, these building components. The main problem was seen to be in the area of quantitation standards.

Webber, Pupons, and Fleser (A37) have at least partially answered the Virta group's concern. They have developed rather detailed recipes for the synthesis of asbestos-containing artificial bulk samples of accurately known composition for use in analytical services quality assurance testing.

The instability of chrysotile can be a confusing factor in its identification and quantitation. Werner and Geissler (A38) showed how to identify thermally transformed chrysotile in dust from work environments. They used optical microscopy, X-ray diffraction, and infrared spectroscopy to study forsterite, the end product in high-temperature dehydration of chrysotile. Some attention was also paid to characteristics of annealed chrysotile and its semitransformed congener.

A fluorescence staining technique, possibly specific for chrysotile, is outlined by Albright and co-workers (A39) using Morin (2',3',4',5,7-pentahydroxyflavone) a classic spot test reagent.

B3. Miscellaneous Analytical and Microchemistry. Silcox and fellow workers (B31) formulated fundamental research problems and opportunities involving microchemical

and microstructural characterization of interfaces. They review aspects of solar energy technology related to these suggested new areas of endeavor.

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Nucleonics

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This has been a quiet 2 years in nucleonics, our home place. No catastrophic disasters (nuclear or other) have engaged our attention since November 81 through November 83. Instead, the continuing erosion of confidence in nuclear technology has been accompanied by a similar erosion of preeminence in nuclear analytical techniques. Newer methods such as the inductively coupled plasma (ICP), lasers, and variations on the mass spec theme such as resonance ionization mass spectrometry (RIMS) continue to push nuclear techniques aside and to claim analyses that were once the exclusive province of NAA. Electronics and computer modeling have shoved tracer techniques into the background. Coupled to all these pushings and shovings of nonnuclear accomplishments has been the thrust of environmental fear-mongers who have equated everything nuclear with disaster. The present prosperous state of nucleonics, given these hindering actions, is a tribute to those who practice it. For nucleonics indeed still lives. There are some new aspects that we discuss below and the books, data compilations, and conference proceedings still flow; these latter items are listed as usual in the tables. We have mentioned in previous reviews that one will most probably find the "state-of-the-art" at a timely scientific meeting. Thus, we spend a fair amount of time looking at such meetings. Unfortunately, we find that conference proceedings often follow the conference from one to more than three years! This makes it very difficult to correlate the meeting with the printed output. Another complication is that these proceedings appear in a variety of formats: special journal editions, in-house or technical society reports, or conventional book form. Often, we do not recognize that the information is really a proceedings. Therefore, in this review we have placed less emphasis on the source of the information; we prefer to let each entry speak for itself. Also, we have dropped our table of meetings since this listing did not seem to add materially to the quality of the review.

The number of publications that contain a nuclear flavor is prodigious. Therefore, as in past reviews, we have limited our table entries to those items that are of most interest to the analytical chemist. Physicists and nuclear engineers had best search additional sources. As usual in the body of the text, we have tried to indicate trends by referencing one or two representative papers, innovative or potentially useful new techniques are given a bit more description.

ACTIVATION ANALYSIS

We have found only one really innovative idea in activation techniques this period: Ghannam, Loveland, and Baum-

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gartner (1) report on the use of stable activable tracers to mimic the chemical behavior of toxic organic molecules. They used tris(dibenzomethane)dysprosium(III) monohydrate and the triacetylacetonate trihydrate to substitute for such compounds as toxaphene, dieldrin, etc. Neutron and charged particle activation analysis continue to be applied, however, to many types of samples. As investigators press for ever greater sensitivity, separations are needed. Removal of sodium on Kryptofix 221B polymer (2), column electrolysis on a carbon-fiber electrode for trace elements in seawater (3), preconcentration on activated charcoal (4), coprecipitation with lead phosphate (5), preconcentration on reagent loaded and unloaded polyurethane foam (6), and, in a search for organic halides, extraction with hexane (7) are some of the techniques reported.

Charged particle and proton activation and proton induced X-ray emission (PIXE) continue also to be used. Determination of fluorine in food (8) and metallic impurities in nio-