Energy savings in comminution – Innovative routes for mineral ores embrittlement

Contribution to deliverable 4.4

Promine project (FP7)

BRGM/RP-60127-FR

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SEVENTH FRAMEWORK PROGRAMME

ProMine

Geoscience for a sustainable Earth
brgm

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Synopsis

Promine project, “Nano-particle products from new mineral resources in Europe” is a FP7 European project that aims at enhancing the efficiency of the overall European production chain putting higher quality and added value products on the market. A part of Promine project focuses on a specific research action aiming at evaluating the capabilities of two ores embrittlement technics to reduce the energy consumption during comminution. Weakening was performed through µwaves heat treatment and electro-hydraulic fragmentation (SelFrag technology).

Lubin Run-Of-Mine (ROM) and especially black shales were the first target samples for embrittlement tests because of the difficulties they induce during grinding and flotation operations. Black shales strongly reacted during µwaves treatments because of their high organic carbon content(9%) and because of the presence of sulphides that heat-up very quickly when exposed to a microwaves radiation. Incandescent red spotswere observed on shales blocks during µwaves treatment and the presence of calcinated grains (microscopic observations) was noted. According to Bond procedure, once µwaved, Lubin Black Shales (LBS) grindability appears to be worsening when microwaved but a comminution energy gain of 13 % was measured when electro-fragmented.

A second material was then looked for and pebbles from AITIK mine comminution circuit (coming from the output of the primary mill currently in operation) were sent to BRGM by Boliden. The behaviour of AITIK ore towards µwaves treatment appeared different from the one of LBS. Bond Work Index (BWI) of AITIK harder rocks decreases when pebbles are µwaved. Subsequently, a second embrittlement technic was tested: the electrohydraulic fragmentation. SelFrag Company kindly offered to perform pre-weakening tests on pebbles and LBS. Even with rather low energy consumption (5 kWh t^{-1}) electric-fragmentationallowed improving AITIK ore grindability. As well, sulphides liberation of AITIK ore revealed to be improved thanks to this pre-weakening stage.

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1. Introduction

1.1. PROMINE PROJECT SUMMARY

The objectives of ProMine IP address the Commission's concerns over the annual 11billion € trade deficit in metal and mineral deposit. Europe has to enhance the efficiency of its overall production chain putting higher quality and added value products on the market. ProMine focuses on two parts of this chain, targeting extractive and end-user industries.

Upstream, the first ever Pan-EU GIS based mineral resource and advanced modelling system for the extractive industry will be created, showing known and predicted, metallic and non-metallic mineral occurrences across the EU. Detailed 4D computer models will be produced for four metalliferous regions. Upstream work will also include demonstrating the reliability of new (bio)technologies for an ecoefficient production of strategic metals, driven by the creation of on-site added value and the identification of specific needs of potential end-users.

Downstream, a new strategy will be developed for the European extractive industry which looks not only at increasing production but also at delivering high value, tailored nano-products which will form the new raw materials for the manufacturing industry. ProMine research will focus on five nano-products, (Conductive metal (Cu, Ag, Au) fibres, rhenium and rhenium alloy powders, nano-silica, iron oxyhydroxysulphate and new nano-particle based coatings for printing paper), which will have a major impact on the economic viability of the extractive industry. They will be tested at bench scale, and a number selected for development to pilot scale where larger samples can be provided for characterisation and testing by end-user industries. It will include production, testing and evaluation of these materials, with economic evaluation, life cycle cost analysis, and environmental sustainability.

ProMine with 26 partners from 11 EU member states, has a strong industrial involvement while knowledge exploitation will transfer ProMine results to the industrial community.

1.2. PROMINE WP4 OBJECTIVES

In the framework of ProMine, workpackage 4 named "Ecoefficient metal production methods and utilization of secondary materials" has the general following objectives:

- to implement demonstration operations of innovative technologies (including on mining sites), with new integrated flowsheets and equipment design for mineral processing;

- to demonstrate that these new technologies will enable the exploitation of more diversified and complex existing European resources. Particular attention will be placed on secondary resources (mining wastes);

- to raise the revenues of current exploitations and significantly improve the ecoefficiency of mineral processing methods;

- to reduce the metal grades of wastes and to minimise the environmental impact of the final discharges.

1.3. PROMINE DELIVERABLE 4.4

This document specifically reports on the results obtained at BRGM in workpackage 4 for the first two years of the project. It covers BRGM activities for task 4 “Process Development Supports and Prospective R&D” on pretreatment and concentration aspects.

Context elements

Although considerable advances have been brought as well in mining (automation, very large-scale equipments) as in pyro or hydrometallurgy, almost no improvement have been brought in milling. Yet, comminution remains by far the largest energy consumer onmost mine sites, with reported specific energy consumption varyingfrom a few kWh/t for crushing, to 10–60 kWh/t (cf. Figure 1) for AG/SAGmilling and ball milling, and to over 100 kWh/t for ultra-fine grinding (cf. Wang *et al.*, 2011).The high energy consumption implies both high operationalcost and greenhouse footprint. Thus, comminution accounts for 30 to 70 % of the power draw of a typical mineral processing plant and for 20 to 50 % of the capital costs. However, the comminution process is only 1% efficient in terms of the energy required to generate new surfaces (cf. Whittles *et al.*, 2003).

