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Image analysis estimation of iron ore particle segregation in epoxy blocks

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ABSTRACT

The phenomenon of particle segregation during epoxy block preparation for characterisation studies by optical or SEM microscopy, and its potential detrimental effect on the results of such characterisation, is well known. However, the absence of a proper study showing the possible scale of errors caused by it, and the existing belief that in certain circumstances the effect can be neglected, result in the continuing practice where important characterisation is performed using traditional horizontally sectioned epoxy blocks.

This article presents the results of optical image analysis of the particle segregation effect during epoxy block preparation for different size fractions of iron ore fines using the CSIRO Mineral4/Recognition4 software. The differences in mineral abundances and liberation characteristics for different horizontal layers of vertical sections of epoxy blocks were analysed. Also the effects of particle layer thickness, epoxy block mixing techniques and the relative ratio of the iron ore sample to the epoxy during initial specimen preparation were investigated and discussed.

The work performed shows that the difference between layers in the epoxy block due to density segregation can be unacceptably high for iron ore samples and, similarly, to any ore samples with significant density differences between particles of different texture. The traditional block preparation technique, with its random selection of the analysed horizontal layer, can therefore result in large errors in estimation of mineral abundances, mineral liberation and textural classification characteristics. It is suggested that vertical sections of epoxy blocks are used for image analysis where significant density difference between particles is suspected.

1. Introduction

Optical and electron microscopy are widely used for the characterisation of different mineral deposits. In modern mineral production crushing and grinding of ore to smaller size particles (less than 4–5 mm) is a very common procedure during mineral beneficiation. Small aliquots of such crushed ores are used for generating representative ore samples for the characterisation of deposits and understanding potential routes for further ore beneficiation, e.g. magnetic separation, de-sliming, flotation etc.

For the purpose of microscopy characterisation, the process of sampling, which begins with the actual collection of the ore material for the preparation of representative blocks or sets of blocks with polished surface exposing cut particle sections, is the most critical part of the whole procedure. If during sampling or at the further stage of block preparation there are intrinsic, systematic errors, the final characterisation result will have an even larger error due to the other random errors involved.

During the standard procedure of block preparation for ore fines/particles characterisation, which can vary slightly between laboratories, a small aliquot of ore fines/particles is mixed in the casting mould with liquid compounds such as epoxy resin and hardener. To assist in the complete impregnation of particles and the removal of air bubbles from the epoxy the samples are then placed in a vacuum chamber, and two to three cycles of vacuuming and compression are applied. The samples are then transferred to a pressure vessel or curing oven. The label identifying the sample can then be put onto the bottom of the block, and then extra epoxy is added in order to achieve a certain thickness of the final block that is suitable for grinding and polishing. After the additional epoxy has hardened, the epoxy block is separated from the mould. The block is then turned over, so that the bottom surface, where ore particles have settled in a layer, becomes the top surface of the block, as this is the orientation required for ore characterisation. (The terms “top” and “bottom” of the epoxy block throughout the paper will be referring to this new orientation, the reader should remember that the actual settling occurs in the opposite direction). Finally, the top of the block is slightly ground and polished to achieve an optically flat, high quality surface without scratches or mineral pull-outs.

During mixing of the particles with liquid epoxy resin (where they are subject to shear forces and gravity) and further during settling down

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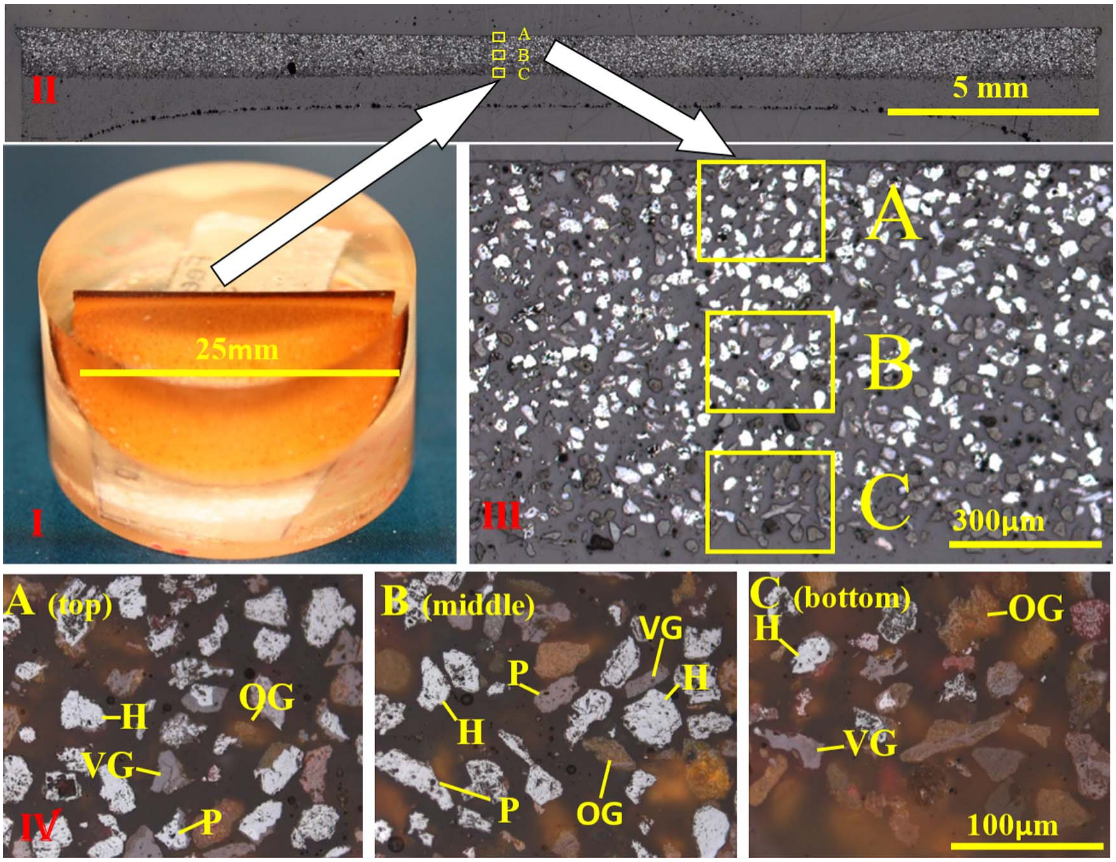

