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## Distribution of Sb minerals in the Cu and Zn flotation of Rockliden massive sulphide ore in north-central Sweden

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#### **ABSTRACT**

The Rockliden massive sulphide Zn–Cu deposit contains minor amounts of Sb minerals. The Sb mineralogy is complex in terms of composition, micro textures and mineral associations. The main Sb minerals comprise tetrahedrite, bournonite, gudmundite and Sb–Pb sulphides such as meneghinite. The presence of these minerals is especially critical to the quality of the Cu–Pb concentrate. To study how they are distributed in a simplified flotation circuit and what controls their process behaviour Sb-rich drill core samples were selected from the Rockliden deposit and a standard laboratory flotation test was run on the composite samples. Scanning electron microscope-based automated mineralogy was used to measure the Sb mineralogy of the test products, and the particle tracking technique was applied to mass balance the different liberation classes to finally trace the distribution of liberated and locked Sb minerals. The mineralogical factors controlling the distribution of Sb minerals are mineral grain size, the degree of liberation, and associated minerals. Similarities in the distribution of specific particle types from the tested composites point towards systematics in the behaviour of particles and predictability of their distribution which is suggested to be used in a geometallurgical model of the deposit.

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#### 1. Introduction

Generally, antimony (Sb) is known to occur as a trace element in massive sulphide ores. It is regarded as penalty element because it lowers the quality of Cu concentrates produced from ore and sent to the smelters [\(Minz, 2013](#page--1-0) and references therein). Typically, penalty charges are imposed for Sb contents of 0.1– 0.3 wt% in copper (Cu) concentrates [\(Larouche, 2001](#page--1-0)). However, Sb has usage, for example as alloy in lead (Pb) batteries, if it can be separated from massive sulphide ores ([Anderson, 2012; Awe,](#page--1-0) [2013\)](#page--1-0). In addition to producing valuable products from massive sulphide ores, the Sb content of the tailings ought to be considered since the Sb content of the tailings is a potential environmental hazard, particularly in the discharge to rivers, comparable to arsenic [\(Wilson et al., 2010](#page--1-0)).

Rockliden is a polymetallic copper–zinc (Cu–Zn) massive sulphide deposit located in north-central Sweden ([Minz et al.,](#page--1-0) [2014](#page--1-0)). The inferred mineral resource is 9.2 Mt with 4.0 wt% Zn, 1.8 wt% Cu, 0.4 wt% Pb and 48 g/t Ag ([New Boliden, 2014\)](#page--1-0). The Rockliden ore contains not only bonus elements such as silver (Ag) but also deleterious trace elements such as arsenic (As) and antimony (Sb). At Rockliden, drill core samples with Sb grades higher than 0.2 wt% represent about 25% of the length of all analysed massive sulphide drill core intersections down to a depth of about 900 m below surface. The abundance of Sb-rich massive sulphide drill core intersections is higher in the upper parts of the deposit (i.e. above 400 m below surface). Particularly in the Zndominated massive sulphides from the upper part of the deposit, the Sb grade is higher than 0.2 wt% in more than half of the analysed intervals.

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Flotation tests on Rockliden composite samples have shown that the threshold of 0.2 wt% Sb is exceeded in the Cu–Pb concentrate even if the Sb grade of the blended feed material is below this value ([Bolin, 2010; Minz, 2013](#page--1-0)). This is related not only to the flotation behaviour of common Sb minerals but also to the mineral association and the degree of liberation, which are suspected to

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influence the distribution of Sb minerals between different flotation products ([Lager and Forssberg, 1990; Minz et al., 2013\)](#page--1-0). Mineralogical studies have shown that the mineralogy of Sb is complex in the Rockliden massive sulphide ore [\(Minz et al.,](#page--1-0) [2013\)](#page--1-0). The main Sb minerals are listed in Table 1, together with an expected ideal distribution of liberated particles during Cu–Pb and Zn flotation.

In geometallurgical projects it is common to run ore variability tests in the form of laboratory flotation tests in order to study the variation in the metallurgical response [\(Baumgartner et al., 2011;](#page--1-0) [Lamberg, 2011](#page--1-0)). Scanning electron microscope (SEM)-based automated mineralogy focusing on minor and trace minerals in ore products is frequently done for processed material from noble metal deposits (e.g. [Chetty et al., 2009; Goodall, 2008; Zhou](#page--1-0) [et al., 2009](#page--1-0)). In contrast to the complex Sb mineralogy of the Rockliden case, the gold (Au) mineralogy of ore deposits is usually simple (e.g. [Zhou et al., 2009](#page--1-0)). Complex trace mineralogy is reported, for example, for uranium (U), silver (Ag), rare earth element (REE) and platinum group element (PGE) ores (e.g. [Bowell](#page--1-0) [et al., 2011; Chetty et al., 2009; Grammatikopoulos et al., 2013;](#page--1-0) [Kelvin et al., 2011; Rohde et al., 2014](#page--1-0)). In such cases a large number of mineral grains, and thus a large number of samples, are mea-sured by automated mineralogy tools ([Chetty et al., 2009\)](#page--1-0). Instead of running the flotation test for a large number of samples, a different approach was chosen for the work presented here. This study was constrained to a limited number of samples. To allow for sound statistical interpretation of the distribution of Sb minerals into different products, naturally Sb-enriched samples were collected, in an approach similar to that of [Byrne et al. \(1995\).](#page--1-0) Two composite samples were selected and compared in this study. The two composites represent Zn-dominated massive sulphide ore with a Sb grade above 0.2 wt% from different parts of the Rockliden deposit. Given the complexity of the Sb mineralogy, it was the primary aim of this study to quantify the Sb mineralogy in the ore and in the products of the flotation test using SEM-based automated mineralogical tools. Finally, the target is to define the factors controlling the distribution of Sb minerals between different products like Cu–Pb, Zn concentrates and tailings.