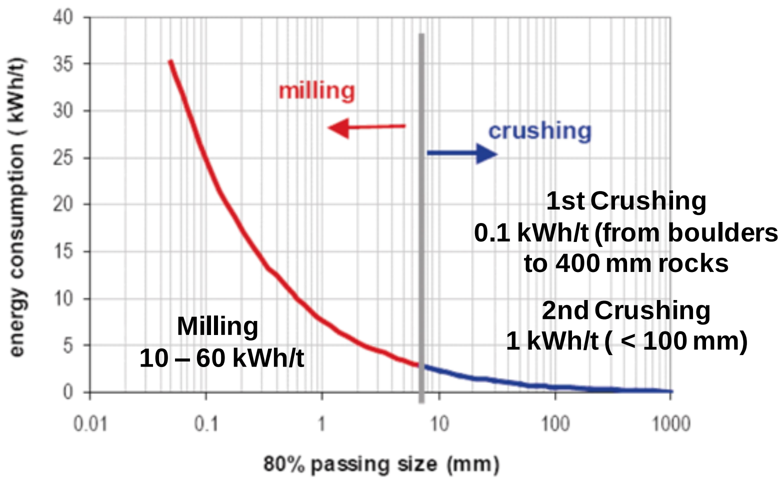

Figure 1 – Energy consumption of crushing and milling operations (from Mineral Processing Handbook, Australian Institute of Mining and Metallurgy)

Energy as a force underlies much of the world. Efficient application of energy to the right location at the right time is often the goal of good engineering. Mining is not an exception. Using energy properly to Run-Of-Mine (ROM) ore is key for comminution to the liberation size. Although mechanical systems have dominated research on methods of grinding rocks, thermal and electric fragmentation technologies hold promise for certain applications. Early experiments showed that thermal energy broke rocks inefficiently but recent developments indicate more efficient applications:

- One such development is microwave heating that weakens rocks through differential thermal expansion to improve later secondary breakage methods,
- Another development applies high voltage pulsesthrough blocks immersed in water in order to induce selective fragmentation.

The objective of the present study was to investigate both innovative comminution routes so that to assess their capabilitiesto decrease the overall energy consumption upon grinding.

2. Materials and methods

2.1. MATERIALS

2.1.1. Lubin Run-Of-Mine

The copper deposit mined by KGHM Polska Miedź S.A. substantially differs from typical, worldwide extracted porphyry deposits. The main differences are the presence of silver and a specific mineralization that causes serious issues especially during mineral processing stage.

One of the most important features of the ore processed by KGHM Polska Miedź is the presence of three types of rocks with different kind of mineralization, i.e. sandstone, shale and carbonate. The average composition of Lubin ore (see Figure 2) during 1985-1991 is as follows:

- the host rock consists mainly of sandstone (54%). The aggregates can contain sulphides located between the quartz grains but other aggregates grains do not contain any ore minerals. The size of grains along the short axis is over 100 µm, and along the long axis is about 200 µm;

- the gangue also contains dolomite grains (37.9%) that do not contain any ore minerals. Dolomite grains are smaller (short axis 100 µm, long axis 150 µm);

- Non homogeneous pieces consist mainly in black shales (8.1%). Some of them contain ore minerals. They are the smallest aggregates in the samples (isometric shape, 100 µm).

Sandstone ore has a form of light-grey, fine grained, compact sandstones, containing mainly quartz and small amounts of feldspars and other minerals, bound by carbonate or clayey binder. Metal bearing minerals are mostly not bigger than 0.200 mm, and in general, within the range between 0.050 and 0.200 mm.

Carbonate ore occurs in the form of lime dolomites and less often of dolomite limestones. Minerals of gangue are mainly dolomite, calcite, anhydrite and clay minerals. Metal bearing minerals are mostly from 0.030 to 0.200 mm.

Shale ore contains about 85 % of clay minerals and carbonates, about 7 % of organic matter and small amounts of quartz. Copper minerals are predominantly from <0.005 to 0.040 mm. It is the richest part of the ore in copper and contains many accompanying metals.

As the three ore lithological components have different and variable chemical, crushing and grinding properties and different flotability, specific challenges both at grinding and flotation stages have to be overcome. Shales cause particular problems during flotation and dewatering.

By means of microscope observations performed during the Bioshale project (Augé *et al.*, 2006), the following copper minerals were identified. In a descending line in accordance to their quantity in the sample (as % of the total amount of Cu minerals), one can find chalcocite (40%) > chalcopyrite (20%) > bornite (20%) > tennantite (10%) > covellite (10%).

The average sizes of ore minerals are nearly equal:

- chalcopyrite – 50 x 80 µm,
- tennantite – 30 x 40 µm,
- bornite – 20 x 40 µm,
- chalcocite – 20 x 30 µm.