Fig. 1. Feed −38 + 25 µm size fraction: (I) Epoxy block with a cut half of the original "thin" block embedded; (II) the "thin" particle layer section (x25); (III) magnified view of the "thin" particle layer indicating the locations of images; (A, B, C) images from the top, middle and bottom of the "thin" block correspondingly (x200). Notations: "H" – hematite, "VG" – vitreous goethite, "OG" – ochreous goethite, "G" – gangue, "P" – porosity.

in the epoxy, the particles segregate by density, size and possibly shape. The effect of particle segregation within epoxy blocks has been known for a long time. It is generally understood that the concentration of heavier, denser particles near the top of the block is higher than near the bottom or in the middle of the particle layer, and, vice versa, the concentration of lighter, less dense particles is higher in the bottom of the particle layer (see Figs. 1 and 2).

Petruk (2000) in his description of mineral characterisation of iron ore and concentrator products, noticed that a suitable correlation was observed between assayed and calculated Fe contents from image analysis data for sub-fractions of concentrates and tails, but a poor correlation was observed when the sub-fractions contained significant amounts of both Fe oxides and gangue. He suggested that the results for the samples were significantly worse due to the large difference in density between particles consisting of hematite and magnetite (both with specific gravities (SG) around 5.2 g/cm^3) and gangue, generally consisting of quartz (SG = 2.65 g/cm^3). In reality, for some iron ores the difference can be even higher. For example, the SG of particles consisting of porous kaolinite, a common gangue mineral in iron ores, can be as low as 1.5 g/cm^3.

Researchers working with automated scanning Electron Microscopy (SEM) imaging techniques such as QEMSCAN (Gottlieb et al., 2000, Donskoi et al., 2014) and MLA (Gu and Guerney, 2000) noted the effects induced by particle segregation (Speirs et al., 2008; Coetzee et al., 2011). In order to provide a supporting matrix, to minimize settling and segregation of particles by density, to keep the individual particles from touching each other, and to maintain random orientation of the particles as well as to ensure an even distribution of all grains, graphite was added to the samples when preparing the samples (Speirs et al., 2008; Gottlieb et al., 2000). However, graphite itself tends to float up and segregate from the sinking sample in the epoxy resin (Kwitko-Ribeiro, 2012).

Even though the detrimental effects of particle segregation on ore fines characterisation are well known and some techniques to reduce particle segregation effects have been proposed (e.g. utilisation of vertical sections – Kwitko-Ribeiro, 2012), many laboratories still use traditionally prepared blocks for ore fines characterisation. This causes difficulties when reconciling results from optical image analysis with those from X-ray fluorescence (XRF) or X-ray powder diffraction (XRD) measurements. Untested assumptions such as "segregation is not important if the particles are smaller than 50 µm or the particle layer is thin" are quite common. Possible reasons for such assumptions include the lack of actual studies of these effects that estimate potential errors in mineral abundance and liberation measurements when traditional block preparation is utilised.

This paper attempts to fill that gap and investigate the particle segregation effect by analysing the segregation of different size fractions of iron ore fines in vertical sections of polished blocks with different particle layer thicknesses. The effects of the mixing procedure and the ratio between ore sample and epoxy are also discussed.

2. Vertical segregation

2.1. Sample preparation

To study the segregation effect, vertical sections of epoxy blocks for three size fractions of a hematitic-goethitic ore were prepared. During sample preparation, the + 25 µm and − 25 µm fractions were separated via wet sieve screening, and the + 25 µm fraction was dry screened via sieve sizing. The –25 µm size fraction obtained after dry screening of the