

STUDY OF MINERALS AND ARTIFICIAL MATERIALS IN POLISHED SECTIONS ON THE MICRO SCALE

The Third Annual Regional Conference, 2-3 May, 1969

PAULY, H. *et al.*: Study of minerals and artificial materials in polished sections on the micro scale. Third annual regional conference, 2-3 May, 1969. Abstracts of twenty one papers delivered at the conference. *Bull. geol. Soc. Denmark*, vol. 19, pp. 319-340, December 17th, 1969.

A joint meeting between the Société Minéralogique de France and the Mineralogical Society of London was held in Paris in April, 1965 and a similar meeting, organised by the Dutch Mineralogists' Association and the Mineralogical Society of London, took place in Amsterdam in early April, 1968. These meetings turned out to be more than discussions restricted to participants from the organising bodies and could rather be regarded as deliberations by a group of mineralogists, from several countries of the region, with a common interest in the theme of the meetings: *Reflected light microscopy with all its physical and chemical ramifications.*

The very high rate of development of instruments for the study of materials on a micro scale during the past decade has created difficulties for the individual research worker. Not only does he require to keep informed about the availability and applications of the new equipment; he feels the need to discuss his routine employment of the instruments with other users. The joint meetings offered excellent opportunities to meet these needs and also clearly demonstrated that scientific progress in the various fields might result from such meetings.

The organisers of the Amsterdam meeting, S. H. U. Bowie, N. F. M. Henry and W. Uytenbogaardt, proposed that a follow-up meeting be arranged to take place in Copenhagen in 1969. The proposal was accepted by the Mineralogical Petrographical Club of the Geological Society of Denmark and Hans Pauly was appointed Danish organiser. The organisers decided to try to establish these meetings on a formal basis and the meeting in Copenhagen was therefore designated the *Third Annual Regional Conference*, as it was considered reasonable to hold the meeting as a regular annual event. This view was accepted by the participants and it was with pleasure that the assembly accepted F. Trojer's invitation to hold the Fourth Annual Regional Conference at Leoben.

The venue for the first sessions was the Technical University of Denmark where the meeting was held under the auspices of the Mineralogical Institute of the University. On the second day the meeting

was held at the Mineralogical Museum of Copenhagen University under the auspices of the University Geological Institutes.

Eighty participants, twenty from Denmark and the rest from fourteen different countries, attended the Conference. "Polishing procedures" was chosen as a special topic and a supporting exhibit of grinding and polishing equipment was provided by the manufacturers, the Danish companies A/S Scandia and H. Struers Chemical Laboratories.

Twenty one papers had been submitted and abstracts were prepared and distributed to participants before the meetings. The abstracts are appended to this report.

The programme for the first day was opened by welcoming addresses by the head of the Technical University, E. Knuth-Winterfeldt, and the president of the Danish Geological Society, Tove Birkelund. Arne Noe-Nygaard gave an opening lecture on the development of geology in Denmark since Niels Stensen's famous work in 1669.

Topics discussed on the first day were reflectivity standards and polishing and preparation procedures. The session ended with a discussion on the feasibility of establishing a group to discuss the complex field of opaque optics and the assembly decided to form a working group open to those who feel they can contribute to and promote understanding and systematisation within this field. The Conference Dinner was held on the evening of the first day.

The second day started with a welcome from the head of the Geological Institutes, A. Berthelsen. This was followed by discussions on minerals and materials and, finally, on new methods in the study of polished surfaces.

After the discussion on future arrangements, in which F. Trojer invited the next Conference to Leoben for the 16th and 17th April, 1970 a closing reception was held in the Mineralogical Museum.

This provided an opportunity to thank the companies A/S Aalborg Portland Cement, Scandia, H. Struers Chemical Laboratories, and Haldor Topsøe for their valuable financial support to the Conference.

*Hans Pauly
Mineralogical Institute,
Tech. Univ. of Denmark,
Building 204, Lyngby
25th August, 1969*

Observations on SIKA Standards

N. F. M. Henry and A. H. v.d. Veen

The SIKA (SiC) standards at present in use have been subjected to a detailed examination. The required condition is stringent, because the area of macroscopic calibration is near 300,000 times greater than the 5 micron diameter area used for accurate microscopic measurements of reflectivity.

A proposal will be put forward to C.O.M. (Commission on Ore Micro-

scopy—International Mineralogical Association) for a control procedure with the aim of improving still further the standards for reflectivity measurements. Improved polishing and selection of material is necessary.

The influence of Oxide layers on R % of Si and ellipsometer measurements on Si

Knud Rosendal

On the basis of a plane boundary model an expression for the reflectance of silicon covered with a SiO₂-layer is derived. From reasonable estimates of optical constants the reflectance has been calculated as a function of layer-thickness for various wavelengths in the visible spectrum.

The use of ellipsometry for the determination of the real and imaginary part of the optical constant of silicon is briefly reviewed, together with some experimental results obtained by R. J. Archer, using $\lambda_{vac} = 5461 \text{ \AA}$, leading to the conclusion of a slightly lower reflectance at this wavelength, than apparently believed at present.

$$R \% = 36.50 \pm 0.15 \% \text{ at } \lambda = 5461 \text{ \AA}$$

An attempt to produce »independent« reflectance references from SiC, Si and Hg

Hans Pauly

Diamond polished basal plates of SiC, finished with MgO in water can be used as reference taking the reflectance as reported in literature or calculated from the refractive indices likewise to be found in literature, e.g. N. W. Thibault 1944 (Am. Min. vol. 29, p. 327). The SiC should be inspected for internal reflections and only material free of internal reflections should be used. In well polished specimens the R % will probably be less than 0.1 % below the theoretical value.

Si polished in various laboratories and with different methods were found to give exactly the same R-values at various wavelengths. It is therefore considered a nice material for a standard, contrary to what was reported to C.O.M. last year. We have, however, found that Si when insufficiently polished gives higher reflectance values than the well polished material. Here may be the reason for the unreliability of Si pointed out last year.

