

Characterisation of Ore Properties for Geometallurgy

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Large-scale mapping of drill core for cobalt minerals and their textures using scanning micro-XRF technology.

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The successful implementation of geometallurgy largely depends on the continuous collection of high-quality, multi-scale, multi-dimensional quantitative data on the geology, geochemistry, mineralogy, texture, and physical properties of an orebody. This can then be used to build and improve, amongst other things, ore deposit models, comminution strategies, waste management, and downstream mineral processing and extractive metallurgy routes. The present contribution provides a brief overview of the key types of data collected, and analytical techniques used, in geometallurgical programs. It also highlights important developments that are currently underway, which may generate a significant impact in the near future.

KEYWORDS: mineralogy; texture; elemental deportment; liberation; ore characterisation

INTRODUCTION

Before the invention and implementation of the geometallurgical approach, the exploration and mining industry had relied largely on a traditional element-based methodology to evaluate commercially important mineral deposits. This involved a simple but cost-effective approach whereby samples were submitted for chemical assays (e.g., XRF, ICP-OES, ICP-MS, see TABLE 1 for abbreviations) for the elements of interest. For example, in a gold exploration program, key elements such as Au, Ag, and As would be of main concern, while Pb, Zn, Fe, Ag, and As would be important for a Pb–Zn deposit. Only the most essential elemental assays were commissioned, largely to save time and money. Whilst this was considered practical at the time, and often worked to some degree for most ore bodies, ore characterisation is best served by more than just chemical assays. This is because, at the fundamental level, we mine minerals and not elements; in addition, we process complex particles and not pure mineral grains. This leads to the inevitable conclusion that, to fully understand, predict, and improve the performance of an ore during its life cycle from extraction to marketable products and waste, the geology, mineralogy, texture, elemental deportment, and breakage characteristics must be known and their effects on processing well understood.

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A cursory look at geometallurgy may suggest that it is simply what was previously called ‘ore characterisation’, but it is far more than that today. Technology has developed in the last ~20 years such that ores can now be imaged and analysed in 2D and 3D, at high resolution, and in practical time frames, providing all the key parameters to optimise mineral processing and extraction. There are many types of samples that can be investigated with modern analysis and imaging methods for geometallurgical purposes. Mobile or hand-held devices can be used in the field for in-situ

material (e.g., outcrop, mine-face). Once removed from their geological and physical context and transported to a mineral processing plant or analytical laboratory, ex-situ samples can be examined online, in-line, and in 3D- and 2D-sections (Dehaine and Esbensen 2022).

MINERAL AND ORE GEOMETALLURGICAL PROPERTIES

We often make the distinction between primary ore properties (intrinsic to the ore), as opposed to secondary properties (describing ore behaviour during processing; Frenzel et al. 2023 this issue). The current paper focuses on primary ore properties as measured by geochemical and mineralogical analysis, while secondary ore properties, which are quantified as the response to a geometallurgical test and are used to predict the performance of the mineral processing and metallurgical operations, are described in Pereira et al. (2023 this issue). Both primary and secondary properties of ores are directly linked to the minerals making up the ore and their respective mineral properties. While minerals are characterised by numerous different properties, only a few are relevant for mineral processing and metallurgy: hardness, density, magnetic susceptibility, electrical conductivity, hydrophobicity, and chemistry (cf. Pereira et al. 2023 this issue; Chetty et al. 2023 this issue; FIG. 1).

Key ore properties, which need quantification before processing, include elemental assay, modal mineralogy, grain size, grain shape, mineral associations, elemental deportment, and the presence of deleterious minerals (e.g., clays, asbestiform minerals, or minerals containing elements that cause contamination or harm to the environment (so-called penalty elements)). The same parameters