The chemical composition of the ROM ore from the Lubin deposit is given in Table 1.

chemical element	unit	Lubin ROM
Si	%	22.9
C (mineral)	%	10.2
Ca	%	9.1
Mg	%	3.7
Al	%	2.8
C (organic)	%	1.2
S (total)	%	1.19
Cu	%	1.01
Fe	%	0.94
Pb	%	0.32

chemical element	unit	Lubin ROM
Na	%	0.2
Mn	%	0.16
P	%	0.04
Zn	mg/Kg	430
As	mg/Kg	264
Ag	mg/Kg	82
Cr	mg/Kg	74
Ni	mg/Kg	57
Mo	mg/Kg	31
Cd	mg/Kg	10

Table 1 -Chemical composition of the Lubin deposit run-of-mine ore

In the black-shale fraction of Lubin ore (see Figure 3), because of the plastic character of the shales, ore minerals are deeply associated with the gangue and have no free surface for contacts with reagents. These characteristics impede ore processing. The largest mineral grain sizes of this fraction are less than 100 µm and the average size is about 30 µm. This peculiarity reflects the fine dissemination of the ore into the host rock. The relative quantity of the different copper ore minerals in the black-shale samples (as % of their total amount) is as follows: bornite 30 %, chalcocite 30%, tennantite 30 %, chalcopyrite 5%, covellite 5%. Black-shale fraction chemical composition is given in Table 2.

High silver content in the ore (~ 80 mg/kg) and high stock exchange silver price cause that this metal represents a very important target. As a consequence, the copper concentration process is also designed to enable the highest possible silver recovery. Apart from its own phases, silver forms isomorphic replacements in the copper sulphides.

Figure 2 – Studied Lubin run-of-mine rocks

chemical element	unit	Lubin ROM	chemical element	unit	Lubin ROM
Si	%	13.8	**Mn**	%	0.16
C (organic)	%	8.94	**P**	%	0.16
Ca	%	8.36			
Cu	%	6.64	**Co**	mg/Kg	428
Al	%	6.48	**As**	mg/Kg	390
C (mineral)	%	4.28	**Ni**	mg/Kg	337
K	%	3.3	**Ag**	mg/Kg	315
Fe	%	3.14	**Cr**	mg/Kg	205
Mg	%	3.09	**Mo**	mg/Kg	164
S (total)	%	3.05	**Pb**	mg/Kg	151
Na	%	0.59	**Zn**	mg/Kg	98
Ti	%	0.24	**Cd**	mg/Kg	10

Table 2 -Chemical composition of theLubin deposit black-shale fraction

In the deposit, undesirable elements for the copper production process are also present. They worsen the production parameters and reduce final concentrate or copper price. The most important undesirable elements are lead, arsenic, sulphur, chlorine, fluorine and nickel.

Figure 3 – Studied Lubin black-shales rocks

2.1.2. PEBBLES FROM AITIK PRIMARY BALL MILLS

Aitik mine description

The Aitik mine is located 60 km north of the Arctic Circle, and 20 km east of Gällivare in Norrbotten, Sweden. This open pit mine is Boliden’s biggest mine, and produces copper ore that also contains gold and silver. The metal grades are low but are compensated for by high levels of productivity and efficient concentration processes.

Today the production level is 18 Mt of ore per year. An expansion is planned to 36 Mt of ore per year, starting in 2010. At this production level, the mine will be in operation until 2025. The depth of the open pit mine will then be 600 m.

From the 18 Mt of ore, the concentrator produces around 200 000 t copper concentrate containing 28.5 % Cu, 8 g/t Au and 250 g/t Ag. The recoveries are approximately 90 % for copper, 50 % for gold and 70 % for silver. After metallurgical treatment, 50 000 t copper, 50 t silver and 1.6 t gold are produced annually.

Boliden has a total staff complement of around 400, making it the single largest private employer in the Gällivare municipally.

Aitik ore description

The 1.9 billion year old ore body is located in a major crustal shear zone, which can be followed from Kiruna to LakeLadoga in Russia. The low-grade copper mineralisation occurs as disseminations and thin veinlets of chalcopyrite and pyrite within a westerly dipping ore zone of metamorphosed and altered volcanic and sedimentary rocks. It was discovered in the 1930's and brought into production in 1968. The detailed examination of the ore body was carried out by diamond drilling in vertical sections 80 m apart. Exploration work has delineated the main ore zone down to 800 m below the surface (Wanhainen, 2005).

The mineral of economic interest in the ore body is chalcopyrite $CuFeS_2$. The average mined grades for 2005 was 0.43 % Cu, and 0.2 g/t of Au and 3.2 g/t of Ag. The cut-off grade varies between 0.14-0.17 % Cu, depending on ore quality and transport distances.

The ore zone is 2900 m long and reaches a maximum thickness of 400 m. The hanging wall contact dips at 45º to the west and the footwall has a dip of ca. 50º. The ore body plunges fairly steeply towards the north-west. A vertical profile right across the ore body is shown in Figure 4. In the year 2025, the open pit will reach Z600.