A well polished Si slice was reground with diamond paste so as to produce zones with scratches of varying coarseness corresponding to 7 microns, 2.5 microns, 1 micron, and 0.5 micron. Measuring these zones we found the following R-values at 546 nm using the C.O.M. value 37.0 % as a basis:

42.5 %	43.0 %	41.8 %	39.5 %	and 37.0 %
7 μ	2.5 μ	1 μ	0.5 μ	0 μ

The dispersion curves were parallel apart from that for the 7 microns zone which shows a drop in the blue end.

We have no explanation for this phenomenon. It may be related to the high refractive index, 4.05, of Si. In thin plates, less than 4 microns thick, Si is translucent with a golden brown colour.

Sphalerite, CdS, SiC, hematite, and galena did not show this peculiar phenomenon. They gave lower R-values when not sufficiently polished.

The increase in polish of Si after the $\frac{1}{2}$ micron diamond stage through repeated MgO stages can be followed as a fall amounting to about 0.2 % in R-value. This may be used as an indicator of or a check on the polishing.

Mercury – double distilled chemically pure (for Polarography) – was, after filtering through a bamboo cane, found to give

λ nm	437	481	546	590	644
R %	78 \pm 1	78.0	78.1	78.0	77.5

Measured against a Leitz silverbacked glass, uncorrected.

The same values were found on natural mercury from Alpi Apuana in Italy. This was also measured after filtration through a bamboo cane. The Hg was placed in a tiny jar made from the bottom part of a sample glass tube.

The Lanham Specimen Changer Stage as an aid to precision in reflectance measurements

P. R. Simpson

The method for determining reflectance preferred by mineralogists involves the consecutive measurement of standard and unknown specimen under identical optical and electronic conditions. The Lanham Specimen Changer Stage is designed to increase the precision of measurement and to reduce the operator fatigue involved in this operation. It will fit on a standard microscope stage. There are facilities for the initial alignment of the specimen and standard in the correct position in the optical path and for interchanging specimen and standard into the optical path of the microscope. Specimen and standard are precisely returnable to the positions initially selected for measurement by means of a manually operated slide.

The Specimen Changer Stage works equally well for specimens measured in air or oil immersion and is therefore particularly well suited for the precise measurement of dispersion of reflectance (R), refractive index (n), absorption coefficient (k) and absorption index (kappa) in mineral specimens.

Data are presented here for R in air and oil, n, k, and kappa, in the wavelength range 400–720 nm for six minerals which were measured in this way.

Table 1 gives the best values for four standard wavelengths.

Table 1. Optical data at 4 standard wavelengths

COVELLITE		R%air	R%oil*	n	k	κ
o	470	13.40	3.60	2.02	0.46	0.23
	546	7.15	1.10	1.65	0.31	0.20
	589	4.20	1.40	1.38	0.32	0.24
	650	5.90	8.45	0.97	0.49	0.51
e	470	29.10	14.30	3.16	0.75	0.23
	546	23.70	9.90	2.86	0.39	0.13
	589	21.15	8.20	2.60	0.51	0.20
	650	23.00	9.75	2.67	0.64	0.23
STIBNITE						
a	470	44.40	29.60	3.18	2.30	0.72
	546	42.10	26.80	3.60	1.95	0.50
	589	40.45	25.25	3.58	1.77	0.48
	650	40.45	25.20	3.55	1.67	0.44
b	470	30.80	16.10	3.00	1.13	0.38
	546	31.10	15.20	3.20	0.92	0.28
	589	30.65	15.00	3.21	0.77	0.23
	650	29.35	14.70	3.20	0.65	0.22
c	470	52.60	38.80	2.80	3.02	1.09
	546	48.10	33.40	3.45	2.60	0.74
	589	45.25	30.40	3.48	2.30	0.65
	650	42.15	27.20	3.48	2.00	0.57
BOURNONITE						
a	470	36.30	21.85	2.78	1.78	0.65
	546	35.60	20.70	3.16	1.52	0.48
	589	34.70	19.55	3.42	1.22	0.36
	650	32.70	21.85	3.23	1.12	0.35
b	470	35.50	20.65	3.06	1.56	0.53
	546	33.95	18.90	3.28	1.24	0.38
	589	32.95	17.95	3.29	1.10	0.33
	650	31.65	20.65	3.21	1.02	0.31
c	470	37.65	22.85	2.99	1.80	0.60
	546	35.50	20.35	3.36	1.36	0.41
	589	34.25	19.25	3.33	1.20	0.37
	650	32.70	22.85	3.25	1.09	0.33
CHALCOCITE						
a	470	36.70	21.25	3.49	1.38	0.38
	546	33.35	18.25	3.39	1.04	0.32
	589	31.72	16.90	3.22	1.05	0.33
	650	29.62	15.35	2.97	1.07	0.36
b	470	36.70	21.25	3.49	1.38	0.38
	546	33.35	18.05	3.53	0.85	0.22
	589	31.82	16.90	3.28	0.91	0.28
	650	29.72	15.35	3.04	1.01	0.32

		R%air	R%oil	n	k	κ
c	470	36.72	21.35	3.49	1.38	0.38
	546	33.12	17.85	3.53	0.85	0.22
	589	31.47	16.45	3.35	0.80	0.24
	650	30.17	15.45	3.24	0.78	0.23
ARSENOPYRITE						
a	470	50.80	34.90	—**	—	—
	546	52.80	37.55	—	—	—
	589	53.20	38.50	—	—	—
	650	53.60	39.10	4.56	2.90	0.63
b	470	48.70	33.90	3.43	2.66	0.77
	546	51.85	37.35	3.76	2.93	0.78
	589	52.80	38.30	4.04	2.98	0.73
	650	52.95	38.90	3.51	3.08	0.87
c	470	51.85	36.95	4.19	2.82	0.67
	546	51.90	37.20	4.10	2.83	0.69
	589	51.70	37.05	4.12	2.82	0.67
	650	51.30	37.00	3.57	2.92	0.82
PYRRHOTITE***						
⊥ c	470	30.80	18.75	1.97	1.60	0.82
	546	34.75	22.20	2.04	1.81	0.88
	589	36.90	24.15	2.09	1.93	0.93
	650	39.50	26.75	2.08	2.08	0.99
≠ c	470	35.50	23.25	1.92	1.84	0.96
	546	39.90	26.80	2.13	2.08	0.98
	589	41.60	28.20	2.28	2.21	0.97
	650	43.25	29.90	2.32	2.32	0.99

* The immersion oil used is Cargille Type 'A' at 22.5°C.

