Automated characterization of metal ores by multispectral reflected light microscopy

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ABSTRACT

This paper describes an innovative multispectral reflected-light microscopy system that provides quantitative mineralogical information comparable to that obtained from automated mineralogy systems based on SEM-EDS (Scanning Electron Microscopy with Energy-Dispersive X-Ray Spectroscopy), but at a fraction of the cost and with less stringent environmental requirements.

The system is built on a research-grade motorized optical microscope, which has been adapted to allow the acquisition of multispectral images of specular reflectance beyond the visible spectrum. A software application controls the different elements of the system and performs automated scans on polished sections, guaranteeing the accuracy and repeatability of reflectance measurements and a precise registration between image bands. The basic system acquires up to 20 reflectance bands spanning the VNIR (visible and near infrared) range of the spectrum (between 350 and 1000 nm), plus an optional fluorescence band to distinguish gangue and resin in polished thin sections prepared by a specific procedure from milled ore samples. An advanced version is able to capture additional reflectance bands in the SWIR (short-wave infrared) range (between 1100 and 1700 nm), to improve discrimination between some minerals.

After acquisition, another software application processes the images of a sample and automatically identifies the mineral corresponding to each pixel according to its multispectral reflectance values, by comparison to a built-in spectral reflectance database, producing a map of the mineral phases in each image. These maps allow computing the modal analysis, and to isolate particles and mineral grains, which are then processed to achieve a complete microscopic characterization of the sample.

Results of pilot tests of the system on samples from several copper mines are discussed in the paper.

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INTRODUCTION

The optimization of processes for the industrial use of mineral resources requires today a quantified, precise and rapid mineralogical characterization, basic support for Geometallurgy (s. Becker et al, 2016; for optical microscopy, Pirard, 2016). The traditional tool for studying metal ores, the reflected-light microscope, has been partially relegated in recent years by automated mineralogy systems based on Scanning Electron Microscopy with Energy-Dispersive X-Ray Spectroscopy (SEM-EDS), which are more expensive and not always reliable, but offer high performance. However, automated optical microscopy can provide comparable (and even more reliable, in some cases) performance at a significantly lower cost and with less stringent requirements for infrastructure. This is proved by the system now being presented, called AMCO (Automated Microscopic Characterization of Ores), the result of a recent R&D project funded by EIT RawMaterials.

The design of automated industrial applications for ore microscopy has been a constant endeavor of the Applied Microscopy Laboratory (LMA) of the Madrid School of Mines (Escuela Técnica Superior de Ingenieros de Minas y Energía), Universidad Politécnica de Madrid (UPM). An outstanding result of this effort was the CAMEVA System [Castroviejo et al. (2009), Catalina and Castroviejo (2017)].

The objective of the CAMEVA System was to **automate the identification and quantification of the most common ores of industrial interest** through the analysis of digital images of polished sections acquired with a fully-motorized reflected light microscope (**RLM**). The classical RLM study of ores relies on polarized light to define optical properties bound to crystal anisotropy and therefore to the orientation of the mineral particles studied, to be checked rotating the stage. However, the large motorized XY stages used to scan polished sections cannot be rotated. Oil immersion was also ruled out, due to difficulties in avoiding image artifacts caused by air bubble entrapment. Therefore, **the identification of the minerals was based on the measurement of their specular reflectance, R**, with neither polars nor immersion, **in the VNIR** (visible & near-infrared) range by acquiring multispectral images of polished sections. The measured R values are consistent and, as predicted, intermediate between the published minimum and maximum values of QDF3 (Criddle and Stanley, 1993).

The AMCO System, upgrading CAMEVA, has been developed within KAVA Upscaling project AMCO (EIT Project No. 15039, 2016-2018), led and coordinated by UPM, with the participation of the partners Université de Liège (ULiège, Belgium), the mining companies Cobre Las Cruces (CLC, Spain) and KGHM Polska Miedz (KGHM, Poland) and the SME ThinSectionLab (TSL, France).

The specific objectives of the AMCO project were:

- To improve the discrimination of minerals by increasing the number of bands in the visible range and extending the spectral range to the near ultraviolet (from 350 nm to 1000 nm), and, optionally, to integrate an auxiliary camera and filter wheel assembly to acquire additional bands in the SWIR (short-wave infrared, between 900 and 1700 nm).
- To build a multispectral specular reflectance database collecting precise measurements of specular reflectance for each of the considered minerals, covering at least the VNIR range (ideally between 350 and 1000 nm), with the possibility of extending it to the SWIR.