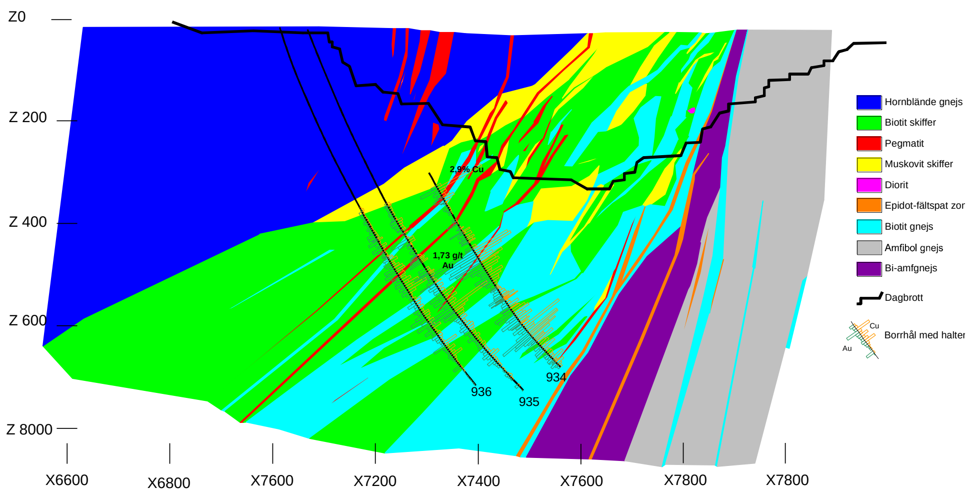

Figure 4 - Vertical profile at 4000 Y in the northern part of the Aitik ore body

Ore reserves have recently tripled to 747 Mt (2009 figures), due to successful exploration and lower production costs, which is the basis for the expansion at Aïtik.

Proven ore reserve grades are 0.25 % Cu, 0.14 ppm Au and 2 ppm Ag. This total breaks down into 518 million tonnes of provenore reserves and 229 million tonnes of probable ore reserves.Measured and indicated mineral resources amount toan additional 1,370 million tonnes.

The average Mo-grade is 30-50 ppm within the mineralization. Molybdenum is present as molybdenum sulphide, MoS_2. Molybdenum grades are higher in the northern part of the ore body and lower in the southern part.

Boliden comminution circuit and pebbles production

Boliden ore processing plant at Aïtik consists in autogenous and ball mill grindingthat is followed by conventional flotation.The system design was enhanced by the installation of Microcel flotation columns.The new AG mills supplied by Metso are the largest in the world (22.5 MW primary mills with wrap around motors measure 11.6m in diameter and 13.7m long), each with a grinding capacity of 2,200 t per hour. They are fed with run of mill sized minus 400 mm that are delivered from a stockpile with a capacity to store 200,000 t.The grinding technique used in the Boliden concentrator is two stage fully autogenous grinding where the crushed ore constitutes the only grinding media in the primary mills.Pebbles (50-80mm) are extracted from the primary mills to make up the grinding media in the secondary ‘pebble’ mills. The screen operating at the outlet of the primary mill gives also pulp (0-10 mm) and middle size particles (10- 50 mm). The pulp goes all the time to the secondary mill. The middle size goes most of the times back to the primary mill. Pebbles goes to the secondary mill whenever it is needed and some periods to the bin for the re-grinding mills and rest of the times goes back to the primary mills.

The pebbles that go out the primary mill are the hardest rocks since it has survived at least one pass through the mill. The size above 20 or 30 mm is about the same hardness. The smaller pebbles are crushed by the biggest rocks in the mill.

For the purpose of this study, the hardest rocks (pebbles 50-80 mm) (see Figure 5) were hand-picked from the conveyor belt. An exact size could obviously not have been but this was not critical for the study.

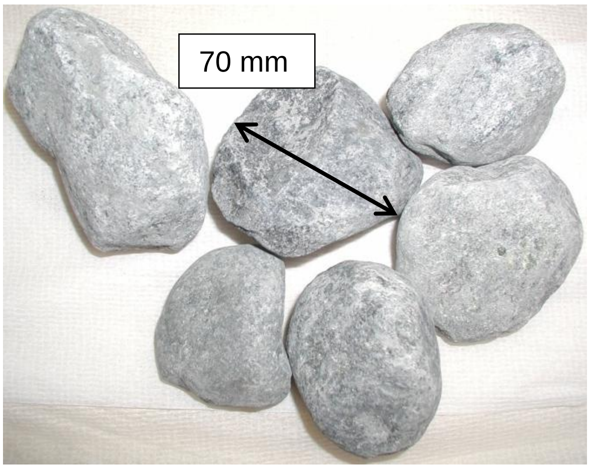

Figure 5 - Studied AITIK pebbles

2.2. EMBRITTLEMENT TECHNICS

Embrittlement technics studied in this project are based on the following observations. Rocks are very strong when compressed and weak under tensile strength (cf. Figure 6). The compressive strength of rocks is high since mineral grains are strong and they have grown together. However mineral grains boundaries are weak.