** Values represented by a dash cannot be determined with sufficient accuracy by the method used in this work.

*** Pyrrhotite is the monoclinic variety, but the section normal to c is isotropic.

Preparation of polished sections adjusted to the modern demands of reflectivity testing

B. van de Pijpekamp

Two polishing procedures, depending on the hardness differences of the associated minerals and quality requested for accurate reflectivity measurements, were developed.

1. A long-run process which takes 20 running hours for obtaining high polish on 12 of the most difficult samples (grain samples of quartz-pyrite-sphalerite-chalcopyrite-galena). High polish is considered to show areas of at least 30×30 micron free of scratches and of other defects, tested by means of an interference objective $20 \times$. All other sections with minerals of diverging hardness can be polished in shorter time.

Materials used: For grinding emery 305 and borcarbide 0-2 and 2-4 micron on mirror glass plate laps (all laps 22 cm diameter) for coarse polishing 0-2 micron, 0-1 micron and 0-0.5 micron diamond dust on respectively lead laps alloyed with 1 %, 0.5 % and 0.25 % Sb. For fine polishing 0-0.25 and 0-0.1 micron diamond spray on lead laps with 0.25 % Sb. Final polishing alumina I (Depiereux) and/or MgO II (Depiereux) on pure lead laps. In all cases abrasive is applied once.

2. A short-run process which takes about 8 running hours for 12 of the same difficult samples. In principle we make in every stage demanded several (up to 4) runs of 15 minutes each. Each run takes place on 0.01-0.025 carat diamond powder. We are nearly ready to substitute the long run process by the short run process without loss of quality. See galena in the table.

Mineral	R % at 546 n. m.	Running hours
galena	43.8 max.	20}
chalcopyrite	49.0 max.	20}
galena	43.5 max.	8 (test grain sample)
arsenopyrite	51.0-51.5*	6}
bismuthite	37.8-50.2*	6}
cassiterite	10.9-12.4*	5.5}
wolframite	16.0-18.7*	5.5}
carborundum	20.3 max.	4
synthetic PbS	43.2 max.	0.75

Reflectively measured with a Leitz M.P.V.

* Burke, Amsterdam.

Impregnate by applying high vacuum technique and repeat this during the polishing process if necessary; use a heated centrifuge for getting rid of bubbles; grind briefly on abrasive not exceeding 10 micron and apply water (prevents impregnation failures); use straight edge for controlling relief; test your polish with an interference objective.

A quantified polishing scheme is proposed and it is shown that high quality polishing is not depending on the equipment used, but on the materials, quantification of handlings and accurate maintenance of the optimal conditions by the technician. His touch or feeling played hitherto an important role because of the lack or the negligence of information. Polishing must be a science rather than an art.

Some aspects of grinding and polishing ore specimens

J. J. Prins and H. J. Roorda

This paper reviews the results of recent studies directed at finding a simple and quick method for preparing polished ore specimens which would be representative of the true structure of the material at the plane of sectioning. In order to achieve this objective most of the grinding and all of the polishing was done with a continuously flowing

stream of water since it was suspected that conventional methods of polishing with oil resulted in a loss of information.

The following minerals were examined: cassiterite, pyrite, galena and chalcopyrite.

1. *Grinding by hand on a glass plate with 600 and 1000 carborundum.* This type of grinding using water results in a satisfactory surface in a short time (1–2 minutes). Although not perfectly flat, the surface is suitable for the next step.

2. *Mechanical grinding on a revolving cast-iron disc with 1000 carborundum.* Since water resulted in excessive friction, fine machine oil was used at a rate of about 10 drops per minute after a paste of oil and carborundum had been rubbed onto the disc. After about 15 minutes the surface is inspected for flatness and if satisfactory, ready for polishing.

3. *Polishing with diamond paste on a revolving pella disc.* Since tests on metal discs were not successful, subsequent experiments were made with 6 inch pella discs rotating at 150–200 rpm using water as a lubricant. In order to ensure a continuously flowing film, a wetting agent, Aerosol OT (dioctylsodium sulfosuccinate) was added at a rate of 20 drops per liter of water. The weight on the specimen ranged from 250 to 500 grams and did not seem to affect the final results.

After polishing with 6 micron diamond paste we found:

- a. A high degree of flatness was necessary to ensure polishing over the whole surface, so control of the flatness must be done before polishing in this way to obtain satisfactory results.
- b. When using conventional diamond paste, a cake is formed on the pella disc from grit and oil which hinders the polishing and causes relief. With water dissolving paste, this does not happen as the water washes nearly all of the grit from the disc and no cake is formed. The relief is very small.
- c. Polishing with water produces pittings which vary with crystal planes and different minerals. There are minerals with a strong tendency to pitting (chalcopyrite) and others, galena and cassiterite, which do not have this tendency. The pitting is not a relict of the grinding procedure, but is produced by this polishing method. Due to these pittings the crystal plane best suited for hardness tests can be selected and less tests are necessary to obtain a reliable hardness figure. It also shows when unreliable hardness figures can be expected.
- d. Already after 15 minutes satisfactory polish can be obtained.
- e. Due to the continuous water addition and with the aid of a time clock no supervision is required during the polishing.
- f. The "theory" of the melting of sulfides and filling up of pits and holes can not be applied to this polishing method. No smearing and disappearing of cracks takes place and this surface gives special information about the real condition of the ore.
- g. Holes, pits and cracks are not filled with grease, grit and diamonds and do not interfere so strongly with the polishing.

h. The discs can be cleaned from dust etc. by washing under the water tap.

After using 6 micron the polishing was continued with 1 micron diamond paste and water as lubricant.

This improved the surface very much after 15–30 min. polishing. Continuing with polishing on $1/4$ and $1/10$ m μ paste did not give much improvement.

However final polishing can be done better with γ -alumina and water.

The preparation of refractory materials for examination in reflected light

Frank Rutter

It is shown that a satisfactory polish can be obtained on refractory material specimens only after the complete impregnation of the pores in the material.

