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PHOTOELECTRIC APPARATUS FOR REFRACTIVE INDEX DETERMINATION BY THE IMMERSION METHOD

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ABSTRACT

To reduce the subjective error in refractive index determination by the immersion method a photomultiplier together with phase contrast was used. The special adaptation of the photomultiplier diaphragm rendered it possible to determine the temperature of matched refractive indices of the liquid and the measured material based on the photomultiplier output.

INTRODUCTION

In most commonly used processes for measuring the refractive indices of solids by the immersion method, the reliability of the acquired values is not proportional to the expended time and effort. The use of thermistors (Chromý 1965) in conjunction with relatively precise thermal (Gaubert 1922) and double variation methods (Emmons 1929) further reduced the time over ordinary immersion techniques, but even then the accuracy of the results depended on the visual determination of a match between the refractive indices of grain and liquid. The resultant error depends upon the investigated effect (Becke line, double diaphragm method, etc. (Saylor 1935)) and upon the size of the measured grain, the amount of inclusion, etc. It is smallest when using phase contrast where it is possible to reduce it even more by a photoelectric element and to remove the biggest source of subjective error when measuring phases.

PRINCIPLE OF METHOD

The refractive index match between mineral and liquid is obtained either by heating the preparation on an electrically heated support glass with built-in thermistor, or in the measuring cell of a stage with a revolving glass thread (Chromý 1966). The heating and cooling processes of the preparation can be easily controlled and the temperature can be observed on the microammeter of the thermistor thermometer. The refractive index of the mineral is then taken as the refractive index of the liquid for the temperature and wavelength, at which matching takes place. This latter index is measured on a goniometer with an electrically heated prismatic stage, the temperature of the liquid being measured by the same thermistor thermometer.

During the heating process (n-mineral$<n$-liquid) the reduction of contrast, observed by phase contrast techniques, occurs between the mineral

grain and its surroundings. As the effect of the phase contrast appears on the whole grain surface, it is possible by scanning the light intensities to determine the temperature of the minimum contrast, this being the temperature of match between the refractive indices of mineral and liquid.

METHOD OF MEASUREMENT

Phase contrast was measured with Heine's condenser (Messrs. Leitz-Wetzlar). Monochromatic illumination was obtained from either a sodium vapor lamp or a microscope lamp equipped with suitable interference filters.

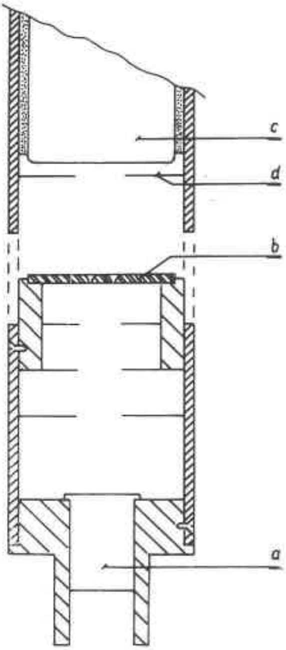

FIG. 1. Sketch of photoelectric scanner. (a) eye piece, (b) diaphragm, (c) photomultiplier, (d) metal partition with opening.

Light intensity was scanned by a photoelectric scanner (Fig. 1). This scanner consists of two parts. The lower part with the eye piece (a) is set on the Leitz-Ortholux microscope replacing the camera and is provided with an exchangeable diaphragm with a small opening (b). The upper part is revolvingly mounted on the lower part and contains the photomultiplier (c); only a deflected beam of rays falls on the photomultiplier from the microscope visual field image as projected onto the diaphragm. To measure the refractive indices, the diaphragm at *b* was prepared with two openings, precisely identical, placed within a short distance symmetrically along the center and the anterior-posterior symmetrical axis of the visual field. The position of the openings makes it possible, when looking into the microscope, to locate the measured grain at one side of the cross-hair intersection, so that its image is projected only onto one opening (Fig. 2a). The front face of the photomultiplier in the upper part of the scanner was covered with a semicircular metal partition at *d* in Figure 1. Its shape made it possible to cover one of the openings but not the other by slight rotation of the upper part of the scanner (Fig. 2b). For each 1°C increase in temperature of the preparation, the upper part of the scanner was slightly rotated to block out first one opening and then the other. For each opening, the photomultiplier voltage was recorded as shown in Figure 3. On the basis of this record the temperature at which the light intensities impinging on the two openings match can be very precisely determined. This is the looked-for matching temperature of the refractive indices of the measured grain and the immersion liquid. With grains containing inclusions or transformation products, it is necessary before the actual measurement to find a location for the measured grain at which the light intensity impinging on both openings in the diaphragm is equal in the light field.