- To develop advanced classification methods to identify the mineral corresponding to a pixel in a multispectral image by matching its specular reflectance values to those in the database.
- To apply these classification algorithms to generate a map of the distribution of minerals in each image that allows to compute the modal analysis of a sample and carry out particle size, liberation and composition analyses.
- To develop a system prototype suitable for industrial use, with a user-friendly interface that facilitates the learning of ore microscopy, making it also useful for university teaching and for lifelong education (LLE).
- To validate the system with statistical analyses, comparison with external data and tests on real industrial problems.
- To reach a TRL (Technology Readiness Level) of 7-8.

Most of these objectives were achieved during the AMCO project, and the rest have been completed and enhanced through two Start-Up & SME Booster projects.

Therefore, a system for automated microscopic characterization of metal ore samples is now available that can be used in an industrial laboratory without the need for an expert in ore microscopy.

METHODOLOGY

The AMCO System is a multispectral microscopy system that allows the acquisition and processing of images composed of a number of spectral bands of specular reflectance covering the VNIR range. It consists of an **instrument** and two specific **software** applications.

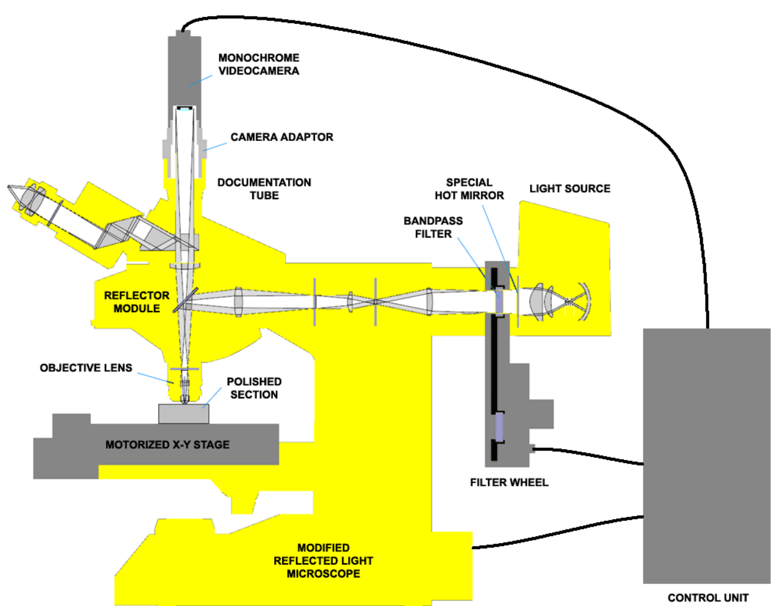

Figure 1 Schematic view of the instrument used in the AMCO System

The **instrument** is based on a research-grade fully-motorized reflected light microscope, whose halogen light source has been adapted to allow the acquisition of images beyond the visible spectrum. The microscope is fitted with some auxiliary elements (a filter wheel with a large number of bandpass interference filters, a high-resolution monochrome video camera and a high-precision motorized XY stage), all of them controlled from a computer. Figure 1 shows a schematic view of the instrument, where the auxiliary elements are depicted in dark gray.

The AMCO System **software** is arranged as two independent applications:

- *amcoCapture* is the program that controls all the elements of the instrument and performs the acquisition of series of multispectral images of fields of a polished section, guaranteeing the accuracy and reproducibility of the reflectance measurements and a perfect registration between all the bands. Images can be captured one by one in manual mode, or automatically by the unattended scanning of the area specified by the operator on the preparation.
- *amcoAnalysis* is the program that allows the operator to examine the images of a series and perform different types of analysis on them. The operator can select the image displayed on the screen, viewing individual bands or linear combinations of them in true or false color, zoom in to examine details, adjust levels, gamma, etc. Polygonal zones can be delimited interactively on the images, in order to perform manual analysis: the mineral corresponding to a zone is identified by comparison of its multispectral specular reflectance values with the database of considered minerals. The program can automatically classify all the pixels of an image, obtaining the modal analysis and the map of the distribution of the mineral phases of the field. It can also automatically process all the images of a series, obtaining the modal analysis of the whole sample. Finally, for polished thin sections of milled ore samples prepared with fluorescent resin, it can perform particle size analysis, as well as modal, liberation and particle composition analysis, by particle size fractions.

The methodology adopted in the AMCO System to carry out the characterization of a sample consists of two stages:

- In the first stage, a polished block (**PB**) or a polished thin section (**PTS**) prepared from the sample is loaded into the microscope and the *amcoCapture* program is used to acquire and save to disk a series of multispectral specular reflectance images of microscopic fields.
- In the second stage, the *amcoAnalysis* program is used to process the series of images stored on disk and perform the analyses that allow the characterization of the sample.