A variety of impregnating compounds are assessed and it is found that the non-wettability of carbon-containing refractories requires special treatment by a high vacuum technique.

All refractory materials can be quickly and successfully prepared with diamond polishes although other media are discussed.

Other specialised techniques including deliberate relief polishing and chemical etching are illustrated.

New data on the optical properties and micro-hardness of galena, bornite, pyrite, chalcopyrite and magnetite

K. v. Gehlen and K. Hausmann

Selected "best" values obtained with a Zeiss MPM microscope photometer and a Zeiss MHP microhardness tester are summarized in the following table:

Mineral	Vickers		wavelength nm	Reflectance values	
	hardness (100 p) kp/mm ²	hardness*) kp/mm ²		in air %	in oil %
Bornite	100	103	470	16.6	5.7
	95–105	100–106	546	18.5	7.7
	sf		589	21.5	10.2
			650	25.8	14.1
Chalco- pyrite	195	203	470	37.3	27.9
	185–205	193–213	546	48.3	36.5
			589	49.8	37.3
			650	49.6	36.4
Galena	70–81	74–84	470	45.5	30.1
	66–85	70–88	546	42.4	29.3
			589	41.8	26.5
			650	42.1	26.6

Magnetite	534-577	538-581	470	19.7	8.0
	504-607	508-611	546	19.8	8.0
	sf		589	20.0	8.1
Pyrite (111)	1334	1262	470	46.5	32.8
	1254-	1186-	546	54.0	40.2
	1414	1337	589	55.4	41.6
	f		650	55.9	42.4

It was found to be more reasonable physically to perform a series of hardness measurements at different loads (below the fracture load) and to derive the Vickers hardness from the slope of the straight line connecting the data in a d^2/P plot. These Vickers hardness values are given above in the third column marked by *).

On the bireflectance of the mineral loellingite

O. Vaasjoki

The spectral reflectance for R_1 and R_2 of an anisotropic mineral commonly shows that the difference $R_1 - R_2$ does not change the sign if considering the respective values at differing wavelengths. There are cases, however, when the sign for $R_1 - R_2$ may change for a mineral section if differing wavelengths have been applied. Such a situation has been demonstrated *e.g.* for the mineral mawsonite (Cervelle *et al.* 1968: Sur l'inversion du signe de la biréflexance de certains minéraux absorbants: cas de la mawsonite. *Bull. soc. française Min. et Crist.* 91).

In the case at hand the spectral reflectance of the mineral loellingite

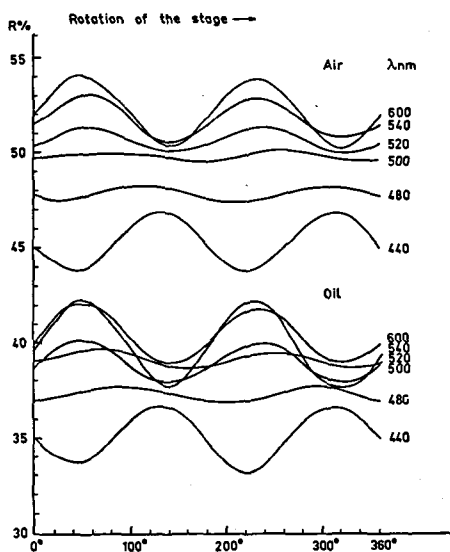


Fig. 1

has been determined by measuring the values R_1' and R_2' corresponding to the directions of the maximum and minimum reflectance in sections at random orientation (fig. 1 and table 1).

It appeared that at wavelength 440 nm for the loellingite R_1' is greater than R_2' , whereas at wavelength 600 nm the relations are reversed. R_1' equals to R_2' at about 490 nm and at this wavelength the spectral dispersion curves for R_1' and R_2' of the loellingite reveal a common point (fig. 2).

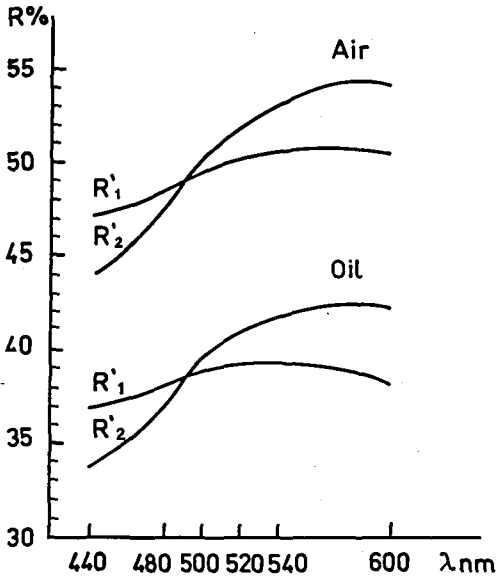


Fig. 2

The phenomenon as observed might be considered as a new optical criteria in separating loellingite, say, from arsenopyrite, which does not reveal a common point with regard to the spectral dispersion curves of the reflectance. As the measurements here have been carried out on sections at random there has not been sufficient evidence for further optical interpretation of the phenomenon as observed.

Table 1. Values of reflectance based on fig. 1

λ nm	Air		Oil	
	R_1	R_2	R_1	R_2
440	46.9	43.8	36.7	33.5
480	48.3	47.4	37.9	36.9
500	49.5	50.1	38.8	39.7
520	50.0	51.3	38.8	40.7
540	50.6	53.0	39.0	41.9
600	50.2	54.0	38.0	42.2

For the measurements has been used the U.S.S.R. pyrite standard donated by the Dept. of Mineralogy and Petrology, University of Cambridge, England.

Studies on some Norwegian ilmenites and pyrophanites

F. M. Vokes, S. Bergstøl and Tore Vrålstad

The results to date of a continuing investigation of ilmenites and pyrophanites from Norwegian localities are shortly presented. Values of R_E and R_O at a wavelength of 542 nm have been determined on oriented polished sections of crystals, while uniaxial reflectivities have been determined on ore specimens.

The chemical compositions of the measured specimens have been determined by means of an ARL EMX electron microprobe using analysed mineral standards and metal standards with programmed corrections, mainly after Springer.