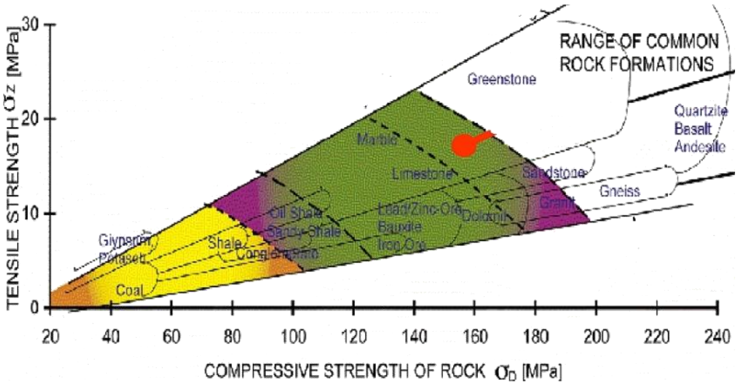

Figure 6 – Mechanical strength of materials

Conventional milling technics apply compressive forces on materials and remains for that reason highly inefficient. In this work, the selected technologies under study apply the energy straight within the material at grain boundaries. According to the energy application mode (microwaves or electric pulses), materials are destabilized out of their equilibrium state and weakened. The energy consumption to perform this weakening is *a priori* limited because of its optimal use.

2.2.1. Microwaves

Interest in the application of microwave radiation tominerals started in the mid-1980's (Kingman *et al.*, 2004) when results werereported concerning the heating of 40 mineral typesindividually with microwave energy.The test results indicated that most silicates, carbonates and sulphates reported to microwaves transparent minerals, whilst most sulphides, metal oxides,sulphosalts and arsenides reported to highly reactive minerals for which considerable heat was generated in the mineral matrices.Severalrecent studies have investigated the application of lowpower microwave radiation to mineral ores. MassiveNorwegian ilmenite ores exposed to microwave radiationfor varying times showed reductions in Bond Work Index ofup to 90% (Kingman *et al.*, 1998)

The first embrittlement technic studied in this research action was microwave heating that weakens rocks to improve later secondary breakage methods. Microwaves can penetrate rocks and, as electromagnetic energy is converted to heat by rapid vibration of molecules at a location inside the rock, can create a thermal inclusion (kind of hot spot) within the rock matrix. The hot sport then expands according to its specific thermal expansion, whereas the surrounding cooler rock does not. The resulting swelling of the hot spot inside the rock causes tensile stresses in the surrounding rock, eventually weakening or fragmenting it.The energy is generated using a variable power (0–6 kW) microwaves generator operating at 2.45 GHz manufactured by Sairem (cf. Figure 7). The samples are exposed in a multimode microwave cavity that allows treating ores samples in batches in the range of 1 to 3 kg. Settings of this treatment are power level and time period.

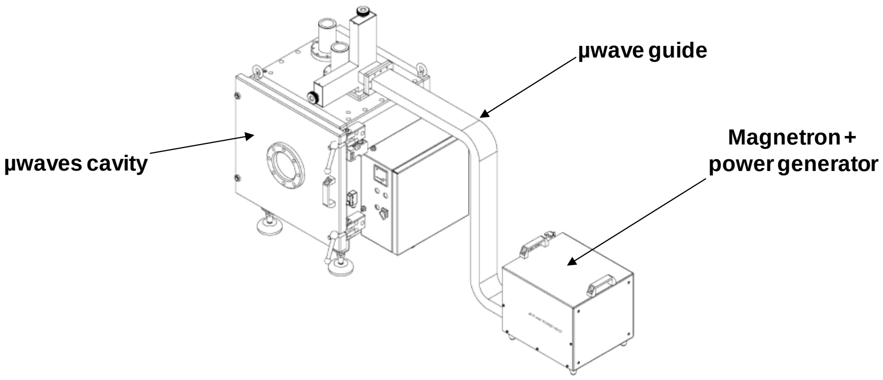

Figure 7 – Multimodes microwaves furnace used for embrittlement tests

2.2.2. Electric pulses

Another development applies high voltage pulsesthrough blocks immersed in water in order to induce selective fragmentation. This technique utilizes a voltage of up to 200 kV between an electrode and a counter electrode in a water-filled discharge vessel. As soon as a discharge channel is built up, a current of up to 20 kA flow through the material and deposits within nanoseconds energy of several hundreds of MW selectively along each grain boundaries. This energy also creates a shockwave which propagates through the material and weakens the material due to alternative compressive and tensile stresses (electrodynamic fragmentation). Additionally, the shockwave agitates the process water inducing electrohydraulic fragmentation of the material.

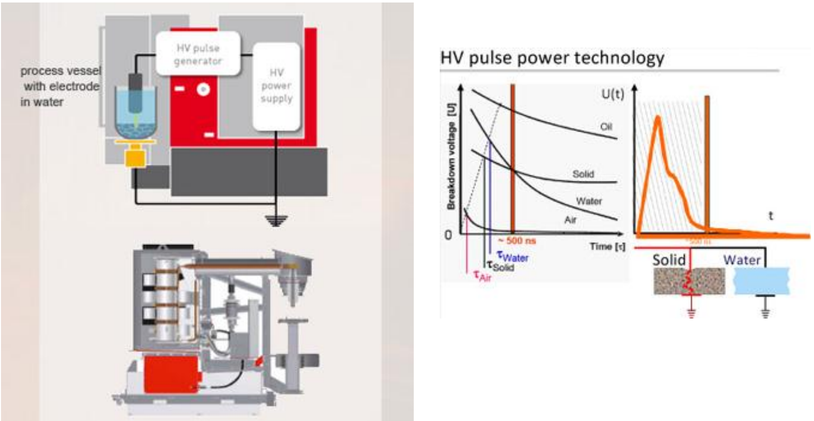

Figure 8 – High voltage electric pulses generator (SelFrag lab) used for embrittlement tests

2.3. METHODOLOGY FOR ENERGY CONSUMPTION CALCULATION

Internationally accepted mineral metallurgical standard test procedures to determine the physical properties of ores include the Bond ball mill Work Index (BWI) and the Bond rod mill work index. In this work, the BWI is used to determine the improvement or the worsening of rocks grindability.

