VERY HIGH INDEX OF REFRACTION MEDIA FOR PRACTICAL MICROSCOPIC MINERAL IDENTIFICATION

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INTRODUCTION

The need for immersion media for the rapid identification of minerals have very high indices of refraction is sorely felt when examining concentrates from alluvial deposits. In the United States, many minerals of strategic value may be economically recovered only from such deposits. Evaluation of these complex placers and determination of economic recovery and separation methods and equipment requires that the entire mineral assemblage be identified.

Macro and micro examination permits identification of a certain percentage of the mineral grains and crystals, but many of the black sands minerals, after transport and attrition, cannot be identified visually. The considerable differences of refractive indices of many of the valuable minerals found in alluvial deposits suggests the desirability of identifying such minerals by use of the petrographic microscope and immersion media. The method is rapid, and in the case of minerals of moderate indices of refraction, comparatively simple.

Commrcially Available Media

Liquids having known indices of refraction, up to 2.11 are readily available, and much used. Investigation of samples taken during exploration of alluvial deposits broadens the schope of this technique and makes the petrographic microscope, with a sutiable assortment of immersion media, a valuable tool for the minerals industry.

Imersion Media of Very High Indices of Refraction

Mention of melts of sulfur and selenium, and of arsenic selenide and selenium for very high index of refraction media is made by several early workers. The most complete and readily available work on the refractive index method of mineral identification is U. S. G. S. Bulletin 848, "THE MICROSCOPIC DETERMINATION OF THE MONOPAQUE MINERALS" by Larsen and Berman. On page 16 of the second edition of this publication is a graph indicating indices of refraction of various combinatons of sulfur and selenium in homogenous melts.

Data for the theoretical percentages of arsenic selenide to selenium for melts having indices above 2.72, the upper limit of the sulfur-selenium melts, are very limited. To obtain the required ratio of arsenic selenide to selenium for preparation of melts having the desired higher indices of refraction, available data was plotted.

Required percentages for melts having the desired indices of refraction, as taken from the curves, are listed in the tables.

TABLE I
SELENIUM/SULFUR BY WEIGHT

^{n}Li	%Se	^{n}Li	%Se
2.13	44.3	2.34	74.0
2.15	48.3	2.36	76.0
2.19	56.0	2.40	80.0
2.20	57.0	2.42	82.0
2.21	58.5	2.43	83.0
2.23	61.2	2.45	84.0
2.24	63.0	2.47	85.6
2.25	64.0	2.49	87.0
2.26	65.0	2.55	91.0
2.28	68.0	2.60	94.0
2.31	71.0	2.62	95.0
2.31	72.0	2.72	100.0

TABLE II
ARSENIC SELENIDE/SELENIUM BY WEIGHT

^{n}Li	%As_2Se_3	^{n}Li	%As_2Se_3
2.81	28.0	3.02	79.5
2.819	30.05	3.063	86.4
2.903	54.5	3.073	87.7
2.94	63.7	3.146	96.3
2.999	76.2	3.18	97.8
3.0	77.0	3.2	100.0

Preparation of Media

Ingredients for the preparation of the melts should be of the greatest available purity, and if not obtained in powder form, should be pulverized. The weighed portions required to obtain melts of the desired indices of refraction are well mixed in fairly large test tubes and carefully melted over a burner in a well ventilated hood. Adequate ventilation is a must, as the fumes evolved are highly toxic. Only sufficient heat to melt the mixture completely should be used, and is most readily obtained by manipulating and varying the height of the test tube above the burner. Excessive heat will cause volitization and increase of refractive index. The front of the hood should be lowered to avoid inhalation of fumes, allowing only sufficient space between front and floor of hood to permit entrance of the hands and arms. The mixture should be stirred during the melting, to ensure complete blending, using heavy nichrome or stainless wire. Test tubes should not be reused for melts of other indices, and the wire should be burned off until all adhering material is consumed before reuse. Refractive index of each melt is checked by immersion of grains of known minerals.

Special Equipment Requirements

Examination of minerals having very high indices of refraction immersed in the highly colored melts requires some special equipment. A small electric hot plate with temperature control and a high intensity illuminator are required. A variac for control of light is convenient. A filter for transmission of light approximating wavelength of lithium light, 6707 A, may be made by melting a mixture of approximately 75% selenium and 25% sulfur on a 35mm glass slide and pressing another firmly upon it to spread the melt thinly and evenly. This filter is used in the light train with all others removed. A simple magnifying lens between light source and substage mirror is of value in increase of light intensity.