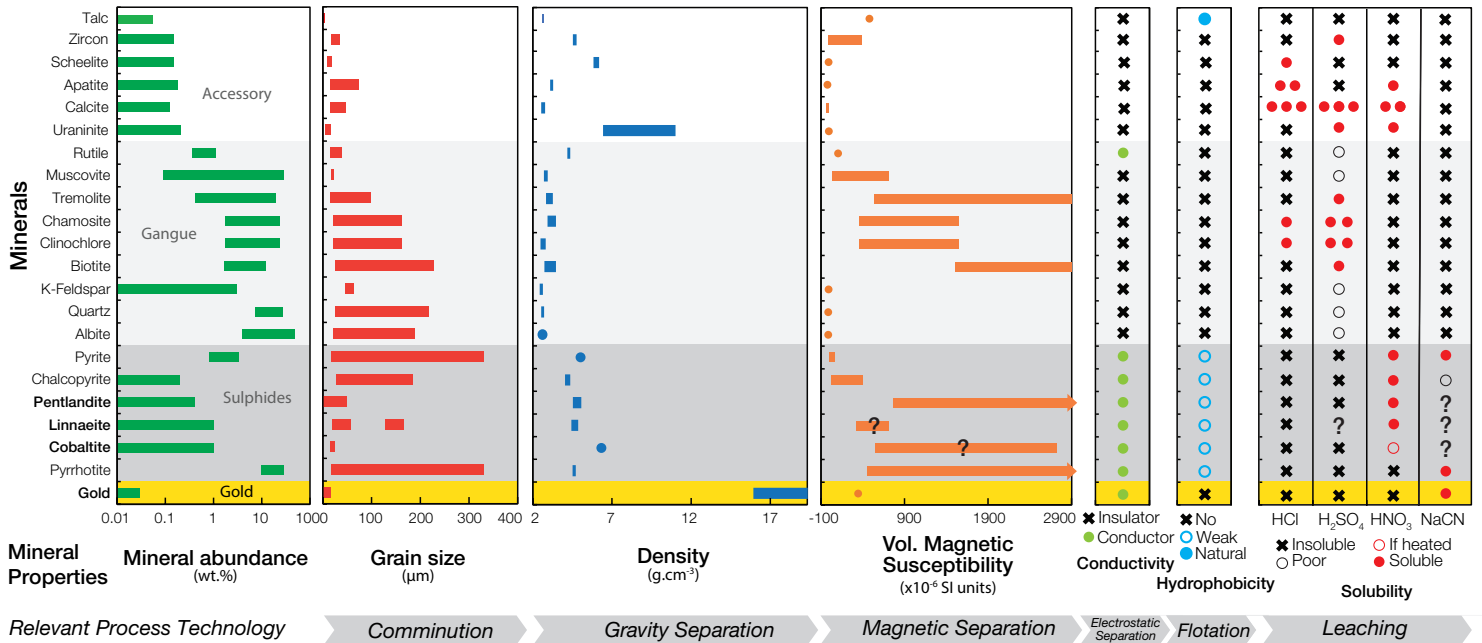


FIGURE 1 Illustration of the variability of some key mineralogical properties for a gold-cobalt ore deposit (target minerals/metals highlighted in bold) and their impact on relevant process technology. MODIFIED AFTER DEHAINE ET AL. (2021).

can be established for ore particles generated by comminution, along with particle shape and size, and degree of liberation of key phases (by surface area or volume). Finally, materials that have been subjected to further downstream physical and chemical processes can be scrutinised in detail for purity, losses, and potentially harmful minerals or elements (cf. Parbhakar-Fox and Baumgartner 2023 this issue).

MINERALOGICAL CHARACTERISATION

Methods of Mineral Characterisation

An overwhelming number of modern geoanalytical techniques are available for the imaging and analysis of Earth materials, including ore (TABLE 1). To evaluate them, it is often useful to consider the type of energy source on which they are based, as this determines their practicality, capabilities, and applications. For convenience, we consider the following types of sources (FIG. 2): visible and

