Brewster Conoscopy for the Measurement of High Refractive Indices in Gemstones

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BIOGRAPHY

Brad Amos worked on cell motility and cell division before entering the field of optical instrument design. He and John White developed the laser scanning confocal microscope which was later commercialised by Bio-Rad. Dr Amos' lectures on optical microscopy – including those at the Plymouth (UK) Microscopy Course – led to an interest in the fabrication of crystalline demonstration specimens and to the craft of gem faceting. He is a prizewinning member of the UK Facet Cutter's Guild and has published a series of articles on gemstone birefringence.

ABSTRACT

A method is presented for the determination of high refractive indices in gemstones such as diamond and diamond substitutes, above the limit of 1.8 that can be measured by a conventional refractometer. Measurement requires access to only a microscopically small area of a facet, with an accuracy of ±0.05 in refractive index. The principle is to determine the Brewster angle, as in existing instruments, but using a polarizing reflection microscope. The result is obtained by examination of the back focal plane of an oil-immersion objective and can be recorded in an image. A patent is being sought for the method.

KEYWORDS

light microscopy, polarized light microscopy, refractive index, Brewster's angle, refractometer, gemmology, diamond, moissanite

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INTRODUCTION: THE PROBLEM

To identify a gemstone or other mineral, it is often necessary to measure its refractive index (RI). Unfortunately, the conventional refractometer invented by Abbe cannot be used for RIs higher than that of the contact fluid (maximum 1.8). This means that diamond, with an RI of 2.42, and many diamond substitutes, are out of range.

Several ways around this problem have been tried. In the past [1] the Abbe range was extended by heating both the instrument and gem in order to permit the use of fluids of higher mass concentration (with problems of inflammability and toxicity). Currently, three less hazardous approaches are used. A reflectance measurement yields the index, but the surface must be perfectly polished and the photometric area closely defined. The ratio of real to apparent depth is equal to the index, but requires a microscope with calibrated focus and no slippage, and is hard to measure when the gemstone is mounted in a jewellery setting that impedes direct measurement of the real depth. Finally, there is a method which depends on the measurement of Brewster's angle *B*. If a ray of light is incident at this angle and is polarized parallel to the plane of incidence, the reflected ray is totally suppressed. If the refractive index of the gem material is *n*, $\tan B = n$ [2]. Brewster angle refractometers are commercially available (e.g. through the Gemmological Association) and one design has been patented [3].

The way that these instruments work is that a polarized laser beam is directed at the facet surface (usually the flat top or table of a gem) and the angle is varied until a minimum reflectance is found. The chief difficulty with these instruments is that no image is available of the facet that is being measured.

For the method described here, a microscope is used instead of a refractometer. The microscope is used in reflection mode and the measurement is taken from the reflected light in the back focal plane, in a manner analogous to the conoscopic mode in transmission. This approach has the advantage that the RI can be measured in areas only a few tens of micrometres in diameter. Surprisingly, this method appears to be novel.

APPARATUS AND METHOD

The apparatus consists of an epi-reflection microscope (Figures 1 and 2), equipped with a polarizer in the illumination path, which ensures that the light entering the beamsplitter is polarized horizontally (in the sense shown in Figure 1) so that the beam reflected into the objective lens is polarized in the east-west azimuth. The gem (or entire jewellery setting) is placed on a microscope slide and secured (e.g. with plasticine) so that one facet is horizontal and faces the objective. The selected facet can be made horizontal by gentle pressure from a second microscope slide held parallel to the first.