Techniques

Mounts are made by cutting or breaking a very minute portion from the cooled and solidified melt and placing it upon the slide on the hot plate. As the indices of refraction of the melts increase, the heat required to remelt the media increases. Shattering of slides during the heating cycle is minimized by gradual heating and cooling. Only sufficient heat to melt the media to a globule sufficiently liquid to hold the mineral grains, and to flow between cover glass and slide when gently pressed with a small cork stopper, should be used. Careful pressure and partial rotation of the small cork distributes melt and mineral grains evenly and thinly, permitting light transmission. Excessive loss of slides may be avoided by placing as many as required around the periphery of the hot plate with about one half inch extending over the edge, facilitating handling, and slowly bringing to desired temperature. The grain mount must be slowly cooled before being placed upon the cooler microscope stage. The mount should be made near the hotter end of the slide, and after cooling, data may be recorded at the opposite end. Mounts are permanent, although some may crystallize in time, requiring reheating before later examination.

The higher the refractive index of mineral and mount, the greater the intensity of light required, usually requiring use of the substage condenser lens when making examination of very high index mounts. Minerals are more nearly opaque as their indices of refraction increase, and smaller size grains must be used, with higher magnification, for satisfactory light transmission. The lower range of high index minerals may give satisfactory results with –100 +150 mesh fractions and moderate magnification, but those of higher index require fragments of –200 +325 mesh, those having moderate index may be satisfactory in the –150 +200 mesh range. Highest magnification and light intensity is required for very high index minerals. Sufficient lght transmssion is most important.

Inference Figures

Only in the lower range of the very high index minerals will it be found ordinarily possible to obtain interference figures of sufficient clarity to be of much use other than for the determination of optical character. Usually only a very hazy figure may be distinguished, but nearly always, except in the very highest indices of mineral and media, it is possible to determine whether the mineral is uniaxial or baixial. The color and opacity of the mount precludes determination of optical sign by use of accessories. In those cases of sufficient importance, all indices may be determined. This is seldom required as there is considerable difference between intermediate indices of the anisotropic minerals usually found in the assemblages under consideration.

Fortunately many of the mineral fragments difficult of identification by other means are isotropic and approximate match of mineral and media, plus consideration of physical characteristics, is usually sufficient for identification. Many of the anisotropic minerals may be identified almost as readily by determination of approximate intermediate indices, especially if an interference figure is obtained.

CONCLUSIONS

Identification of minerals having very high indices of refraction in the very high index melts is a vastly different technique than that used for minerals and oils of lower indices. The same principles apply, but the observed optical phenomena are more difficult to interpret. The method has greater limitations due to the opacity of both mineral and media, and the highly colored melts may often confuse the microscopist until practice and familiarity permit compensation for the color. Within the inherent limitations of the method, it is probably the most practical and rapid means of identifying mineral fragments having very high indices of refraction.

MONITORING GROUND WAVES AND AIR WAVES FROM QUARRY BLASTS*

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INTRODUCTION

In the spring of 1960 a program was initiated at the South Dakota State Cement Plant in Rapid City under the direction of Mr. R. D. Marquardt, Assistant Manager, to monitor ground and air waves from blasts in the Plant quarry. Thirty-one blasts have been monitored to date.

The purpose of monitoring the blasts was to determine if levels of ground acceleration, energy or air pressure due to the blast were being reached at points located between the blast and built-up areas of Rapid City which were likely to result in damage. These levels have never been reached in the Plant quarry blasting since the tests were begun.

GROUND WAVES

Acceleration and relative energy of the ground at a point is measured by means of a Leet Portable three-component seismograph. This instrument reproduces photographically a record directly proportional to ground displacement under the seismograph for frequencies 1.25 sec^{-5} and greater and magnified by a factor of 50. Three displacements are recorded at once: the horizontal and vertical transverse displacements and the longitudinal displacement. The record produced by three light beams is on 70-mm electrocardiogram paper run through the machine at about four inches per second. Time markers 0.01 sec apart, with a marker every 0.1 sec darker than the rest, are placed on the record by the instrument.

* This work supported by the South Dakota State Cement Plant.

Most waves recorded are approximately damped sinusoidal waves. The undamped part is given by Eq. 1. Amplitude y_o and frequency f may be read for each of the three waves from the record. The acceleration is given in Eq. 3.

Values of acceleration for each of the three mutually perpendicular displacements are added vectorially to give the total acceleration, but since the displacements are generally not in phase, this total acceleration is an upper bound.

The energy E received from the blast by the ground under the seismograph at any instant is proportional to the square of the

velocity of the ground. The velocity is given in Eq. 2. The maximum value of E, E_{max}, follows from this and is given in Eq. 4. The ratio a^2_{max}/f^2 has been defined(1) as the energy ratio E. R. and is a useful measure of the energy in each component of the blast wave.

$$y \cong y_o \sin 2\pi ft \qquad (1$$

$$v = dy/dt = 2\pi f\, y_o \cos 2\pi ft = v_{max} \cos 2\pi ft \qquad (2$$

$$a = d^2y/dt^2 = -(2\pi f)^2 y_o \sin 2\pi ft = -a_{max} \sin 2\pi ft \qquad (3$$

$$E_{max} \sim v^2_{max} = (2\pi f)^2 y_o^2 \sim a^2_{max}/f^2. \qquad (4$$

AIR WAVES

Besides ground waves from blasting there are also waves propagated through the air, and indeed these are the waves most noticeable to the casual observer some distance away from the blast. We undertook to measure the pressure of the air wave at the same point where the Seismograph was set up. We use a General Radio 1551-B Sound Level Meter which is commonly in use as a decibel meter in noise analysis. In addition we use a General Radio 1556-A Impact Noise Analyzer connected to the Level Meter. This device enables one to analyze impact-type sounds in addition to more-or-less steady-state sounds.