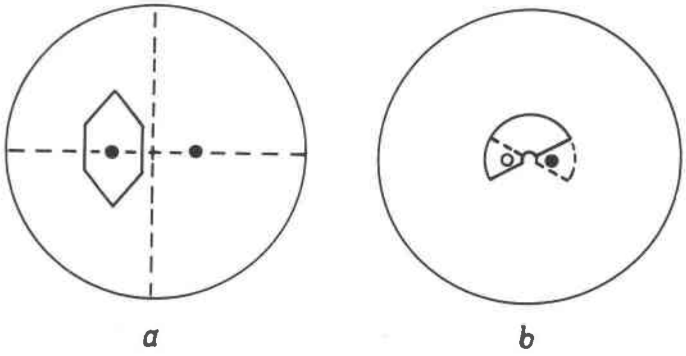

Fig. 2. a—Location of openings in the diaphragm of the photoelectric scanner (with the projection of the eyepiece crosshairs) and the location of the measured grain in the visual field. b—Diagram of covering the openings in the diaphragm when rotating the upper part of the photoelectric scanner.

The described alternating light-intensity scanning of equal faces of the measured grain and its surroundings can be also improvised by only one opening in the photomultiplier diaphragm, or by means of an electric microphotometer connected to a sensitive recorder. On an uncentered microscope stage we locate the measured grain in the center of the cross

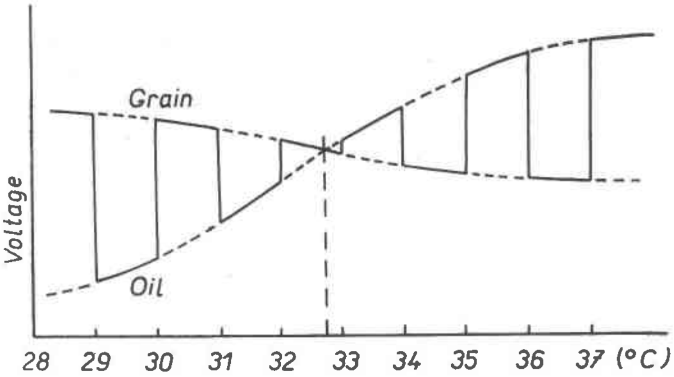

FIG. 3. Appearance of recorded measurements.

hair intersection in such a way as to project its image on the opening in the photomultiplier diaphragm. In optically anisotropic materials the grain must be simultaneously properly orientated in the extinction position. We mark the relevant value of the peripheral stage scale and revolve the stage in order that the grain image may leave the center of the hair cross intersection as it is pictured in Figure 4. When measuring, we alternate both stage positions mentioned above precisely in the intervals of 1°C. The obtained graph is equal to that, when using the photomultiplier diaphragm with two openings (Fig. 3).

The photoelectric method of temperature determination by matching refractive indices is very advantageous especially, when measuring by the double variation method. Three matching points at different illumination wavelengths can be determined very quickly and precisely in the course of one heating and cooling process. Practical measurements have

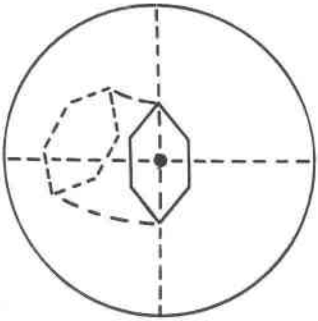

FIG. 4. The position of the measured grain when using only one opening in the photomultiplier diaphragm.

shown that only at rates of temperature change higher than 1°C/min is it necessary to reckon with different results for heating and cooling and to consider average values.

The above method for determining the matching temperature of the indices removes the greatest source of subjective error when measuring phases by the immersion method. It also makes it possible to utilize fully all advantages of the thermistor apparatus. Practically this means a significant shortening of the time necessary for the measurement at a given tolerance. The accuracy of the method increases, as was shown when measuring refractive indices of glasses to ±0.00005.

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