NEW DEVELOPMENTS IN SPECTRAL REFLECTANCE MEASUREMENTS USED FOR MICROSCOPIC OPAQUE MINERAL IDENTIFICATION

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SUMMARY

Reflected light microscopy is the most frequently applied and the most economical method for the identification of common ore minerals. But long experience of the observer is needed to obtain acceptable results. The application of quantitative methods -measurements of microindentation hardness and mainly spectral reflectance-, in addition to the conventional observations increase the identification reliability drastically.
Today, photometers for spectral reflectance measurements work in a similar way: a monochromator is inserted into either the light path or the observation ray path of the microscope, and a photomultiplier is used to sequentially measure the light intensity at various wavelengths in the range of the visible light, 400 nm to 700 nm, normally in steps of 20 nm.
Recent developments of very high sensitivity CCD cameras led to the construction of simple to use, monolithic semiconductor spectrometers. Connected to a microscope they can completely replace the conventional photometers. The spectral resolution of these new instruments is better than 0.5 nm, which means more than 700 reflectance values in visible light. All wavelengths are detected simultaneously.
This new spectrometer type may also be used within the near infrared, 700 nm to 1000 nm. Until today the specular reflectance of ore minerals within this region is completely unknown. It is expected that the spectral extension to 1000 nm would greatly improve the identification possibilities. Preliminary tests have ensured that measurements are possible but several surmountable problems arise: a) conventional heat filters absorb too much light intensity; b) the chromatic aberration of refractive microscope objectives prevents measurements of small grains; c) currently, no IMA-COM approved reflectance standards are available for the near infrared.

EXTENDED ABSTRACT

INTRODUCTION

Since the very beginning of ore microscopy, users have suffered from the fact that the human observation system, the combination eye–brain, is not at all reliable. Therefore, since very long time ago attempts have been made to measure the reflectance of ore minerals with various methods. Based on the idea of Schneiderhoehn, Berek constructed in 1924 the so called “slit microphotometer” in which the incident light is split into two parts, one for reflection on the polished mineral and the other for comparison in a special ocular. Light intensity adjustment of the second path until equality with the reflected light is a measure of the mineral reflectance. It was even possible to apply colored light. A reflectance standard was not needed in contrast to all other methods. Orcel (1927) was the first to use photo-electric cells for measurement in blue and white light. In the following years many improvements were developed concerning all fields of photometer techniques, and sample polishing methods; the microindentation hardness was introduced to the ore mineral identification (Siebel , 1943), as well as color calculations (Piller 1966) . In 1962 the “International Mineralogical Association” (IMA) founded the “Commission on Ore Microscopy” (COM) later renamed to “Commission on ore Mineralogy”. Its purpose was the development of reliable measuring procedures (including spectral photometer equipment, standards, polishing methods, etc) and the publication of quantitative data. The most important publication is the well known QDF-III (Criddle & Stanley, 1993)

On the basis of the IMA-COM recommendations, commercial microscope photometers and reflectance standards became available. They use grating monochromators or continuous interference filter slides either in the illumination or in the observation ray path of the polarizing reflected light microscope. Normally a photomultiplier is used for light intensity detection. Measurements were performed in the visible light, sequentially in steps of 10 or 20 nm from 400 nm to 700 nm. These instruments were fully automated but expensive. In 1987 Bernhardt introduced a low cost automatic photometer which used 16 interference line filters with peak wavelengths between 400 nm to 700 nm in steps of 20 nm (as recommended by the IMA-COM). Identification procedures on the bases of spectral reflectance were developed by Atkin & Harvey (1979), Bernhardt (1982) and Gerlitz et al. (1989).

A NEW SPECTROMETER TYPE

During recent years the development of high sensitivity CCD cameras made extreme progress. As a by-product, monolithic CCD-based so called mini-spectrometers with a great variety of possible applications are now available. They combine a diffraction grating and an array of CCD light sensors as well as appropriate optics and electronics. One of them, HAMAMATSU C10083CA was connected using a glass fibre-optic cable with a Leitz Orthoplan reflected light microscope. Its polarizing filter was replaced by a polarizing prism to increase the light intensity. A special measuring field diaphragm was inserted, and an inclinable substage was positioned on the normal rotatable stage. The fibre-optic cable, which is connected to the spectrometer, is mounted in a modified ocular of the microscope trinocular. The spectrometer is controlled by a normal low cost laptop via USB. An easy to use program for measurement and data processing was developed. The new spectrometer type offers a spectral resolution of better than 0.5 nm, thus more than 700 reflectance values are simultaneously obtained between 400 nm and 700 nm. The diameter of the sample region is about 10 µm when a 20X objective is used. The sampling time for one curve is about one to two minutes. Following the suggestion of Stanley (personal communication) sulvanite (Cu_3VS_4) with its unusual curve shape was used for a test. The spectrum is depicted in fig. 1. It is obvious, that there is no disadvantage using the new spectrometer type but the advantage of higher spectral resolution. The visible small differences in reflectance may be a result of chemical differences.