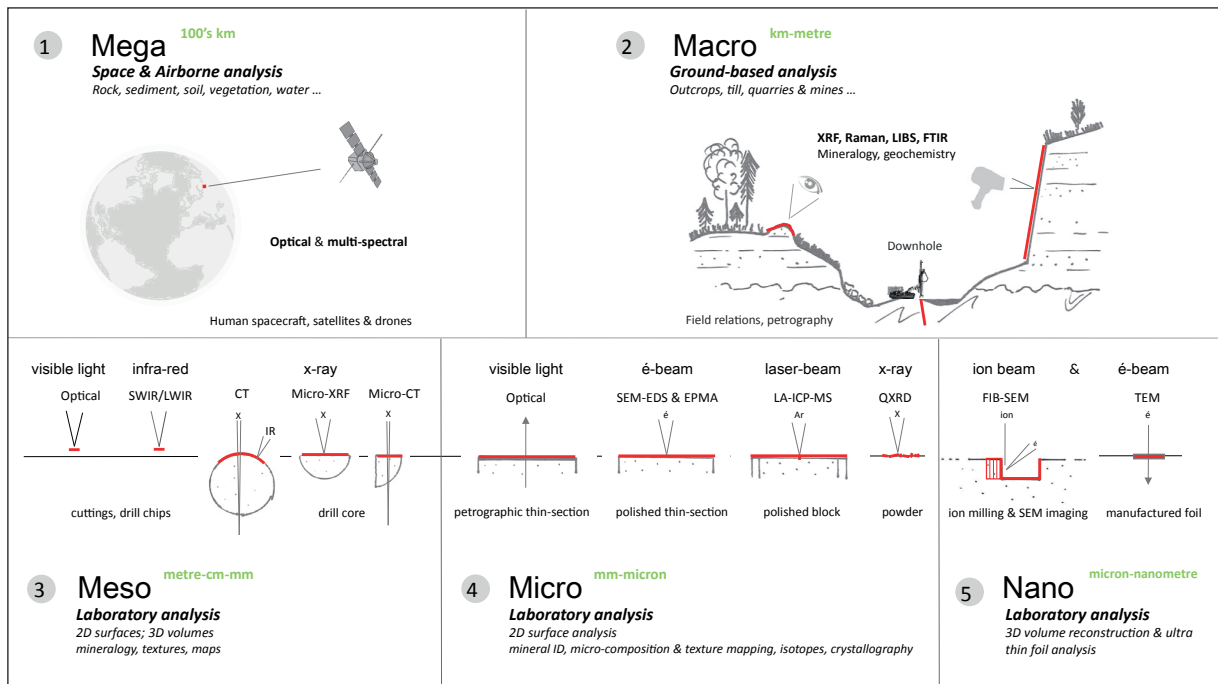


FIGURE 2 Multi-scale, multi-modal, and multi-dimensional characterisation methods used in geometallurgy. (1) **Mega** is at a scale of 100s of km as observed using space and

airborne techniques. (2) **Macro** operates at km to metre scales and is reserved for field-based observations. (3) **Meso**, (4) **Micro**, and (5) **Nano** are all laboratory-scale observations. MODIFIED AFTER BUTCHER (2019).

TABLE 1 NON-EXHAUSTIVE LIST OF KEY ANALYTICAL TECHNIQUES AND TESTING METHODS USED TO MEASURE PRIMARY ORE PROPERTIES IN GEOMETALLURGY.

Type	Method	Type ¹	Scale ²	Minimum Resolution ³	Typical LOD ⁴	Cost/Sample (US\$) ⁵	Properties characterised ⁶	Reference or example study
Geochemical <i>Bulk chemistry</i>	X-ray fluorescence (XRF) ^p	B	–	–	0.01% ^e	30	Major and minor element abundances	Dietrich LeVier (2019)
	Atomic absorption spectroscopy (AAS)	B	–	–	1–10 ppm ^e	15	Major and minor element abundances	
	Inductively coupled plasma-atomic emission spectrometry (ICP-AES)	B	–	–	0.01–1 ppm ^e	40	Major, minor, and trace element abundances	
	Inductively coupled plasma-optical emission spectrometry (ICP-OES)	B	–	–	0.1%–1% ^e	20	Major and minor element abundances	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	B	–	–	1 ppb–0.01 ppm ^e	40–60	Minor and trace element abundances	
	Instrumental neutron activation analysis (INAA)	B	–	–	1 ppb–0.01 ppm ^e	20–50	Major, minor, and trace element abundances	
Mineralogical <i>Mineralogy and texture</i>	X-ray diffraction (XRD) ^p	B	–	–	0.1–5 wt.% ^m	100–300	Mineral identification, mineral abundances (major, semi-quantitative), crystal structure, amorphous content	Parian et al. (2015)
	Hyperspectral imaging (HSI) ^p	S	Meso to micro	25 µm	–	10–50 / m	Imaging, textures, mineral abundances (major)	Johnson et al. (2019)
	Fourier transform infrared spectroscopy (FTIR) ^p	S	Meso	10–20 µm	1–5 wt.% ^m	15–50	Mineral identification, mineral abundances (major)	Dehaine et al. (2022)
	Micro X-ray fluorescence (µ-XRF)	S	Meso to micro	4–20 µm	10–100 ppm ^e	100–500	Imaging, 2D textures, element and mineral abundances, grain size, associations, and liberation	Haschke (2014)
	Optical microscopy (OM)	S	Micro	0.1–10 µm	–	100–500	Imaging, mineral identification, textures	Pirard et al. (2007)
	Scanning electron microscopy (SEM)	S	Micro	0.5–4 nm	0.5%–1% ^e	200–600	Imaging, mineral identification, textures, mineral chemistry (semi-quantitative)	Hartner et al. (2011)
	Raman microscopy/Micro-Raman (µ-Raman) ^p	S	Micro	1 µm	1–5 wt.% ^e	20–50	Imaging, mineral identification, textures	El Mendili et al. (2019)
	Automated Mineralogy (AM)	S	Micro	5–10 µm	0.01–1 wt.% ^m	200–600	Imaging, 2D textures, mineral abundances, grain size, associations, and liberation	Goodall et al. (2005)
	X-ray computed tomography (XCT)	3D	Meso to micro	1 nm to 100 µm	–	300–700	Imaging, 3D textures, porosity, mineral abundances (estimate)	Bam et al. (2016)
Mineralogical <i>Mineral chemistry</i>	Electron probe microanalysis (EPMA)	M	Micro	0.5–10 µm	50–300 ppm ^e	100–500	Major and minor elements in minerals	Frenzel et al. (2019)
	Laser ablation ICP-MS (LA-ICP-MS)	M	Micro	5–200 µm	1 ppb–10 ppm ^e	50–100	Minor and trace elements in minerals	Aylmore et al. (2018)
	Laser-Induced Breakdown Spectroscopy (LIBS) ^p	M	Micro	20–100 µm	1–5 ppm ^e	50–100	Major and minor elements in minerals, grain size, imaging	Fabre (2020)
Petrophysical	Equotip ^p	S	Meso	3 mm	–	NA	Surface hardness	Keeney and Nguyen (2014)
	Gamma-ray attenuation density (GAM)	B	Meso	–	0.01 g	~50	Bulk density	Ross and Bourke (2017)
	Galvanic resistivity (MAFRIP)	B	Meso	–	1 × 10 ⁻⁶ Ωm	~50	Resistivity/conductivity	Vatandoost et al. (2008)
	Magnetic susceptibility ^p	B	Meso	–	<20 × 10 ⁻⁶ SI	~50	Volume magnetic susceptibility	Ross and Bourke (2017)
	Ultrasonic pulse velocity (UPV/p-wave) ^p	B	Meso	–	10 m/s	NA	Compressional (P) and shear (S) wave velocity	Vatandoost et al. (2008)

