CRYSTALLOGRAPHY

1960

SovietRxiv

Abstract

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CRYSTALLOGRAPHY

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A NEW SPECTROPHOTOMETRIC APPARATUS FOR THE STUDY OF MICROCRYSTALS

(Presented by Academician N. V. Belov on 6 VII 1959)

Recently, work has been increasingly expanding on the study of the absorption spectra of minerals, making it possible not only to understand better the nature of the coloration of mineral substances, but also to facilitate their diagnosis. Thanks to the work of N. M. Melankholin, S. V. Grum-Grzhimailo, and others, numerous curves of spectral absorption have been obtained for various minerals and especially for micas. However, most of the published absorption curves have the drawback that the study of minerals was carried out in ordinary, nonpolarized light. It is known that for different directions of optically anisotropic crystals the spectral absorption curves have different forms. Therefore the absorption curves obtained for anisotropic minerals in ordinary, nonpolarized light are averaged and do not have the value possessed by curves obtained in the study of crystals in polarized light. Unfortunately, spectrophotometers and monochromators of the usual types, manufactured by our industry, are not provided with a special device that would make it possible to study crystals in polarized light. In addition, one can work with them only on crystals of considerable size, whereas in working with natural objects—minerals—crystals of very small size are, on the contrary, more often encountered. The existing model of the Berek polarization spectrophotometer (ˆ1) does not permit the study of very small crystals. For this reason, various laboratories have to construct special models of microspectrophotometers, which are available only in those laboratories.

The most convenient model for obtaining absorption spectra of microcrystals is the microphotometer of N. M. Melankholin and B. N. Grechushnikov (ˆ2). Both on this microspectrophotometer and on the Berek polarization spectrophotometer, absorption data can be obtained only after calculation by the formula $D = \lg \frac{I_0}{I}$, where D is the optical density of the crystal under study; I_0 is the intensity of the light incident on the crystal; I is the intensity of the light transmitted through the crystal. Such calculations take much time, especially in mass measurements. We assembled an apparatus that makes it possible to obtain relatively quickly the transmittance and absorption values of crystals measuring tenths and even hundredths of a millimeter, avoiding the calculations indicated above.

The assembled microspectrophotometric apparatus consists of the following

parts: a monochromator—the source of monochromatic light; an MF-2 microphotometer; an FEU-19 electron photomultiplier—the receiver of the monochromatic light; and a stabilized high-voltage rectifier VVS-1, supplying the FEU-19. As the source of monochromatic light, a UM-2 or ZMR-2 monochromator is used with the light-flux modulator switched off. The latter model of monochromator makes it possible to carry out automatic recording of spectra. In front of the entrance slit of the monochromator

A conventional incandescent lamp is installed in the UM-2. The voltage on the lamp is stabilized by a ferroresonant stabilizer or, much better, the lamp is powered from storage batteries of large capacity and from an AC rectifier connected in parallel with them. In the ZMR-2 monochromator a special device is built in which stabilizes the luminous flux. At the exit collimator slit of the monochromator there is a Nicol prism polarizing the outgoing monochromatic beam of light. The polarized light falls on a rotating spherical mirror installed in the lower part of the MF-2 microphotometer. In order to install this mirror it was necessary to remove from the microphotometer the metal tube connecting the slit with the lower reflecting prism.

Polarized monochromatic light, reflected from the mirror, passes through the rectangular reflecting prism and enters the lower objective, which focuses the image of the illuminating slit of the monochromator on the object under study. The latter is fixed on a rotating disk with an aperture in its central part. This disk is specially installed on the movable stage of the microphotometer. The image of the object under study, by means of the upper movable objective of the microphotometer, the upper reflecting prism, and also two interchangeable lenses, is projected onto an observation screen having a rectangular cutout. Light, after passing through this cutout, enters the measuring slit of the microphotometer, which cuts out a beam of rays limited in width and height and corresponding to the photometered region of the crystal. The light, after passing through a lens, falls on the FEU-19 electron photomultiplier, which is installed in place of the photocell, since the sensitivity of the latter is insufficient. The electron photomultiplier is enclosed in a metal light-tight casing mounted on a special stand. The photocurrent is measured by a mirror galvanometer G, built into the microphotometer together with an automatic reading scale. The sensitivity of the galvanometer is 10^{-9} A/mm. The electron photomultiplier is powered from a high-voltage stabilized rectifier VVS-1. For this purpose one may also use other high-voltage stabilized rectifiers, as well as dry batteries of the BAS-80 type. A voltage stabilized by a ferroresonant stabilizer is also supplied to the VVS-1. The optical and electrical diagrams of the entire setup are shown in Figs. 1 and 2.

Fig. 1. Electrical block diagram of the microspectrophotometric setup.
1 —stabilized light source, 2 —EPP-09 self-recording electronic potentiometer, 3 —ferroresonant voltage stabilizer.