The CCD-based spectrometers are designed for use between 200 nm to 1000 nm. First tests have shown that it is possible to perform reflectance measurements between 400 nm and about 1000 nm. No specular reflectance values of this specific spectral region are published until today. It is expected that the extension to near IR improves the identification possibilities for those minerals which cannot be clearly distinguished in visible light.

As conventional heat filters have high light absorption in the near IR, they must be either completely removed (if possible), or replaced by filters which are only absorbing at wavelengths higher than 1000 nm. A second problem arises by the chromatic aberration of refractive microscope objectives because they are only corrected for visible light; thus they prevent from measuring small mineral grains. The focus point significantly moves between 700 nm and 1000 nm wavelength. Due to the fact that light of all wavelengths is detected simultaneously, focus point correction is not possible, as it is for sequential measurements. Tests have shown that mirror objectives, which have no chromatic aberration, can solve the situation. A third problem is the fact that no IMA-COM approved standards are available for NIR or near IR spectra, therefore a device for the (re)calibration of standards is in preparation.

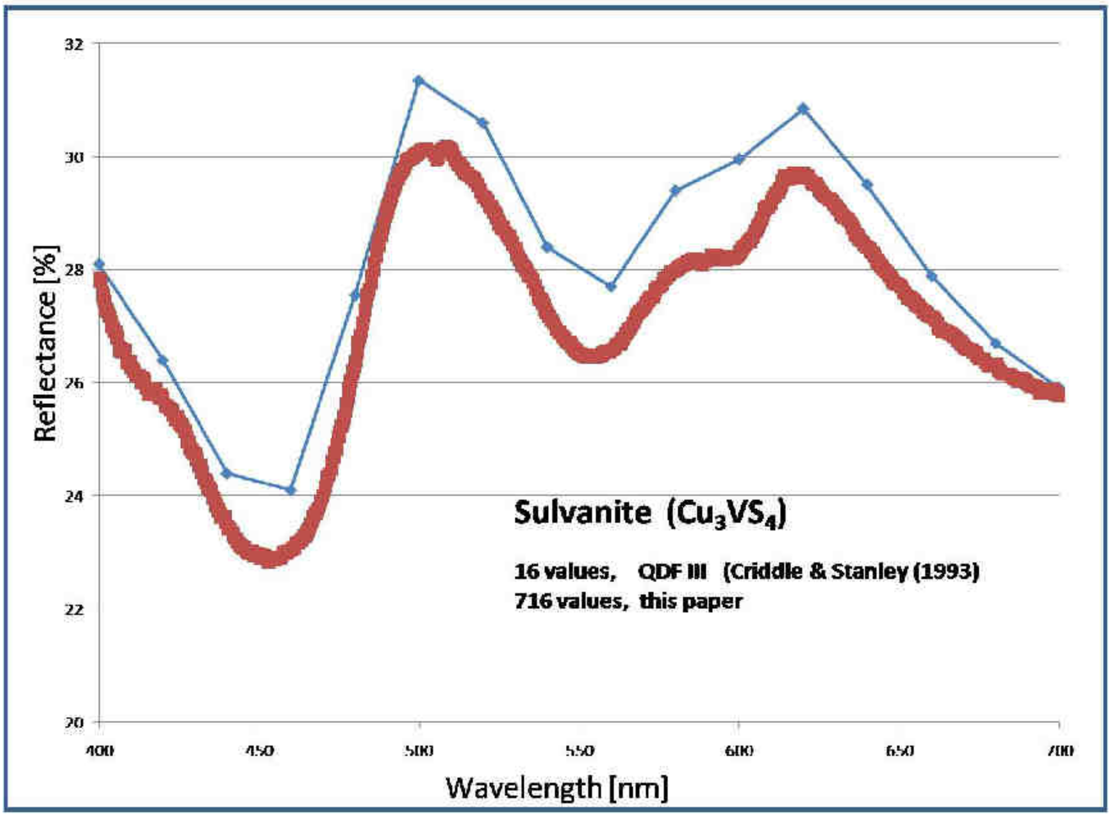

Fig. 1: Sulvanite measured with a conventional and a CCD-based spectrometer

CONCLUSION

Compared with other methods like electron microprobe or x-ray-diffraction, optical microscopy is a technically simple and most economic method for ore mineral identification. Quantitative methods, mainly the determination of reflectance spectra combined with conventional observations improve the reliability of identification results. The new CCD-based spectrometers are easy to use, have very high spectral resolution and offer the possibility to extend the measurable wavelengths region to the near IR. This promises to improve again the determination reliability. That's why it would be very desirable to have a spectra collection of ore minerals from 400 nm to 1000 nm comparable to the QDF III, which is one of the aims of the CAMEVA Project (Castroviejo et al., 2008).

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