2.3.1. Bond Work Index procedure

Principle

The Bond methodology consists in reproducing, in a normalised lab-scale equipment, grinding operations that represent those performed in an industrial closed milling circuit and to interpret data in order to get the specific energy consumed for the characterised material(JKMRC CO., 2006).To be as close as possible to the industrial reality, the dynamic separator is replaced by a screen of P_1 microns openings close of the cut-size of the industrial classifier. The principle of the test consists in reproducing the operation of a closed grinding circuit with a recycling rate of 2.5/1 (cf. Figure 9).

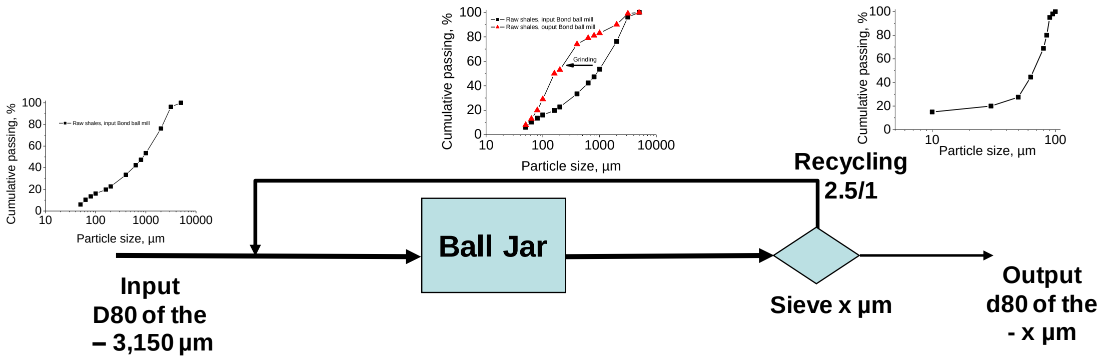

Figure 9 - Schematic diagram of the Bond methodology for the calculation of the energy consumed during grinding

Basic equations

Bond's third theory of comminution has been used successfully to size the ball mills required in three new plants, each treating completely different ores at different feed rates. Basic equations derived from the theory are:

$$W = K \times 11 \times Wi\left(\frac{1}{\sqrt{P}} - \frac{1}{\sqrt{F}}\right)$$

Where *W* = work input (kwh t^{-1}), *Wi* = ball mill work index, *P* = required product size in microns at which 80 % passes, *F* = actual feed size in microns at which 80 % passes, *K* = correction factor equal to 1 in the case of closed grinding circuit (with recycling). In the other cases, K should be calculated according to formulae given in Table 3.

Milling conditions	***K***
Dry grinding	1.3
Mill diameter *D*≈ 2.45 m	$\left(\frac{2.45}{D}\right)^{0.2}$
Fineness correction P < 60 µm	$\frac{P + 10.3}{1.145 \times P}$
Low comminution rate$\alpha = \frac{F}{P} \leq 4$	$\frac{\alpha - 1.22}{\alpha - 1.35}$
Open grinding circuit	1.2

Table 3 – Calculation formulae for BWI correction factor according to milling conditions

The work index is calculated according to:

$$Wi = \frac{44.5}{(P_1)^{0.23} \times (Gbp)^{0.82} \times \left(\frac{10}{\sqrt{P_2}} - \frac{10}{\sqrt{F_2}}\right)}$$

Where Wi = ball mill work index, P_1 = micron openings in sieve size tested, Gbp = the average grams of product undersize per revolution obtained from screen analysis of the undersize product plus the circulating load during the final three lab-scale grinding tests, P_2 = microns at which 80 % of last grinding cycle sieve undersize product passes, and F_2 = microns at which 80 % of the new ball mill feed passes.

2.3.2. Lab Ball Mill equipment

The equipment consists of a Bond Jar lab-equipment (cf. Figure 10), a complete set of test sieves (2.500, 2.000, 1.000, 0.500, 0.315, 0.200, 0.100, 0.080, 0.063, 0.040 mm) and a Bond ball mill charge. The Bond standard ball mill charge should be made of the following distribution of balls (see Table 4). In practise, an adapted distribution is used with the balls available in the lab. This practical distribution is as is given in Table 5.

The operating conditions of the Bond jar are:

- Rotation speed = 70 RPM,
- Filling rate = 12.3 %,
- Material amount in the jar = 700 cm^3,
- Recycling rate at equilibrium = 250 %.