The investigations do not indicate any systematic variation of the two reflectivities with variation in chemical composition, at the wavelength used. Variations in MnO contents of between 0.6 and 3.2 % in ilmenites and up to 35 % in pyrophanites, and in MgO contents of between < 1.0 and 5.00 % in ilmenites have no appreciable effects on the reflectivity values.

The reflectivity measurements show that values of R_O measured on oriented specimens perpendicular to the c-axis are always up to 0.5 % lower than those measured parallel to the c-axis.

The spectral reflectivity of analysed chromites from the Uckopru Mine Area, South West Turkey

T. Engin and R. Phillips

34 separated chromite specimens covering the range 40–57 % Cr_2O_3 have been analysed by XRF and their spectral reflectivities measured. The relationship of reflectivity to composition was discussed.

Optical characteristics of some opaque minerals

A. Lopez-Soler and J. M. Bosch-Figueroa

In the present work we have measured reflectance $R\%$ on polished sections cut parallel to the faces that contain the principal directions of vibration in the following minerals: zinckenite ($PbSb_2S_4$); hematite (Fe_2O_3); and enargite (Cu_3AsS_4).

Data of reflectance have been measured in air and in immersion oil Cargille type A at 20°C, using silicon carbide as standard. Dispersion curves of $R\%$ in air and in oil for each principal direction of vibration for every mineral are given from 440 nm to 660 nm.

The physical constants, complex refractive index, coefficient of absorption and absorption index for every wavelength and the dispersion curves for each constant are given.

Hardness has been measured in micro-indentations by Vickers method and in the same polished sections in which reflectance was measured. Minerals studied have been identified by X-Ray diffraction.

Optical data of Zinkenite, PbSb₂S₄, Hexagonal

λ	Ordinary vibration				Extraordinary vibration			
	%R air	%R oil	ω	k_o	%R air	%R oil	ϵ	k_e
470	38.90	23.36	3.59	1.56	44.63	28.80	4.40	1.62
546	37.67	22.20	3.70	1.29	42.66	27.09	4.07	1.64
589	36.76	21.29	3.72	1.11	41.38	25.96	3.86	1.65
650	35.10	19.75	3.64	0.95	39.33	24.23	3.48	1.69

Optical data of Hematite, Fe₂O₃, Rhombohedral

λ	Ordinary vibration				Extraordinary vibration			
	%R air	%R oil	ω	k_o	%R air	%R oil	ϵ	k_e
470	31.61	16.71	3.08	1.17	28.10	13.46	3.01	0.81
546	30.03	15.18	3.19	0.83	26.50	12.04	3.05	0.44
589	28.93	14.30	3.11	0.78	25.31	11.09	2.99	0.31
650	26.55	12.23	3.05	0.47	23.61	9.79	2.88	0.15

Optical data of Enargite, Cu₃AsS₄, Orthorhombic

λ	Vibration parallel to (001)			
	%R air	%R oil	$n_{(001)}$	$k_{(001)}$
470	29.22	14.17	3.23	0.61
546	28.72	13.96	3.16	0.65
589	28.66	14.00	3.14	0.68
650	28.36	13.77	3.14	0.63

λ	Vibration parallel to (010)			
	%R air	%R oil	$n_{(010)}$	$k_{(010)}$
470	26.91	12.08	3.15	0.10
546	25.88	11.50	3.03	0.35
589	26.07	11.92	2.93	0.64
650	28.07	13.55	3.11	0.64

λ	Vibration parallel to (100)			
	%R air	%R oil	$n_{(100)}$	$k_{(100)}$
470	27.42	12.52	3.18	0.20
546	25.15	10.89	2.99	0.26
589	24.41	10.60	2.82	0.57
650	25.67	11.62	2.92	0.59

Vickers hardness measurement in oriented sections

Load: 100 ponds.			
HEMATITE	Section (0001)	974-1018	
	» (1010)	974-1033	
	» (1011)	1033-1064	974-1064

ZINKENITE	Section (0001)	176- 185	
	» (10 $\bar{1}$ 0)	175- 181	175- 185
ENARGITE	Section (001)	292- 304	
	» (110)	240- 327*	240- 327

* This section has a strong anisotropy for hardness, visible on the microindentations form.

This work was supported in part by a grant from the Fundacion Juas March of Spain.

Staringite, a new Sn-Ta-mineral from North-East Brazil

E. A. J. Burke, C. Kieft, R. O. Felius, and Maria S. Adusumilli

This paper will be published in *Min. Mag.* Vol. 37, no. 288, 1969.

Distribution of impurities in minerals made visible through cathodoluminescence

G. Remond and Ph. Perrot

The authors call attention to the absorption phenomena which impose a threshold to the detection varying with element and sample in the quantitative analyses with the electron microprobe.

Realising the various difficulties in making quantitative analyses with the microsonde, especially those connected with absorption phenomena and those connected with the nature of sample and the nature of the reference, the authors underline the seriousness of these factors when one wishes to determine elements present in very small amounts.

The presence of impurities in concentrations below this limit of detection is however still sufficient to cause certain minerals to emit light when they are irradiated with an electron beam.

Several techniques are proposed to register the variation in this cathodoluminescence and to make visible the often heterogeneous distribution of the trace elements in minerals.

We have undertaken a first series of observations using a microsonde. Irradiating a surface several hundred microns in diameter we obtained, on placing a photographic film at the ocular of the microscope, a "map" giving the variations in luminescence.

Qualitative point analyses have permitted the association, in some cases, of the colour of the luminescence and the presence of certain trace elements. In order to create or modify these emissions it is however sufficient that the elements are present in very small concentrations, often below the limits of detection with the microsonde. We have concentrated on determining the spectra of the luminescence in order to identify the trace elements by comparing the wavelengths of the emis-

sions in the samples and in references containing a certain number of impurities.

The intensity of the cathodoluminescence is increased with the energy and the density of electrons falling on the surface of the mineral: Further in order to register the very faint light flux we have used a technique where a minutely focused electron beam sweeps the surface of the sample. The cathodoluminescence image is made visible on the screen of an oscilloscope through a photomultiplier.