The specimen under study, in the form of an ordinary petrographic thin section or a powder of very small crystals placed on a slide, is fastened with clamps or

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Fig. 2. Optical diagram of the microspectrophotometric setup. 1 —stabilized light source, 2 —monochromator, 3 —polarizer, 4 —objective, 5 —stage, 6 — specimen under investigation, 7 —analyzer

Figure 2: Fig. 2. Optical diagram of the microspectrophotometric setup. 1 — stabilized light source, 2 —monochromator, 3 —polarizer, 4 —objective, 5 —stage, 6 —specimen under investigation, 7 —analyzer

plasticine on the rotating disk under the upper objective of the microphotometer. First the object is examined in white light, which is introduced into the system by means of the rotating spherical mirror fixed in the lower part of the MF-2. An auxiliary illuminator serves as the light source. By moving the upper objective of the microphotometer, the image of the crystal under study is focused on the white screen. Next, by moving the thin section or slide on the disk, the selected crystal or an individual zone of it is positioned so that the aperture of the measuring slit placed behind the screen does not extend beyond the limits of the crystal under study or of the individual region of it. If the object is very small, then, by changing the upper objective, its image can be enlarged. Then, with the aid of the rotating mirror, polarized ···

monochromatic light. After switching on the second Nicol analyzer, placed in front of the screen, the mineral under study, or an individual zone of it, is set to extinction. This achieves selection of the required direction in the crystal. After the mineral has been set to extinction, the analyzer is again removed from the system. Next the movable stage of the microphotometer is secured so that it can move only in one direction. It is then shifted until the aperture of the measuring slit of the microphotometer coincides with the edge of the thin section, where there is only Canada balsam.

Fig. 2. Optical diagram of the microspectrophotometric setup. 1 —stabilized light source, 2 —monochromator, 3 —polarizer, 4 —objective, 5 —stage, 6 — specimen under investigation, 7 —analyzer.

For checking, the analyzer is inserted again. If the crystal is being studied not in a thin section but simply on a microscope slide, the image of the crystal must be shifted away from the aperture of the measuring slit. This makes it possible to measure the intensity of the light incident on the crystal, i.e., I_0. After this, two movable clamps, specially installed on one of the guides of the movable stage, are fixed in order to regulate the interval of movement of the latter. The

clamps must be secured so that, when the stage is moved in one direction as far as the stop (clamp), the selected crystal or an individual zone of it coincides with the aperture of the measuring slit, and when the stage is moved in the other direction as far as the second stop (clamp), it is possible to measure I_0.

The electron photomultiplier, serving as the receiver of the monochromatic light, like any photocell, has a nonuniform spectral sensitivity. If the absorption of the crystals is calculated by the formula

$$D = \lg \frac{I_0}{I},$$

then the change in sensitivity of the photomultiplier as a function of the wavelength of the light incident on it will not affect the results of the measurements. However, such calculations, as already mentioned above, take up

much time. To avoid this, at each new setting of the wavelength drum of the monochromator, the sensitivity of the photomultiplier is brought to a constant value, which is achieved by changing the voltage supplied to power the FEU-19. Owing to the fact that, without changing the orientation of the crystal, we can rapidly move our specimen from the position in which I_0 is measured to the position for measuring I, it is possible quickly to obtain the absorption characteristic of the crystal in the wavelength interval from 400 to 700 mμ, in percent.

This is done in the following way. Having set the specimen for measuring I_0 and selected the required wavelength, one must change the voltage supplied to the photomultiplier until the galvanometer indicates the "zero position." As the latter it is best to take the edge of the linear scale of the microphotometer, i.e., reading 1000. This reading will indicate 100% light transmission. Then, having moved the movable stage of the microphotometer to the second stop and thereby aligned the crystal under investigation, or a separate zone of it, with the aperture of the measuring diaphragm, we obtain on the scale a reading indicating the transmission of the crystal under investigation in the given direction for the given wavelength, in percent. Then we move the stage back to the other stop, set a new wavelength, and, by changing the voltage on the VVS-1, again set the "zero position." Next, again moving the stage to the stop, we obtain on the automatic scale a new value of the transmission. After carrying out such an operation the required number of times, one can immediately proceed to constructing the transmission or absorption curve of the specimen under investigation. The absorption will be the difference that supplements the transmission value to 100%. The spectral absorption curve will appear as the inverted graph of the transmission curve.

The microspectrophotometric setup described makes it possible, in a relatively short time, to investigate not only very small crystals, but also individual zones in them with good reproducibility of the measurement results. This is especially

important in the study of zonal minerals. Grain sizes can be measured not only in tenths, but also in hundredths of a millimeter.

The use of the logarithmic scale of the MF-2 microphotometer makes it possible to calculate the optical density D relatively quickly as well. It will be equal to the difference of the readings for I_0 and I on the microphotometer. By using the ZMR-2 monochromator as a source of monochromatic light, we achieved automatic recording of absorption spectra. For this purpose, the photocurrent coming from the electronic photomultiplier is connected, by means of a switch, not to the mirror galvanometer of the microphotometer, but to the input terminals of the EPP-09 electronic self-recording potentiometer.

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Received
2 VII 1959

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Note: Figure translations are in progress. See original paper for figures.