Φ, mm	number	Weight, g
38.1	43	8 730
31.8	67	7 197
25.4	10	705
19.0	71	2 058
12.7	94	1 441
TOTAL	285	20 131

Table 4 – Standard ball mill charge distribution

Φ, mm	number	Weight, g
40	36	8 768
30	64	7 245
25	10	698
20	55	2 083
12.5	163	1 341
TOTAL	328	20 134

Table 5 – Distribution of the used ball mill charge

Figure 10 –balls distribution and jar used for Bond Work Index assessment tests

3. Experimental results

3.1. LUBIN RUN OF MINE (ROM) AND LUBIN BLACK SHALES (LBS)

The influence of embrittlement on Lubin ores grindability was performed in duple time. First, Lubin ROM was microwaved according to a set of operating conditions and BWI evolution with treatment was determined. The specific case of black shales was then considered because of the particular issues they induce during current conventional grinding (plastic behaviour), flotation and dewatering. The results corresponding to Lubin ROM and Lubin black shales taken aside are given hereinafter.

3.1.1. Microwaves embrittlement results

Embrittlement tests

The first step of the study was dedicated to the measurement of the BWI of Lubin ROM without any treatment, according to the procedure described in appendix 1. The results are given in Table 6. It can be noticed that once microwaved, the materials exhibit an unexpected behaviour that is characterised by a worsening of the grindability with regards to an increase of the BWI. A duplicate was performed so that to check the variability of the results and no real changes were pointed out.

	$BWI_{100\ \mu m}$, kwh t^{-1}		
Size of sieve openings, µm	Raw ROM	Treated ROM 6 kW – 10 s /kg = 16 kWh t^{-1}	Treated ROM 6 kW – 10 s / kg = 16 kWh t^{-1}
100	9.4	9.8	10.0

Table 6 – Influence of microwaves treatment on the BWI of Lubin ROM

Because of their particular behaviour, black shales were then taken aside so that to assess their specific behaviour with regards to microwaves treatment. The results corresponding to black shales themselves are given in Table 7. Again, we showed a worsening in black shales grindability with microwaves treatments. The higher the microwaves energy injected the higher the measured BWI. Whatever the operating conditions (high input power, low input power, high energy, low energy) the global evolution remains unchanged. One can observe that BWI's are close to 19 kWh t^{-1} whatever the energy injected in the material. This indicates that the matrix modification that leads to the worsening of grindability appears in the very first moment of the treatment.

The most marked gap obviously appears when measuring the BWI for a grinding size of 50 µm. At that size, the difference between the treated shales and the untreated ones is equal to 9 kWh t^{-1} in absolute value against 5.7 kWh t^{-1} at a sieve mesh size of 100 µm.

	$BWI_{100\ \mu m}$, kwh t^{-1}			
Size of sieve openings, µm	Raw Shales	Treated Shales 6kW – 3s / kg = 5 kWh t^{-1}	Treated Shales 3kW – 6s / kg = 5 kWh t^{-1}	Treated Shales 3kW – 60s / kg = 50 kWh t^{-1}
100	13.8	19.0	18.5	19.5
	$BWI_{50\ \mu m}$, kwh t^{-1}			
50	26.8	-	-	35.8

Table 7 - Influence of microwaves treatment on the BWI of Lubin black shales

Microscopic observation

As regards the behaviour of shales during microwaves treatment, it was clearly highlighted that they strongly react during the treatment. Incandescent red spots at shales surface appeared during every tests and even after a few seconds of treatment, fumes were given off the samples.

- **Thin sections**

Polished thin sections have been prepared, before and after µwaves treatment through impregnation with a coloured resin so that to observe the effect of the treatment on the mineralogy and on the structure of the black shales.

From a mineralogical viewpoint, the microscopic observation of sulphides, carbonates, silicates and organic matter does not reveal any modification of these phases after the µwaves treatment.

However, black shales appeared to be fractured, more or less deeply. The fractures are mainly localised at the sample boundaries. They correspond to detachment levels between beds and that develop along these beds parallel to the bedding direction, most often at carbonates rich levels.

A set of photographs (see Figure 11) illustrates these comments and show the link between the observed fractures and the different phases of the black shales.