The microscope is then focused on the facet, using a low-power objective, and an area free of contamination or scratches is chosen. Next, immersion oil is placed on the facet and an objective of high numerical aperture (NA) of

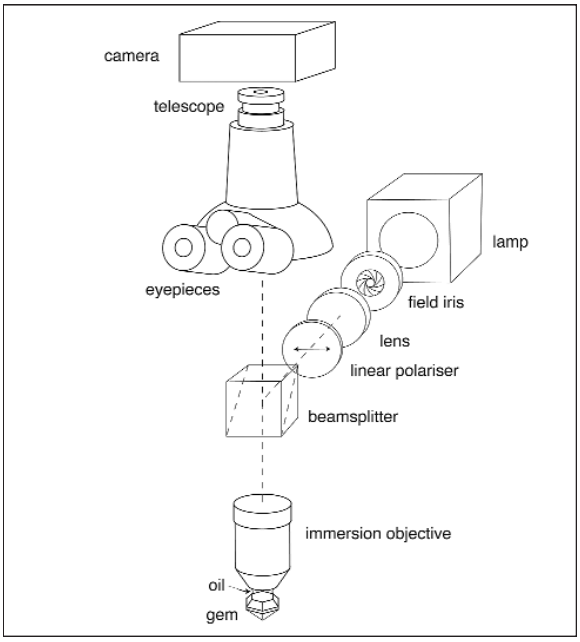

Figure 1:
Schematic diagram of the microscope. The monochromatic filter in the illumination path is not shown. It is better to use an integral Bertrand lens than the phase telescope shown here.

1.4 is swung in and the gem surface is refocused. No polarizer is necessary in the reflected light path, but it is essential to be able to insert a Bertrand lens (or replace the eyepiece with a phase telescope) in order to view the back focal plane of the objective as a bright disk of reflected light (Figure 3). This is known in the field of light microscopy as the 'conoscopic mode' of observation. The disk of light is marked by two dark spots, positioned symmetrically at the east and west sides (see Discussion for the origin of these). The distance between the centres of the two dark spots is constant for any given isotropic material, and increases with its refractive index.

The image may be recorded conveniently, using a camera attached to the microscope. For this work, a standard Nikon LV 150 metallurgical microscope was used, fitted with a Nikon DS 5M L1 camera system. The automatic gain of the camera was switched off and monochrome operation was selected. The brightness of the halogen lamp was adjusted so that the white background was oversaturated but a large range of digital grey levels were represented within the dark spots.

RESULTS

All of the observations shown here were made with an NA 1.4 oil-immersion objective.

Figure 3 shows a series of images obtained with gemstones of known index, ranging from 1.72 to 2.9. It is plain that the separation of the dark spots increases with the refractive index (using published RI values: [3] and [4] for moissanite), although the relationship is not linear (Figure 4). It is also clear that, even by simple visual observation of the distance between the spots in images such as Figure 4, it is possible to distinguish clearly gems such as moissanite, diamond and cubic zirconia. A diamond-simulant gem of unknown provenance gave a result (indicated by the horizontal dotted line UU' in Figure 4) very similar to cubic zirconia.

NIH Image (Scion) software was used to make horizontal scans (Figure 5) through images such as those in Figure 3. It is evident that for the higher index (cubic zirconia) the distance between the peaks is defined with great precision. Measurements were carried out with the software of the Nikon DS 5M L1 camera system, which allows a cursor to be placed at the centre of first one dark spot and then the other, and then displays the distance between the two centres (in arbitrary units).

When nine different diamond specimens were imaged, the readings showed a standard deviation (SD) of 1.03%, but the SD was strongly influenced by one particular stone, which was badly tilted. When this one was eliminated, the SD became 0.54%, which is equivalent to an index error of ±0.02. The contrast in the image was greatly improved by a green filter, though a yellow filter would be preferred, since refractive indices are normally measured at the wavelength of the sodium D line. The contrast in the image was also improved by closing the field iris as far as possible, and by selecting a specimen region with good planarity and polish. Occasionally, a pair of very small dark spots was seen, caused by a single air bubble in the immersion oil, which was easily removed by replacing the oil.