1 3D: 3-dimensional analysis, B: bulk analysis, M: micro-analysis, S: surface analysis.

2 See FIGURE 1.

3 Technical minimum resolution; minimum resolution used in practice would be higher.

4 Indicative Limit of Detection (LOD). LOD varies depending on the analyte (element or mineral), sample preparation (e.g., digestion methods, pellets versus fused beads), the material analysed (matrix effect), and operational parameters (including resolution).

5 Indicative cost, when available, based on laboratory testing company brochures and authors' experience. Price may decrease for higher sample numbers and increase if there is a need for an experienced operator.

6 Major, minor, and trace refer to element (or mineral) concentrations in the range of 0.5%–100%, 0.01%–0.5%, and 2–100 ppm, respectively.

p Technology for which equivalent handheld or portable technologies exist.

non-visible light (optical imaging, hyperspectral); X-rays (XRD, XRF, X-CT, micro-XRF); electron beams (SEM, SEM-EDS, automated mineralogy, EPMA), laser beams (Raman, LA-ICP-MS, LIBs, FTIR), and ion beams (FIB-SEM). See TABLE 1 for abbreviations.

Optical systems have been around since the invention of the petrographic microscope by Henry Clifton Sorby in 1848 and continue to provide a sound basis for mineral identification and textural descriptions. Many commercially important minerals can be readily identified optically. The big drawback to the technique is its dependence on the skills and output of the microscopist, and as with most single-exertion activities, the difficulty to upscale the output. There are also limitations to what the human eye can discriminate and record, which are not consistent between different individuals. It was on this basis that new microscopes were developed that offered operator-independent analysis with higher-magnification imaging capabilities, and higher productivity, such as the modern scanning electron microscope (SEM) developed in the 1930s.

In more recent times (1980s onwards), this has led to a new field known as automated mineralogy, which utilises both the imaging capabilities of the SEM along with the

possibility of conducting micro-chemical analyses along pre-defined lines, grids, or points (electron-based mineral identification, or SEM-EDS), allowing automated data capture on polished surfaces of solid samples or particulates at the micrometre-scale, and at a rate and quantity that is not humanly possible.

The further advantage of the SEM-based approach is that hundreds, thousands, and in some cases even millions of EDS spectra can be digitally processed both online and off-line using dedicated analytical software to provide quantitative mineralogical and petrographic data for use in geometallurgy. The main outputs are digital images of the sample under investigation (FIG. 3), where the composition of each pixel is known, as well as its associations with neighbouring pixels. Thus, modal mineralogy, as well as textural and chemical attributes, can be determined on a pixel-by-pixel basis. Surface mineral maps can be created for polished thin-sections or polished sample blocks up to decimetre-size, providing mineralogy within a textural context. Particle-by-particle analysis is also possible for particle mounts of crushed and processed samples (FIG. 4). Given the high level of measurement automation, statistically valid measurements can be undertaken, which