The reason for dividing the process into two separate stages is that image acquisition requires the exclusive use of the microscope and takes much longer than analysis, even if it has to be performed only once, whereas processing a series of images is usually an iterative process, in which the minerals selected for consideration and the parameters used are gradually fine-tuned.

Another advantage derived from this separation is that it allows multiple instances of the analysis program to be run simultaneously, since it does not make use of the microscope. Thus, it is possible to process several samples concurrently, even on different computers.

Particle analysis of milled ore samples requires the preparation of PTS with a fluorescent resin dye, according to a specific procedure (Grunwald-Romera et al., 2019) developed by LMA-UPM to enable the AMCO system distinguish gangue minerals from the epoxy resin. In this way, a fluorescence image can be obtained that allows masking of the resin to isolate the ore particles.

SYSTEM DEVELOPMENT

The AMCO System prototype is built on a Leica DM6000 M reflected light microscope, with PL FLUOTAR 20x, 10x and 5x objectives, a L5 ET k fluorescence filter cube and a 12V/100W halogen light source. Since the original hot mirror of the light source blocked the IR, it was replaced by a custom hot mirror having partial transmittance in the IR, to allow acquisition of NIR and SWIR bands. The microscope incorporates a large filter wheel containing 20 hard coated bandpass interference filters with center wavelengths (CWL) ranging between 370 and 1000 nm, a 2.3 MP monochrome video camera with CMOS sensor and USB3 interface, and a high-precision scanning stage that can hold up to 6 polished sections. Everything is controlled from a PC workstation with a large monitor. An advanced version of the prototype has also been developed to acquire additional bands in the SWIR, incorporating a motorized dual camera adapter, a SWIR InGaAs camera with GigE interface and an additional filter wheel holding hard coated bandpass interference filters with CWL between 1100 nm and 1600 nm. Figure 2 shows this latter system in action.

Figure 2 View of the advanced version of AMCO System prototype at the LMA

The multispectral images acquired by the AMCO System are typically composed of 21 bands: 13 reflectance bands in the visible range from 400 to 700 nm with 25 nm bandpass (**BP**), 6 reflectance bands in the NIR from 750 to 1000 nm with 50 nm BP, a reflectance band in the NUV centered at 370 nm with 36 nm BP, and an optional fluorescence band obtained with the L5 ET k cube. The advanced version can acquire 6 additional reflectance bands in the SWIR from 1100 to 1600 nm with 50 nm BP.

As an essential tool for mineral identification, a multispectral specular reflectance database has been compiled by collecting specular reflectance data in the VNIR range (between 370 and 1000 nm) from a set of selected samples of each mineral. To characterize the deviation of minerals' reflectance, the specular reflectance values of each and every pixel within the image zones tagged for learning, manually delimited on randomly oriented mineral grains, were included in the database.

The selection of the minerals included in the database has taken into account their abundance and their industrial interest, with 40 species forming the initial group (Figure 3), that later expanded to about 80. However, the database is open to the addition of new species by the operator, and currently contains over 100 minerals (mostly ores, but some common gangues have been also included). The database has been validated by comparing average values with published specular reflectance data (available only in the visible and under polarized light) such as QDF3 (Criddle and Stanley, 1993).