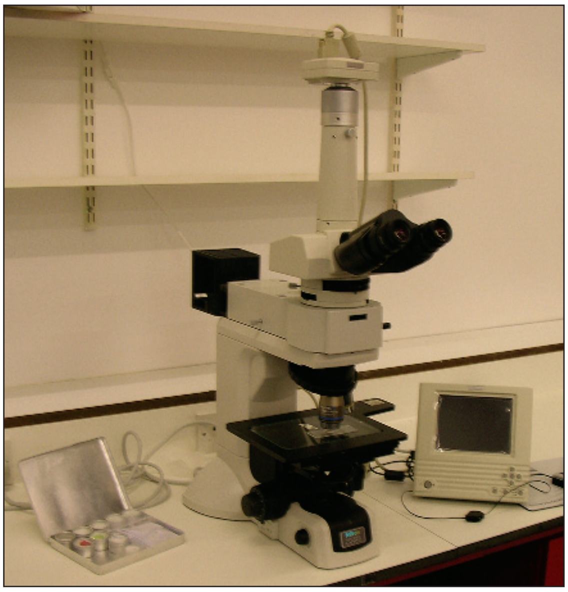

Figure 2:
Left: Nikon LV150 metallurgical microscope equipped with an intermediate attachment containing an integral Bertrand lens and a DS 5M L1 camera system.
Top: Faceted gem of rutile (TiO_2), where the two refractive indices result in double images of certain facets. The colours are due to the dispersion of white light.

When a dry objective with a numerical aperture of nominal NA 0.95 was used instead of the oil immersion with NA 1.4, the margins of dark spots could be seen at the limit of the back focal plane, but a satisfactory distinction between the gem materials could not be made. With an oil objective of NA 1.3, the spots due to diamond were at the outer limit of the back focal plane. NA 1.4 was found to be necessary to give readings on stones of higher refractive index than diamond.

With the strongly birefringent material, rutile (Figure 2), the spot separation varied according to the angle between the plane of polarization of the illumination and the crystalline optic axis (the value for rutile plotted in Figure 4 is merely one measured value). Movement of the spots in and out was demonstrated by rotating on the stage of the microscope a plate of rutile cut with the crystal optic axis parallel to the surface of the plate.

DISCUSSION

The dark spots are due to the fact that the conoscopic view (i.e. the view of the back focal plane of a microscope objective) is an angular map of the light reflected from the specimen [6]. Any point in the back focal plane contains a focused collection of all the rays that have reflected at a particular angle from the surface of the specimen and were (in the vicinity of the specimen) parallel to each other (Figure 6). Rays that are incident at the Brewster angle fall on a circle in the back focal plane, but, because the incident light is linearly polarized, only at two points on that circle are the rays that pass through those points polarized in the plane of incidence and fail to reflect.

It is necessary to explain why an oil immersion objective of numerical aperture 1.4 is needed, when a dry objective with a numerical aperture between 0.9 and 1.0 has a similar acceptance angle. The reason is that in the presence of oil, Brewster's angle is reduced, according to the equation tan $B = n_{gem} \div n_{oil}$, where B is the Brewster angle, n_{gem} the RI of

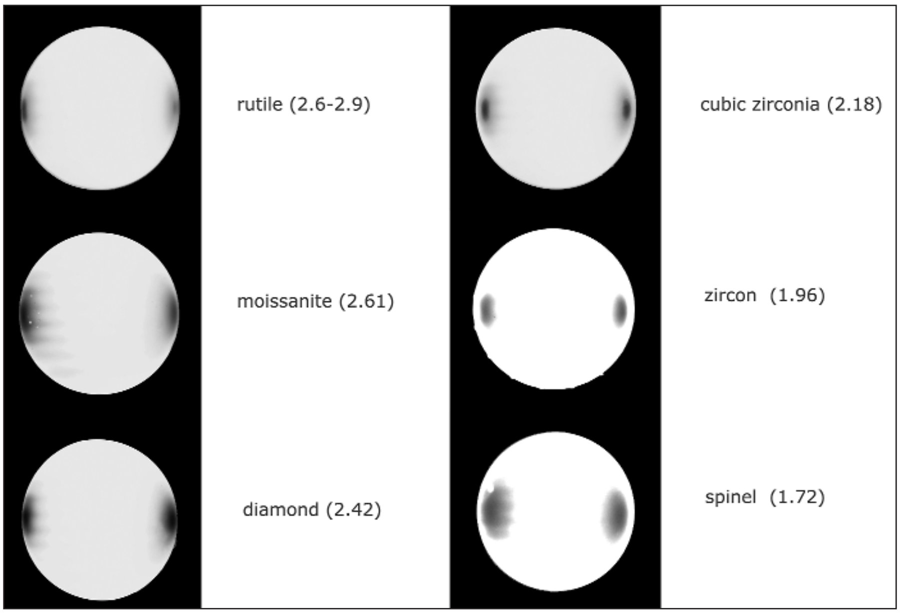

Figure 3:
Conoscopic patterns obtained by the method. Blurred images of the halogen lamp filament run horizontally across some of the patterns.

the gem material and n_{oil} the RI of the immersion oil.