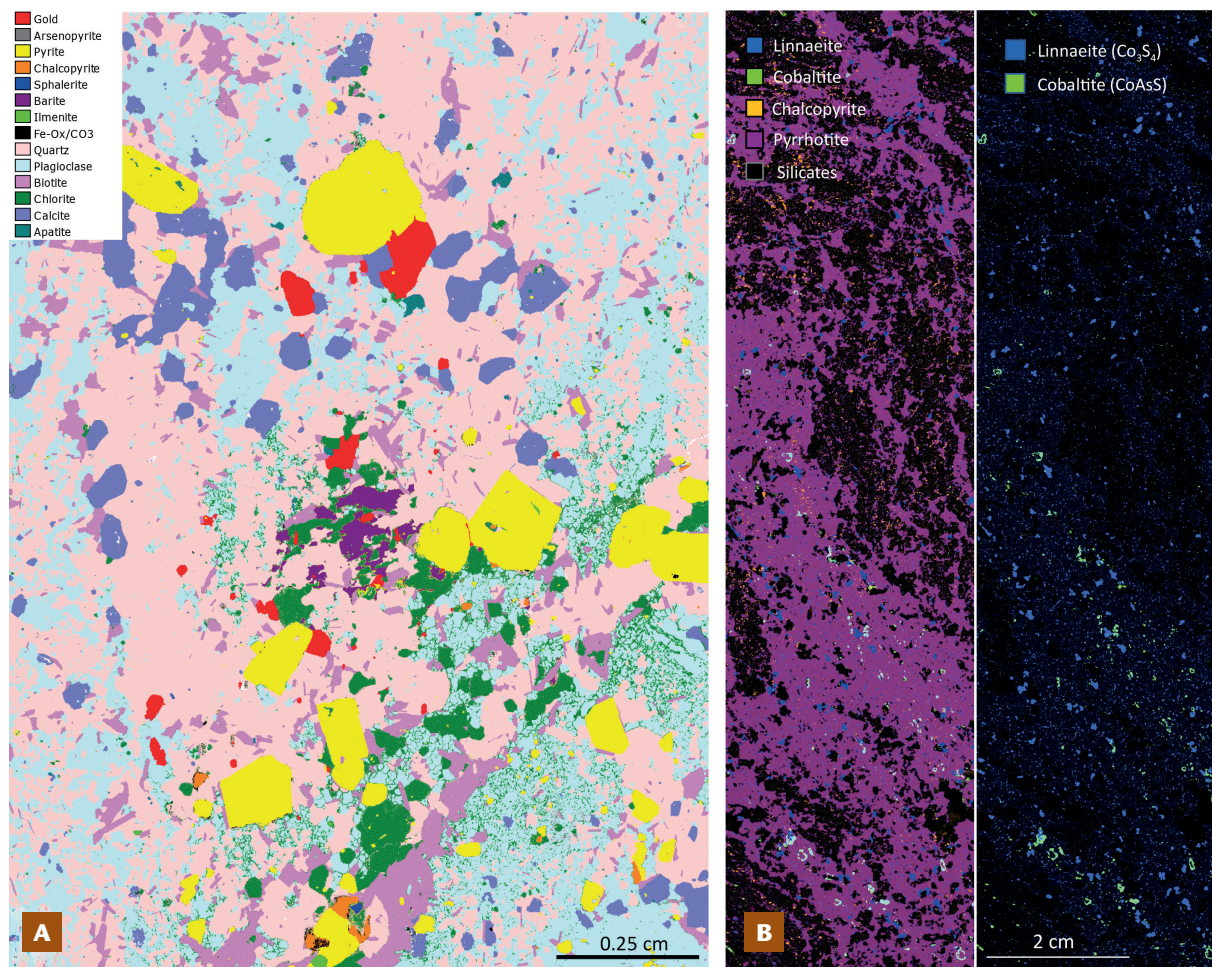


FIGURE 3 Textural variability in ores captured at different scales: **(A)** Mineral map of a polished thin section of a gold-bearing ore illustrating the degree of detail obtainable from SEM-EDS-based automated mineralogy technology. This digital information yields information on gold grain size and association, as well as the presence of other minerals of economic interest (chalcocopyrite, sphalerite), and penalty minerals (arsenopyrite). Estimates of optimum grind size to liberate the gold can be obtained. Resolution: 1 $\mu\text{m}/\text{pixel}$.

(B) Mineral map of a half drill core obtained with scanning micro-XRF, typical of a cobalt-bearing ore type, displaying a metamorphic foliation that controls the distribution of the mineralisation. Two types of cobalt-bearing minerals, cobaltite and linnaeite, can be distinguished based on their mineral chemistry. Resolution: 10 $\mu\text{m}/\text{pixel}$.