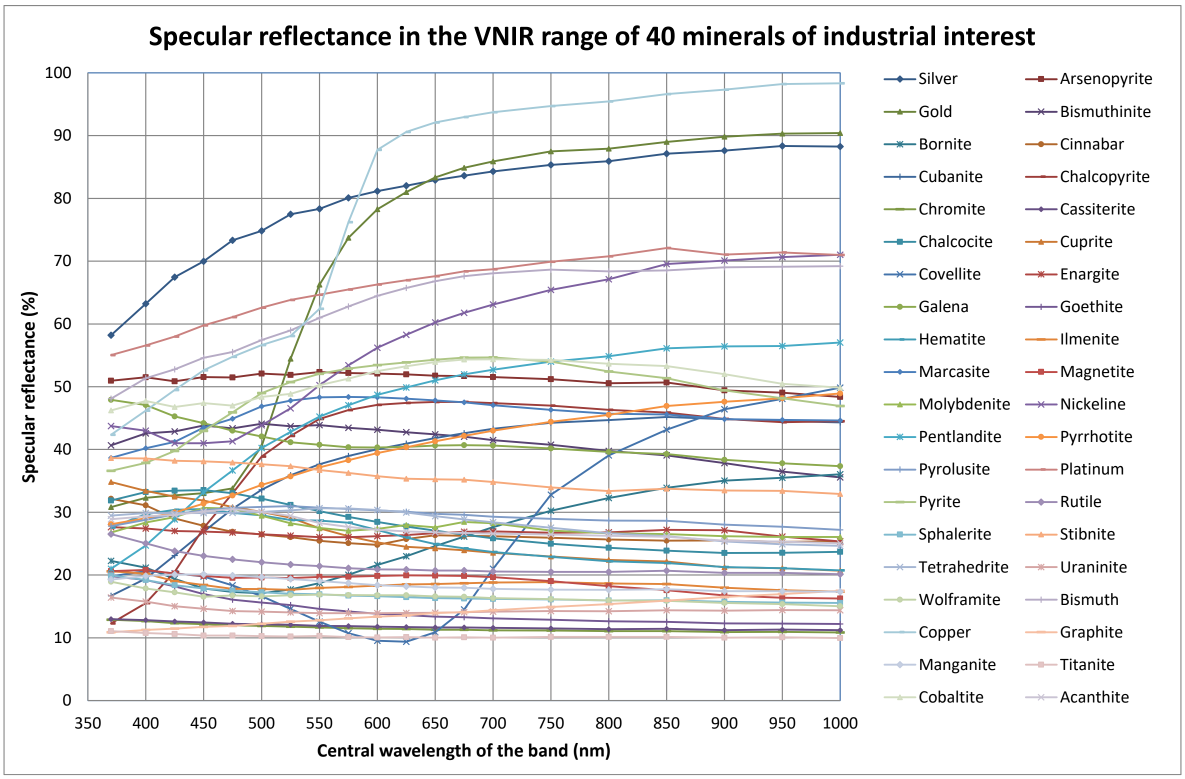

Figure 3 Graph displaying average multispectral specular reflectance of a subset of the database minerals

An auxiliary custom database has been included in the system to comply with natural variations due to peculiarities of certain ores. This is important, because the spectral response of a mineral can vary with the composition (as analyzed in López-Benito et al. (2017)), crystallinity, tarnishing, surface state, etc., as well as with the quality of the polish.

Automated ore identification is achieved by applying advanced classification techniques based on the specular reflectance values of the multispectral image bands, which are compared with the values collected in the database (López-Benito et al. (2020)). Identification is currently performed pixel by pixel, obtaining highly accurate results in the classification of uniform and well-polished ore grains, although results tend to be less reliable in areas adjacent to grain boundaries or with poor polish. Most gangue minerals are grouped into a single class, as their reflectance ranges overlap widely.

RESULTS AND DISCUSSION

A standard PTS can be adequately characterized through the analysis of 10%-20% of its effective area, corresponding to about 120-250 images of a 20x objective for typical PTS sizes. As the automated focusing and capture of an image takes less than one minute, it can be estimated that the acquisition of a representative series of images of a sample would require between two and four hours.

The performance of the AMCO System has been validated through the analysis of PTS prepared by ThinSectionLab from ore samples provided by CLC and KGHM: a set of 18 PTS have been analyzed with AMCO at LMA and with ZEISS Mineralogic Mining at Universitè de Liège. Modal analysis results from both systems are grossly comparable, although there are a number of differences:

- AMCO cannot reliably distinguish gangue minerals, and provides a gangue group value.
- AMCO tends to provide higher gangue contents, but the ratios between ores remain similar.
- AMCO is able to reliably identify minerals with identical or similar composition that cannot be distinguished by SEM-EDS (pyrite/marcasite/melnikovite, digenite/chalcocite/djurleite, magnetite/hematite/goethite, etc.). This advantage can be important in some applications.
- AMCO is less reliable with intermediate compositions in isomorphic series.
- AMCO can analyze many more pixels than SEM-EDS in a given time.
- The percentage of unclassified pixels is much lower for AMCO than for SEM-EDS.

Pilots tests of the AMCO System have been carried out at several mines of the Iberian Pyrite Belt.

CONCLUSION

The AMCO System is a high-performance and affordable automated ore microscopy tool, useful both for geometallurgical characterization applications in mine plants, laboratories or research institutions and for educational purposes in higher education.

The results obtained show that the AMCO System achieves reliable identification and quantification of ores with a performance comparable to that of the systems based on SEM-EDS, surpassing them in some cases, such as the distinction of minerals with identical or similar composition (iron ores, iron sulphides, copper sulphides, etc) or the identification of graphite.

Therefore, the AMCO System represents a real alternative (not only competitive, but also complementary) to the existing Automated Mineralogy systems based on SEM-EDS.

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