The Impact Noise Analyzer measures the quasi peak of the air pressure pulse and stores in its circuit the true peak air pressure and the time average of this pulse, averaged over a given time.

After the blast is over and the seismograph record has been obtained, we cause the Analyzer to display on its meter dial first the peak value and then the time average. We photograph each value with a Land Camera and develop the print in 10 seconds. The Analyzer will store the readings more than a minute with only 1 db loss. We then have a permanent photographic record of both ground waves and air pressure.

The peak reading in decibels is converted to pressure by means of the equation 74 db = 1 microbar = $1.45(10)^{-5}$ lb/in².

DAMAGE CRITERIA

The U. S. Bureau of Mines(2) carried out a five-year survey of blasting and concluded generally that acceleration a less than 0.1 g, where g = acceleration of gravity (386 in/sec²), causes no damage even to plastered walls. For $0.1\,g < a < g$, damage was possible, and for $a > g$, damage was likely.

Crandell(1), Leet(3) and others have pointed out that the energy at a point due to a wave is probably a better criterion for its possibility to do damage than the acceleration at the point. An extreme example of this fact is afforded by an earthquake. Earthquake waves with maximum displacement of the order of 1 inch and frequencies of 1 cycle are compared with blast waves with displacement = 0.01 inch and frequency of 10 cycles. The acceleration due to the earthquake wave is only about three times that of the blast wave, but it is 400-500 times as energetic. The ratio of damage done by the earthquake to that by the blast is more nearly proportional to the ratio of energies.

Crandell(1) defines the energy ratio $E.R. = a^2/f^2$ in^2/sec^2 and as the result of many tests predicts that for $E.R. < 3(144)$ in^2/sec^2 there will be no damage. For $3(144)\ in^2/sec^2 < E.R. < 6(144)\ in^2/sec^2$, damage is possible, and for $E.R. > 6(144)$ damage is likely. The states of Massachusetts and Connecticut have established an allowable upper limit to the energy ratio of $E.R. = 144\ in^2/sec^2$. It is this more conservative figure we have used since we calculate E.R. for only the most energetic of the three ground waves.

Air pressure damages windows before any other parts of a structure. The pressure to break a window is about 1 lb/in^2, produced for example by a 170-mile/hour wind. We use a pressure of 1 lb/in^2 as the danger point for air pressure pulses due to quarry blasts.

It is interesting to note that a human observer can detect easily a displacement of 0.0005 inch due to a 10-cps ground wave. Thus he can feel an acceleration less than 0.01 g and respond to an energy ratio of 0.04 in^2/sec^2, where the damage thresholds are 0.1 g and 144 in^2/sec^2. Displacements from 0.0005 to 0.02 inch at frequencies of 5 to 20 cps are not uncommon in dwelling structures from walking, door slamming and the passing of trucks in the street.

RESULTS

Measurements made from 1960-1963 of the ground waves and air waves due to quarry blasts are summarized in Table I. The test site was 0.5 to 1 mile from the blast and between the blast and a built-up area of Rapid City. In only one of 31 tests did the acceleration exceed 0.1 g, and that by 20 percent. The energy ratio for this record (No. 34) was only 0.41 in^2/sec^2, where a danger level is 144 in^2/sec^2. The highest energy ratio recorded was 3.0 in^2/sec^2.

The highest air pressure pulse recorded in 20 tests was 0.01 lb/in^2. 1 lb/in^2 is generally required to break a window.

TABLE I

RESULTS OF MONITORING GROUND WAVES AND AIR WAVES FROM QUARRY BLASTS

South Dakota State Cement Plant, 1960-1963

Distance from Blast 0.5 to 1 Mile

Acceleration Units of g			Energy Ratio $E.R. = a^2/f^2$ in^2/sec^2			Air Pressure lb/in^2		
No. Values Recorded			No. Values Recorded			No. Values Recorded		
<0.1	0.1-1	>1	<1	1-144	>144	<0.01	0.01-1	>1
30	1	0	26	5	0	19	1	0
Total tests: 31			31			20		

REFERENCES

1. F. J. Crandell, J. Boston Soc. Civ. Engrs. **36**, 222 (1949).
2. J. R. Thoenen and S. L. Windes, **Seismic Effects of Quarry Blasting** (U. S. Bureau of Mines, Bulletin 442; Government Printing Office, Washington, 1942).
3. L. Don Leet, **Vibrations from Blasting Rock** (Harvard University Press, Cambridge, Massachusetts, 1960).