This fact can be used to calculate the maximum refractive index that can be measured with different types of objective. For objectives of the following types, dry NA 0.9, oil NA 1.3 and oil NA 1.4, the maximum indices that can be measured are 2.06, 2.53 and 3.7 respectively. In practice, a cheaper oil immersion objective with nominal NA 1.3 showed the diamond spots at the outer limit of the back focal plane, and could not measure the index of moissanite or rutile. With a birefringent material such as rutile, it is to be expected that only one spot separation should be observable at one time, since either the ordinary or extraordinary ray will dominate, according to the angle between the plane of polarization of the input beam and the crystalline optic axis.

In this respect, the method is less convenient than the Abbe method, which shows two boundaries, corresponding to two indices, simultaneously. As the index falls towards 1.7, the reflectance image becomes dimmer and the peaks broaden, so the method becomes inaccurate (Figure 3): it is therefore complementary to the Abbe method rather than a replacement for it.

FUTURE DEVELOPMENT

This method seems to fill a gap in gemmological instrumentation, to be rapid, accurate and to require only very small facet areas. The chief objection to it may be that, although well-funded labs may have a metallurgical microscope and be able to equip it with an immersion objective of NA 1.4, jewellers never will. The chromatic correction of the objective used here is, however, not essential, and so it may be possible to develop a cheap substitute for this costly lens, or an instrument with a modified geometry which does not need such high numerical aperture.

It is hoped that this article will stimulate the invention of a cheap mass-produced refractometer according to this principle, and also encourage an extension of the method to single-grain index measurements of mineral sections in petrology, though a higher degree of polishing than usual may be necessary. A patent is being sought for the method.

REFERENCES

1. *Webster, R. Gems: their sources, descriptions and identification. 4th Edn. Rev. B. W. Anderson. Butterworth and Co. London, 1983.*
2. *Jenkins, F. A., White, H. E. Fundamentals of Physical Optics McGraw-Hill, New York, London, 1937.*
3. *Hyman, M. Brewster Angle Refractometer. United States Patent 5,572,314. 1996.*
4. *McClintock, W. F. P. Gemstones in the Geological Museum, 4th Edn, rev. P. M. Statham. HMSO London, 1983.*
5. *Emms, E. C. Synthetic Moissanite - the latest diamond simulant. 1999. http://www.precious-stonelab.co.uk/moissanite.htm*
6. *Wahlstrom, E. E. Optical Crystallography. John Wiley and Sons, New York, 1960.*