Concluding we wish to underline that the possibilities of quantitative determination of trace elements are limited, partly due to the limitation in the microsonde and partly due to the uncertainties in the corrections of the matrix effects. The cathodoluminescence allows a speedy detection of the distribution of impurities in monocrystals.

The method used actually permits the registration of very faint variations in the light emission. But the method is in fact only of a descriptive character because, apart from a few cases, the point analysis by the microsonde has shown to be insufficient for the identification of the nature of the impurities responsible for the emission of luminescence.

Alkali-silica reactions in concrete examined by optical microscopy and electron-probe micro-analysis

Lars Hjorth and Niels Thaulow

Concrete is made by mixing cement, water, and aggregates. During the reaction between cement and water (hydration) the cement constituents dissolve releasing among other things K^+ , Na^+ , Ca^{2+} , and OH^- . These ions may react with some of the silica-containing aggregates. The reaction product formed may be an alkali-silicate-hydrate which behaves like a gel. Swelling of such gels now and then causes deterioration of concrete structures.

In order to study crack distribution and signs of deterioration within a concrete specimen, which is representative for the concrete of a structure, large thin-sections (for instance $100 \times 50 \times 0.02$ mm) are examined by optical microscopy. The thin-sections are made by vacuum-impregnation of the specimen with an epoxy resin followed up by a combination of mechanical cutting and polishing and hand-polishing.

Examinations of such thin-sections often show the presence of an isotropic material placed in cracks, voids, and close to silica-containing aggregates. This product is often considered to be the alkali-silica-gel.

In the present case it was essential to know whether this isotropic material found in a thin-section in fact was alkali-silica-gel or something else (such as for instance magnesium hydroxide or some organic material). A large thin-section was made without cover-glass. A typical area containing the isotropic material was found and the area was cut out of the thin-section. This small thin-section was polished and examined by

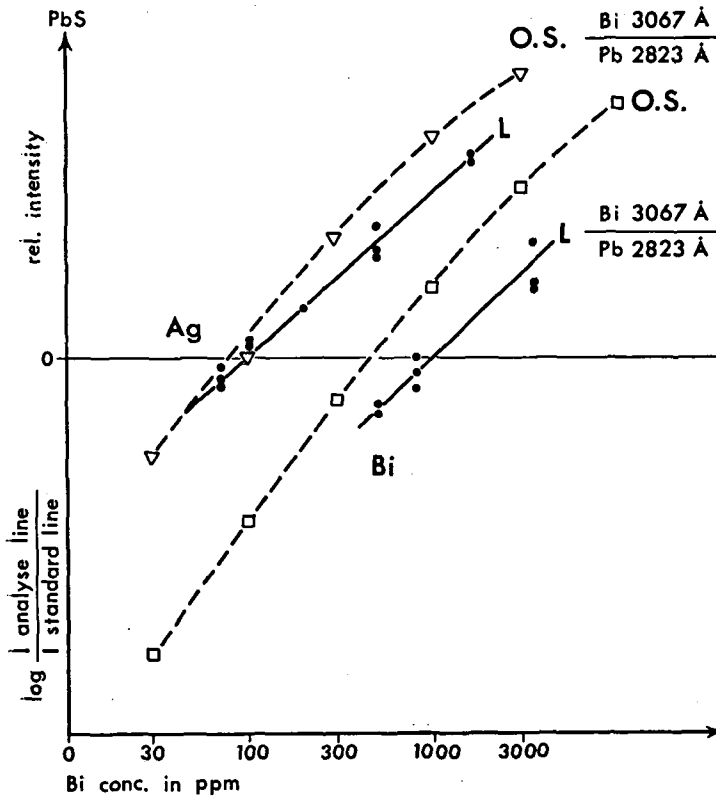
an Electron-probe Micro-analyser. It was found that the material consisted of potassium, calcium, and silicon whereas no sodium and no magnesium were found present. Two types of gel were seen. One low-potassium yellow-coloured type and one high-potassium uncoloured type of gel. By a special colour filter technique a colour photograph was prepared showing the distribution of calcium, silicon, and potassium.

The Laser-microprobe as excitation source in emission spectrography

Haldis J. Bollingberg

A study of the quantitative estimation of trace elements in galena, sphalerite and some silicate minerals has been made by using a neodymium laser excitation source.

The latest type of Jarrel-Ash Lasermicroprobe is used in conjunction with a Hilger Large Quartz spectrograph.



The method is characterised by the vaporisation of the material by means of a focused discontinuous laser beam. The atomised material rises vertically from the sample between two graphite electrodes placed 1–2 mm above the sample, where an auxiliary spark excites the vaporised atoms.

Spectra are recorded photographically and interpreted according to normal spectrographic procedure.

The reproducibility and detection limits establish that at least semi-quantitative analysis can be carried out with the laser-microprobe.

Material consumption in this work has been 6–120 μg corresponding to crater dimensions of 200–400 μm . Major elements and some especially sensitive trace elements can be detected with a material consumption of 0.1–5 μg , corresponding to crater dimensions of 60–120 μm .

Simple calculations show that the detection level is in good agreement with usual emission spectrography.

The example in the figure shows the actual working-curves for Ag and Bi in galena by laser (L)—and arc—(O.S.).

Texture analysis of fine-grained silicate rocks in polished section by optical and electron microscopy

R. Blaschke

The limits of quantitative texture analysis of silicate rocks are demonstrated by micrographs of very fine-grained basalt made by different optical and electron microscopes.