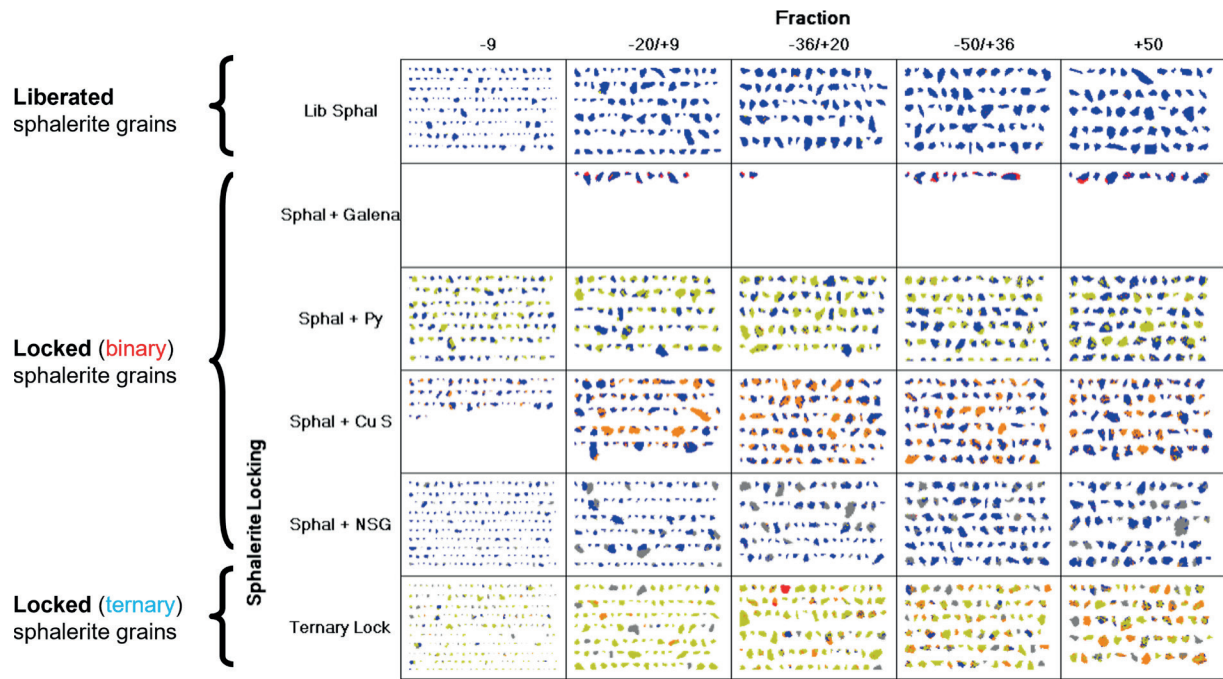


FIGURE 4 Example of digital particle-by-particle classification based on particle properties or combinations of properties (e.g., composition, texture, sample location). Colour legend: blue = sphalerite; red = galena; khaki = pyrite; orange =

chalcocopyrite; grey = non-sulphide gangue (NSG). Binary refers to two phases locked together as a single particle; ternary refers to three different minerals that are locked together; CuS refers to covellite. Fraction sizes are in μm . BASED ON AN IMAGE FROM FEI.

provide quantitative estimates of mineral mode, particle-grain sizes and shapes, liberation, association, elemental deportment, and calculated assay.

If upscaling of textural and mineralogical characteristics from the micrometre- to the metre-scale is of interest, hyperspectral methods are now also available and widely used, e.g., visible-near infrared (VNIR) and shortwave infrared (SWIR), and can be deployed in combination with other technologies. Specifically, scanning micro-XRF is now available with mineralogical and elemental capabilities such that drill cores, for example, can be evaluated at micrometre scales over centimetre to metre distances, either directly on the curved surface, or on a 2D cut surface. Thus, a core pulled straight from the ground (and ideally orientated appropriately) can be used to generate first estimates of mineralogy and potential liberation behaviour of the commercially important minerals in an ore before further (usually destructive) testing is performed.

Stereological Bias in 2D Measurements

Most of the above-mentioned techniques rely on 2D image analysis, which is the current industry standard. However, when measuring 3D objects via 2D sections, a stereological bias inevitably occurs (Gottlieb et al. 2000). This phenomenon mostly concerns the estimation of apparent grain size and apparent liberation (FIG. 5). The sectioning of grains in a polished mount always results in a 2D sectional view where the apparent grain size is less than or equal to the true size of the grains (FIG. 5A; Sutherland 2007). Similarly, the measured apparent liberation in 2D measurements *overestimates* true 3D liberation (Spencer and Sutherland 2000). Indeed, a liberated grain always appears liberated in sectional views, whereas locked grains can appear to be liberated or locked in sectional views depending on where the section cuts the particle in which they are locked (FIG. 5B).

The magnitude of the stereological bias for liberation estimates will vary with particle size, texture, and actual liberation. The bias will be important for particles with a simple texture (e.g., binary or ternary particles, as in FIG. 5B), but virtually negligible for fully liberated grains (e.g., FIG. 5A). The effects of stereological bias can be controlled by careful sample preparation, including sizing of the samples and mounting of the size fractions in distinct polished mounts for liberation analysis (Spencer and Sutherland 2000). While some authors have suggested that the effects of stereological bias are minimal in most real cases (e.g., Petruk 2000), it is not trivial to assess how strong its effect will be in any specific case. Therefore, some authors have developed correction methods to account for the effects of stereological biases (Gay and Morrison 2006).