#### 2. Methods

#### 2.1. Samples

Two sets of drill core samples were collected, representing Znrich massive sulphide ore types from the upper (S-7576) and lower  $(S-7577)$  part of the deposit [\(Fig. 1](#page--1-0)a and b). Each set contains four to five individual drill core samples of about 1 m length. For each drill core sample about 1 kg quartered drill core was collected. The drill core samples were crushed and about 100 g sample of each sample were separated for chemical analysis [\(Fig. 1c](#page--1-0)). Further, the Sb mineralogy was studied in polished epoxy grain mounts prepared from each of the crushed drill core samples. Qualitative studies were conducted using a Merlin – Zeiss Gemini FESEM (Field Emission Scanning Electron Microscope), Luleå

#### Table 1

Common Sb minerals at the Rockliden ore deposit.



**b** Liberation distribution according to [Lager and Forssberg \(1990\).](#page--1-0)

University of Technology, Sweden. Based on similarities in both chemical and SEM-based analyses, three individual drill core samples were selected from each set of drill core samples and combined to form composite samples ( $Fig. 1b$  and c). The composite samples were ground to a  $P_{80}$  of about 50  $\mu$ m in a laboratory rod mill before the flotation test [\(Fig. 1c](#page--1-0)).

#### 2.2. Laboratory flotation test

A simplified laboratory flotation test, based on the test commonly used at the Boliden Mining Company [\(Fig. 1](#page--1-0)), was carried out. The flotation test was conducted using a Magotteaux bottom driven flotation machine with a cell size of 2.7 l. The percentage of solids was about 47 wt% for both composites. The flotation reagents used are indicated in [Fig. 1](#page--1-0)c. The total flotation time for each composite was about 20 min.

Five flotation products were collected from each test: Cu–Pb cleaner concentrate (Cu CC), Cu–Pb cleaner tailing (Cu CT), Zn cleaner concentrate (Zn CC), Zn cleaner tailing (Zn CC) and final tailing ([Fig. 1](#page--1-0)c). Their chemical composition was analysed by X-ray fluorescence spectrometry (XRF) at Boliden laboratories. The flotation products of the composite samples were sieved into three size fractions  $(-20 \mu m, +20 \text{ to } -45 \mu m, +45 \mu m)$  and the chemical composition of each fraction was constrained by inductively coupled plasma atomic emission spectroscopy (ICP-AES) for 21 major and trace elements at ALS Minerals Division, Piteå, Sweden. From each size fraction about 10 g sample was separated for preparation of polished epoxy grain mounts by the Erzlabor, Helmholtz Institute Freiberg for Resource Technology, Germany. Polished epoxy grain mounts of the sieved flotation products were prepared by mixing of 3 g aliquots with graphite and epoxy resin; and finally, the epoxy grain mounts were ground and polished.

#### 2.3. Mineral liberation analysis (MLA) for characterisation of flotation products

All flotation products were studied using MLA measurements. Prior to the SEM studies the samples were carbon-coated with a Leica EM MED020 carbon coater to avoid surface charging due to electron beam interactions. Analyses were carried out with a FEI Quanta 650F equipped with two Bruker Quantax X-Flash 5030 energy-dispersive X-ray spectrometers. MLA Suite 3.1.4 software was used for automated data acquisition and processing at the Geometallurgy Laboratory, Technische Universität Bergakademie Freiberg, Germany. Consistent operating conditions were applied ([Table 2\)](#page--1-0). The grain X-ray mapping mode (GXMAP) was used for MLA measurement [\(Fandrich et al., 2007\)](#page--1-0).

It should be noted that, although distinguishable by careful energy-dispersive X-ray spectroscopy (EDX) spot analysis on a SEM and wavelength-dispersive X-ray spectroscopy (WDS) on an electron probe microanalyser (EPMA) ([Minz et al., 2013\)](#page--1-0), meneghinite and boulangerite cannot be distinguished by MLA measurements under the chosen conditions [\(Table 2\)](#page--1-0). For this purpose the back scattered electron image can be tuned and expanded to have a difference between grey levels of meneghinite and boulangerite, similarly as recommended for distinction of hematite and magnetite [\(Bachmann et al., 2014; Figueroa et al., 2012\)](#page--1-0). However, this was regarded unnecessary in this study since the flotation behaviour of meneghinite and boulangerite is expected to be similar due to their similar mineral chemistry (Table 1).

It should also be noted that challenges were encountered during MLA analyses of the particle size fraction  $-20 \mu m$  including misidentification of minerals along rims of grains and within agglomerations. Such analytical errors might have an influence on the apparent distribution of minor amounts of meneghinite and bournonite reported to products other than the Cu–Pb cleaner

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