Minimal grain size of silicate mineral allowing quantitative texture analysis by such parameters as specific surfaces or area percentage:

Type of microscope	Specimen	Minimal grain size
A. Optical Microscopy		
1. transmitted light (polarising microscope)	thin section 5–20 μm thick	about 50 μm (limited by thickness of specimen layer)
2. reflected light (vertical illumination)		
a) dry objectives	polished section	(limited by low contrast)
b) oil immersion objectives	»	different, about 20 μm (limited by internal reflection, low contrast, low reflection power)
c) low refraction liquid immersion under crossed nicols after E. Stach	»	about 5 μm (limited by minerals of low refraction index)
d) high refraction liquid immersion under crossed nicols after E. Stach	»	about 5 μm (limited by minerals of high refraction index)

B. Electron Microscopy

1. scanning electron microscope

a) reflected electrons	polished section metal coated 100 Å or more	about 0.5 μm (limited by low electron signal/noise ratio)
b) secondary electrons	polished section metal coated	about 0.1 μm (limited by quality of coating and signal/noise ratio)

2. photo emission electron microscope

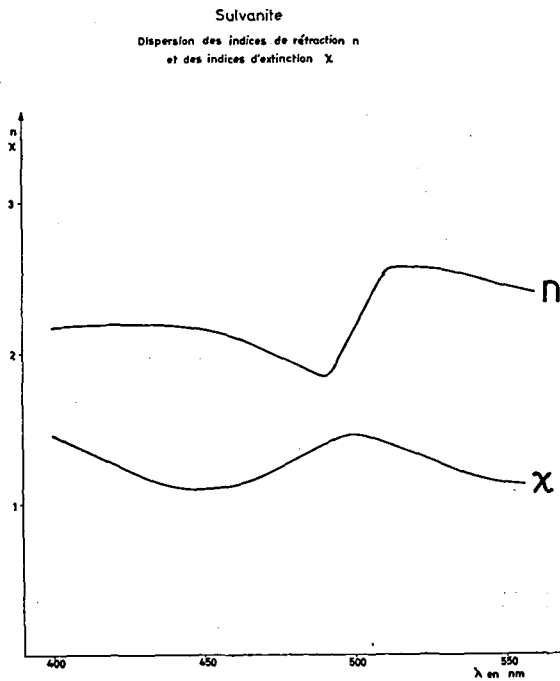
polished section of highest quality	about 0.05 μm (limited by resolution (100 Å) and specimen surface)
--	---

Study of an absorption band in the opaque mineral sulvanite by reflectance measurements

C. Lévy and R. Caye

The dispersion curves of reflectivity of sulvanite present some very important and sharp variations.

It was possible, after having measured the reflectivity in two immersion media, to calculate the variation of the refractive and extinction indices.



The results obtained by the measurement of the reflectivity in air ($N = 1$) and an immersion oil ($N \sim 1.5$) showed that the dispersion curve of the refractive indices presented the characteristic anomalies of a selective absorption band.

New measurements made, especially in another immersion oil ($N \sim 1.6$) in order to obtain more precise values gave no reproducible results for the refractive and extinction indices. For this reason, it was not possible to establish the quantitative relation linking these two indices in this interesting case, where a mineral presents simultaneously metallic absorption and selective absorption.

Nevertheless, even if the absolute values of the refractive indices for various areas vary, the existence of the absorption band is demonstrated by the fact that, on all the dispersion curves, the absorption band appears centred on the same wavelength and with the same limits.

The origin of the variation of the reflectivity for the various areas can be explained by the heterogeneity of natural sulvanite. It will be necessary to obtain synthetic sulvanite to determine precisely the interesting optical properties of this mineral.

The study of opaque minerals in transmitted infrared light

B. D. Cervelle

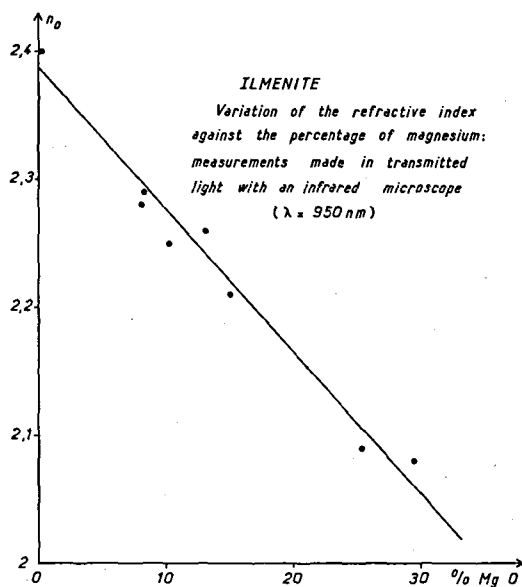
When the opacity of a mineral in the visible spectrum is caused by the existence of a selective absorption band (linked electron layer in resonance), it is possible to eliminate the absorption by changing the wave-length range, for instance into the near infra-red. It has been noted that a certain number of sulphides and sulphosalts are transparent in the infrared at a wavelength of about 1000 nm.

On the initiative of professor Beugnies an apparatus was specially designed for the microscopic examination of ore minerals in transmitted infrared light. This consists essentially of a polarising microscope fitted with nicols, instead of polarising film, and carrying an infrared image-converter. This allows observations to be made on a fluorescent screen of the image produced by the infrared rays transmitted by minerals that are opaque in the visible.

This method has already been applied in Belgium to the columbite-tantalite series and in France to the members of the ilmenite-geikielite series, and refractive-index measurements that have been made by means of the Becke line. One difficulty is that the minerals that can be studied in this way have very high refractive indices (over 2) and so the usual measurement accessories cannot be used, particularly the hemispheres of the universal stage, as their refractive indices are too low for the purpose. It is hoped to overcome this difficulty by replacing the glass hemispheres by silicon hemispheres having a refractive index of 3.6.

It appears that the systematic use of the infrared microscope must lead to a more complete knowledge of the optical properties of ore minerals. At the same time it will provide a method for measuring the

effect of the polishing process on surfaces used for reflectivity measurement since this does not enter into measurements made in transmitted light. Finally, from a theoretical point of view, it should be possible to study the absorption bands and their influence on the optical properties of minerals by the combined use of reflected- and transmitted-light and of visible- and infrared-light.



The application of stereology to ore microscopy

G. C. Amstutz and H. Giger

The intergrowth of minerals determines technical problems such as grain liberation for flotation. It is also the basis for paragenetic studies. With the aid of stereology, i.e. with integral geometry, these problems can be approached quantitatively.

The locking index, the surface density and the matrix of coordination provide the essential information on the metric and topological properties of rock and ore textures. These values are introduced and illustrated with actual examples. In addition, directional properties of a fabric can be obtained simultaneously, yielding results which are comparable to those obtained by methods of petrofabric analysis.