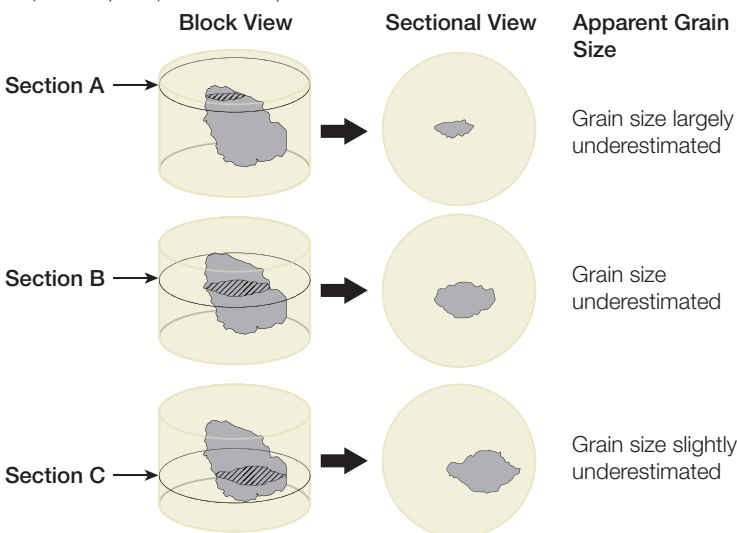
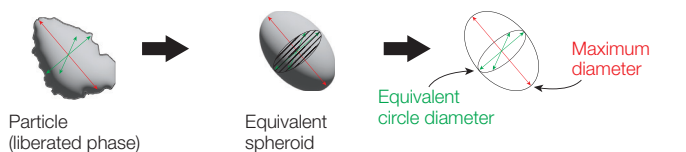
However, only 3D techniques such as X-ray computed tomography (XCT) may ultimately overcome the effects of stereological bias. Indeed, XCT has the ability, if the density contrast between minerals is suitable, to directly measure the 3D liberation of grains. The method does, however, have some limitations in terms of spatial and phase resolution (TABLE 1), and the liberation of small grains in a complex particle may be difficult to resolve.

Sampling

The granular nature of geological and processing samples may introduce significant sampling errors. If not controlled for, these may be orders of magnitude higher than the typical analytical errors of the analytical techniques described above. Obtaining a representative sample is therefore of paramount importance in ore characterisation, as no amount of measurement, even with many different techniques, can compensate for inadequate sampling. While we do not have space to delve into this topic more deeply here, the Theory of Sampling (TOS) developed by Pierre Gy (1998) provides a comprehensive introduction to this issue, including protocols for the sampling and preparation of geological materials to ensure representative results.

A Grain/Particle Size Bias

3D View

**B** Liberation Bias

3D View

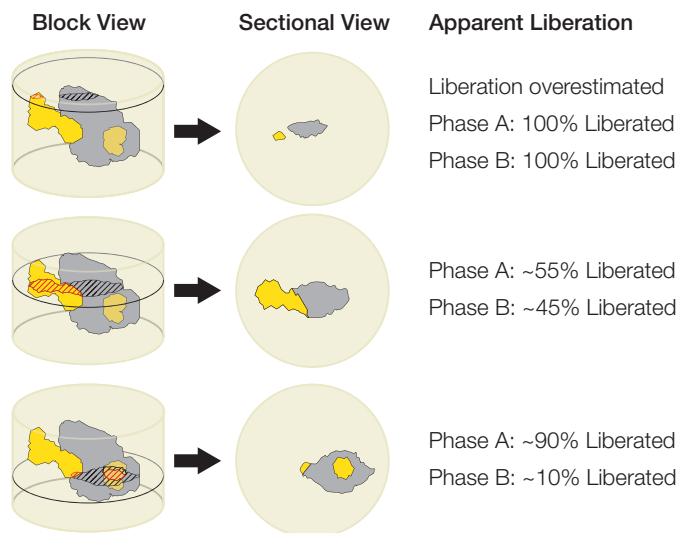
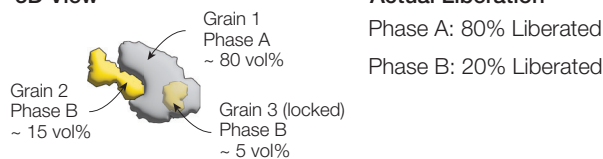


FIGURE 5 Illustration of the stereological bias resulting from different cross-sections through a block for the estimation of (A) grain size and (B) liberation.