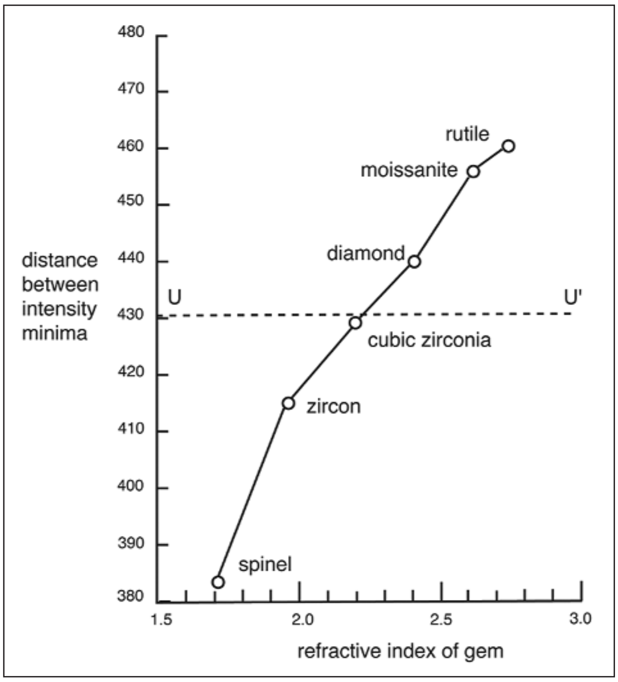

Figure 4:
Plot of known refractive indices versus the distance between the dark spots in conoscopic images. The distance scale is in arbitrary units (pixels at a particular magnification). The solid lines simply link adjacent data points. The horizontal line UU′ represents the spot separation for an unknown stone, providing evidence that it is probably cubic zirconia.

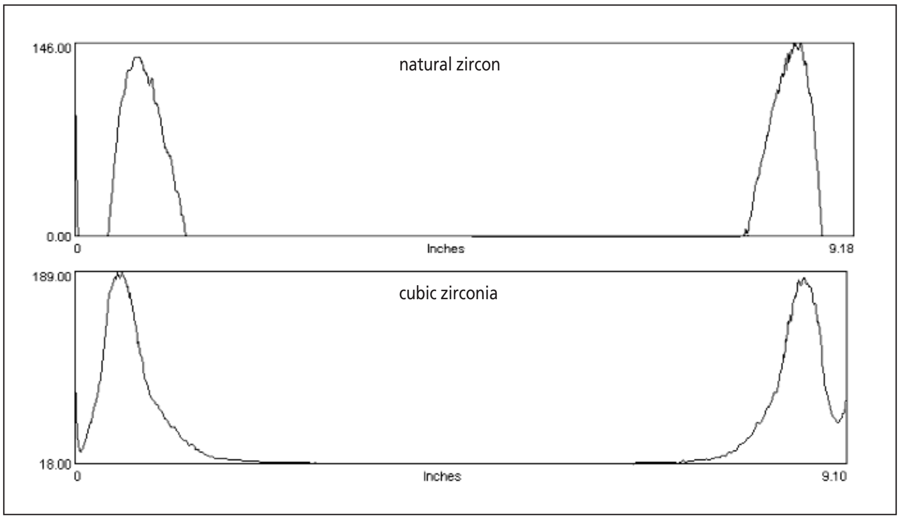

Figure 5:
Plots of image darkness against distance along a horizontal profile of conoscopic images for two gemstones, natural zircon and cubic zirconia.

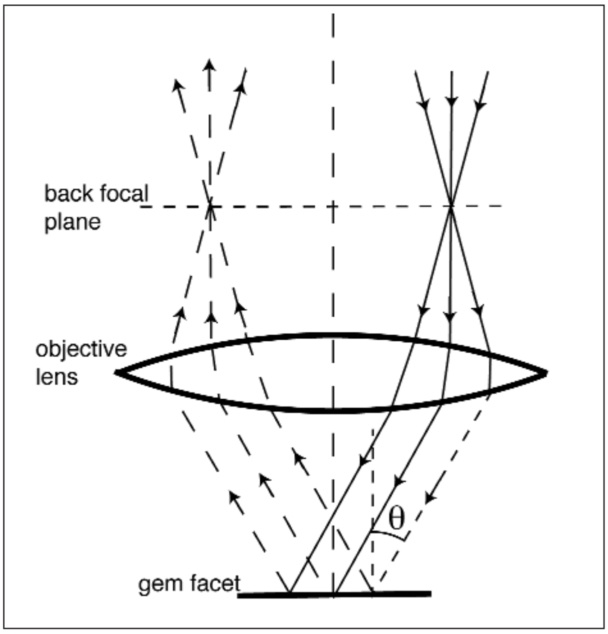

Figure 6:
Ray path through an objective lens (simplified). When the angle of incidence on the gem facet is equal to the Brewster angle, and the plane of polarization corresponds to the plane of the paper, the rays shown by dotted lines are suppressed. This causes a dark spot to appear in the back focal plane. A similar spot is generated by the corresponding rays on the opposite side of the optical axis (not shown).