Relations between reflectivity, microhardness and elementcontents regarding gersdorffite and ullmannite

Hermann Gies

The minerals gersdorffite (NiAsS) and ullmannite (NiSbS), according to microprobe-analyses, differ strongly in their contents of accessory elements. Whereas ullmannite appears practically homogeneous, gersdorffite mostly shows varying contents of Fe and Co. This also is the case in intergrowths with ullmannite in the so-called "korynite".

It was found that gersdorffite in its purest form has a reflectivity value of $\pm 46.6\%$ and a microhardness of ± 580 VHN. These values go up to 48.2% and ± 715 VHN with increasing Fe/Co contents in zoned parts (1 % Fe, 1 % Co). Extreme reflectivity values from $\pm 49.4\%$ and ± 770 VHN of microhardness were found in a particular Co-rich variety (Fe = ca. 1 %, Co = ca. 6.5 %). This gersdorffite mainly appears in the final stage of mineral crystallisation as marginal banding of grains or as veinlets and therefore preliminarily was named "Spätphase".

The ullmannite in all cases practically was free from Fe and Co (Bi < 0.5 %). This stoichiometric "purity" also appears in the close agreement between the values obtained on natural and synthetic (fused!) mineral, particularly concerning the reflectivity.

Mineral	Reflectivity (546 nm)	Microhardness (30 Pond)
GERSDORFFITE:		
a) groundmass ± NiAsS	46.6 % (46.4–47.2)	± 580 VHN (524–630)
b) Zonary arrangement ca. 1 % Fe, 1 % Co	48.2 % (47.3–49.0)	± 715 VHN (687–735)
c) "Spätphase" ca. 1 % Fe ca. 6.5 % Co	49.4 % (48.0–49.7)	± 770 VHN (752–788)
ULLMANNITE:		
a) natural, ± NiSbS	47.5 % (47.2–47.7)	525 VHN (458–591)
b) synthetic, NiSbS	47.5 % (47.0–47.8)	535 VHN (505–568)

Considering the strong influences of chemical variations on reflectivity and microhardness, no standard deviations are given.

Dansk sammendrag

Société Minéralogique de France og Mineralogical Society of London holdt i april 1965 et fællesmøde i Paris. Et lignende møde afholdtes i Amsterdam i april 1968 mellem Mineralogical Society of London og sammenslutningen af hollandske mineraloger. Disse møder påkaldte sig interesse langt ud over de af organisationslandene bestemte grupper, idet der kom deltagere fra mere end en halv snes lande. Interessen for drøftelser af reflexionsmikroskopi og de dermed forbundne fysiske og kemiske undersøgelsesmuligheder er øjensynlig stor.

Udviklingen af nye instrumenter er gået meget hurtigt inden for denne sektor. Den enkelte forsker har derfor et behov for oplysninger om de til rådighed værende instrumenter og om deres anvendelse. Det er værdifuldt at kunne diskutere dagligdags problemer vedrørende anvendelsen af de nye instrumenter ikke blot med leverandøren men også med andre brugere. Fællesmøderne gav glimrende muligheder for sådanne drøftelser og viste tydeligt, at det er befordrende for fremskridt indenfor flere områder af denne forskning, at man får direkte kontakt med hinanden.

Amsterdammødets organisatorer, S. H. U. Bowie, N. F. M. Henry og W. Uytendogaardt foreslog derfor, at man søgte at arrangere et følgende møde i København. Forslaget blev accepteret af Mineralogisk Petrografisk Klub, og Hans Pauly blev bedt om at forestå organisationen deraf. Drøftelser mellem organisatorerne viste, at der kunne være grund til at lade disse møder blive af mere varig karakter. Mødet i København kunne betragtes som det tredje møde i en konferencerække, man ville søge at gøre til en årlig tilbagevendende begivenhed.

På konferencens første dag mødtes man på Mineralogisk Institut, Danmarks tekniske Højskole, Den polytekniske Læreanstalt. Møderne den anden dag afholdtes på Mineralogisk Museum, Københavns Universitet.

Der deltog 60 personer fra 14 forskellige lande foruden en snes deltagere fra Danmark. Som særligt emne for denne konference havde man valgt »polerings teknik«, og i forbindelse hermed var der arrangeret udstilling af slibe- og polerudrustning fra firmaerne Scandia og H. Struers kemiske Laboratorier.

Der var anmeldt 21 foredrag, som i form af resumeer blev udsendt til konferencedeltagerne inden mødet.

Ved åbningsmødet bød rektor E. Knuth-Winterfeldt, Danmarks tekniske Højskole og formanden for Dansk geologisk Forening Tove Birkelund deltagerne velkommen. Arne Noe-Nygaard præsenterede i den første forelæsning udviklingen i dansk geologi, siden Niels Stensen i 1669 offentliggjorde de undersøgelser, der skulle blive fundamentet for de geologiske videnskaber.

I de følgende foredrag behandlede standarder til refleksionsmålinger og præparationsteknik ved fremstilling af polerprøver. Dagen sluttede med en diskussion om betimeligheden af at oprette en åben gruppe, hvis arbejdsområde skulle være at diskutere optiske forhold for uigennemsigtige stoffer. Om aftenen afholdtes en fælles middag for konferencens deltagere.

På konferencens anden dag bød A. Berthelsen deltagerne velkommen i de geologiske institutter, Københavns Universitet.

Der blev fremlagt resultater fra mineralogiske undersøgelser såvel som fra undersøgelser af beton og keramiske produkter. Videre beskæftigede flere foredragsholdere sig med nye metoder i forbindelse med studiet af polerprøver.

Efter foredragene diskuteredes den fremtidige form for aktivitet. Konferenceideen vandt almindelig tilslutning, og F. Trojer fremførte en invitation til at afholde den fjerde regional konference i Leoben, Østrig, den 16. og 17. april 1970.

Konferencen afsluttedes med en reception i Mineralogisk Museum, hvorunder der udtryktes en varm tak til de firmaer, hvis økonomiske tilskud havde bidraget til konferencens vellykkede forløb: A/S Aalborg Portland Cement, Scandia, H. Struers kemiske Laboratorier og Haldor Topsøe.

Sammendrag af de afholdte foredrag findes på side 320-339.