DATA INTEGRATION OVER DIFFERENT LENGTH SCALES AND MODALITIES

It is common practice in other disciplines of science and engineering, where complex imaging and analysis are required, to create what are known as *workflows*. This is a concept where samples are examined in a particular order using a variety of techniques, providing multi-scale, multi-modal, multi-dimensional, and multi-disciplinary information. Geometallurgy is ideally suited to adopt this approach given that rock textures and ore types are developed at different scales of observation. We might, for example, need to know information at the grain boundary-scale (for liberation or processing behaviour), as well as at the ore deposit-scale (for resource modelling). It is only by using the workflow approach that we can bridge these important scales of observation.

The order in which the different technologies and methods are applied usually follows the scale, from macro to micro, and from in-situ non-destructive methods to bulk or destructive methods. The choice of techniques depends on the type of information needed. Some workflows may include, for instance, two distinct methods that may seem redundant, like two (semi-) quantitative mineralogical analytical methods, e.g., XRD with Rietveld refinement (QXRD) and automated mineralogy. But each method has its advantages and limitations. XRD is often better at phase identification than automated mineralogy and furthermore can differentiate polymorphs like pyrite and marcasite. Typically, XRD cannot quantify amorphous phases (unless an internal crystalline standard is used), or minor and trace minerals below 1–3 wt.% abundance (TABLE 1). However, XRD phase quantification includes crystallites down to the nanometre scale, while SEM-based automated mineralogy systems typically only cover mineral grains down to 2 μm. Thus, both methods are complementary and should be used together.

It is also necessary to ensure in multi-method analysis that the methods are cross-validated and calibrated. This is achieved by taking a sample and analysing the same representative aliquot by more than one method and checking for the consistency of results, e.g., XRF, QXRD, Raman, and ICP-MS in the case of powders; or optical microscopy, SEM-EDS, micro-XRF, and EPMA, combined with image registration software, in the case of the 2D surface of a thin section or polished block.

In addition, the modal mineralogy data obtained by automated mineralogy can be combined with mineral chemistry data obtained by EPMA to back-calculate the bulk chemistry of each sample based on its mineralogical composition. This can then be compared to the measured bulk chemistry of the sample obtained by bulk chemical analyses (e.g., XRF and ICP-OES/MS) to cross-validate the results, and to refine modal mineralogy data, the accuracy of which is often an order of magnitude below those of chemical analytical methods.

The result of such an integrated multi-scale approach is to have full visibility of all minerals present over any relevant scale and, thus, knowledge of how they occur (FIGS. 1 and 2).

NEW TECHNOLOGIES AND FUTURE DEVELOPMENTS

Going forwards, geometallurgy will depend more (not less) on geological-, mining-, and minerals engineering-related data being made available. This will require continued development in the improvement of analytical techniques, the speed of data acquisition and processing, as well as data handling, archiving, and retrieval architectures for geologists, mineralogists, miners, and processors. The faster, cheaper, and more efficient collection of information will also enable better interdisciplinary collaboration along the processing chain.

Whole core scanners are already available that can produce a 3D archive of drill cores in practicable time frames. Hyperspectral scanning has also advanced in recent years, and now offers the possibility of near real-time analysis. Scanning micro-XRF has the potential to make mineral and texture maps of entire drill core trays and provide mineral and liberation information at, or near, the drillsite. Nevertheless, we still lack the ability to comprehensively scan drill cores and process the acquired data at a rate that keeps up with drilling and allows for immediate access to the processed and interpreted data for decision makers. Indeed, this is a field where active developments are occurring at the time of writing, and great improvements are expected over the next few years.

Based on these increasing rates of data acquisition for primary ore properties, and increasing diversity of available data types, geometallurgy will likely continue to contribute to the design of ever more sophisticated mineral processing plants. Ideally, geometallurgical campaigns should efficiently establish the mineralogical and textural signatures, including their variability, for the main ore types, each with different processing performances, along with secondary properties (cf. Pereira et al. 2023 this issue), which together can be used to estimate overall process performance.

In a mineral processing plant, the different ore types, or even specific ore properties, will then ideally be identified

with characteristic spectral measurements. For example, a combination of XRF, FTIR, and Raman spectrometry may be measured by an instrument cluster positioned over the mill feed belt that could estimate the proportions of the different ore types entering the mill. An automation system would then influence the speed and fill rate of the mill.

The ultimate outcome of all these measurements, monitoring, and associated optimisation routines, will be the highly efficient recovery of minerals of commercial interest. However, to fully achieve this potential, geometallurgy must take full advantage of developments in artificial intelligence to process the big data sets generated in modern mines. This is starting to occur at the time of writing, and we anticipate new breakthroughs in the upcoming years.

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