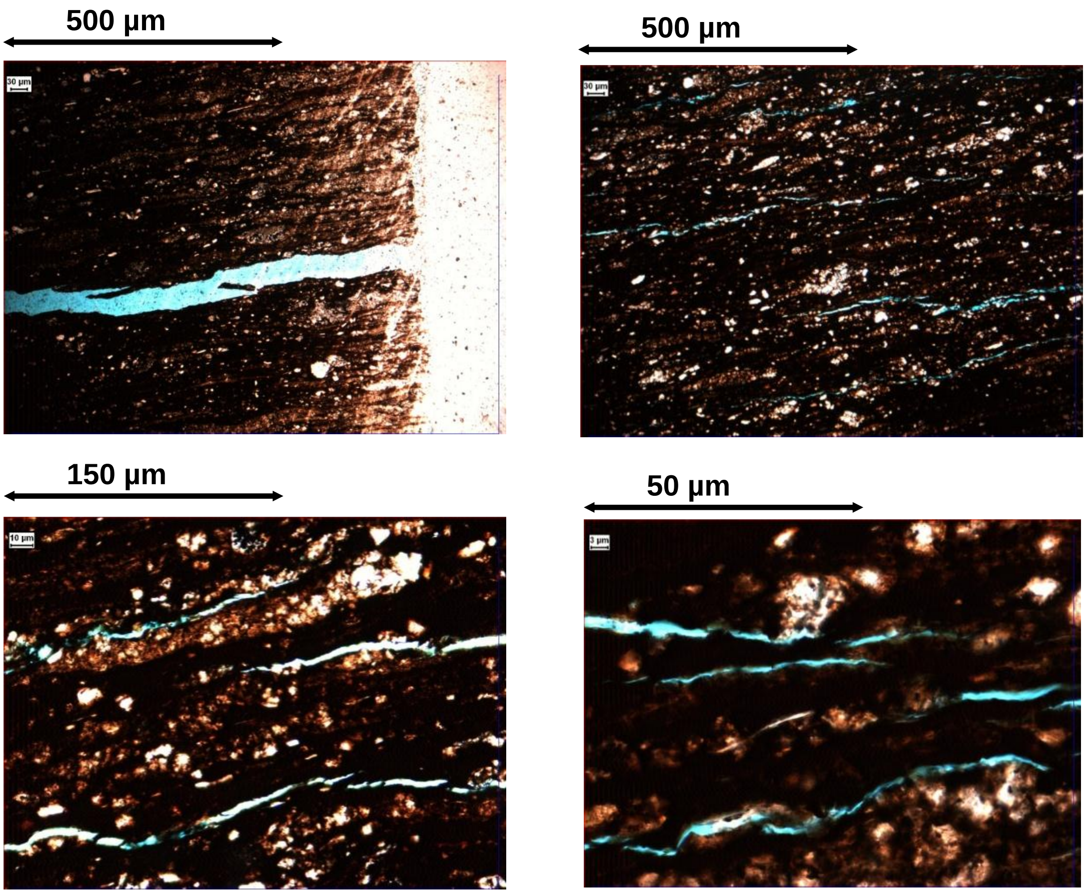

Figure 11 – Microscopic observation of the fracturation of treated shales

- **Microscopic observations of the +500 – 1,000 µm fractions**

In order to understand the effects of the µwaves treatment on the worsening of black shales grindability, comparatives observations of the +500 – 1,000 µm fractions (treated and untreated ones) have been performed. From these observations, it can be concluded to the presence of:

- particles with a modified mineralogy, slag-like looking with holes that were not observed on the untreated shales samples;
- totally liberated minerals (chalcocite, chalcopyrite) that was not observed on the untreated shales;
- flat particles with very little mechanical strength that easily crumble when humidified

It is well known that carbon as well as sulphides strongly react with the microwaves field and heat-up very fast (up to 900°C min^{-1} for $CuFeS_2$). The major part of the microwave energy is consumed to heat-up organic carbon rich phases that exhibit a plastic mechanical behavior. Further investigations could be performed specifically on the organic matter (rock eval, reflecting power) so that to evaluate the induced modification of the organic carbon rich phases with the µwaves treatment. Anyway, observed micro-cracks do not permit to improve globally the grindability of this specific material.

3.1.2. Electric pulses embrittlement results

Faced with this unexpected behaviour of shales towards microwaves heating, a second embrittlement technic was evaluated. As described in the second section of this report, this technology applies high voltage pulses, a few kV per cm, through blocks immersed in water in order to induce selective fragmentation.

Two tests were performed on two batches of 5 kg each,whereas to ensure representativeness, in-between 20 to 30 kg of materials should have been processed (but there was not enough material left).For each batch a specific energy equal to 5 kWh t^{-1} was applied. After treatment, shales were recovered, dewatered, dried and grinded in a roller mill so that the whole material passes through the 3.5 mm openings sieve. This recovered ore was then put in the Bond Jar and grinded (200 rotations). The particles size distribution of shales before milling (green bars, -3.5 mm), shales without fragmentation after 200 rotations (blue bars) and embrittled shales after 200 rotations (red bars)are given in Figure 12.

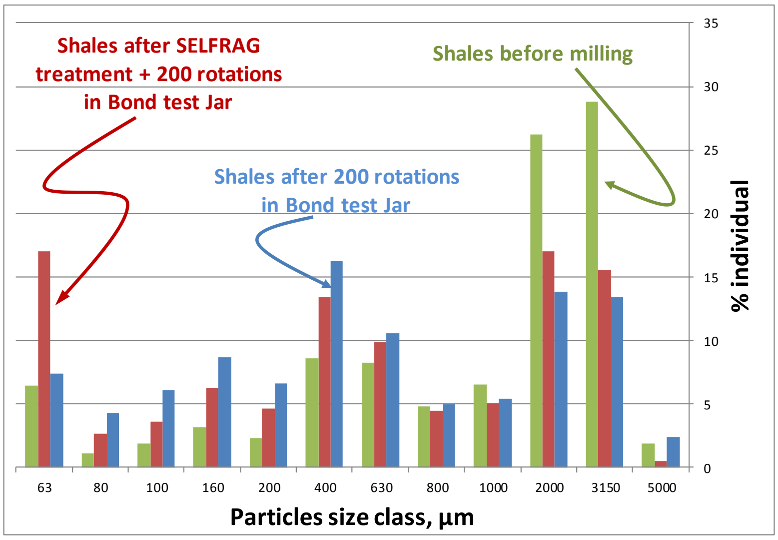

Figure 12 – Evolution of particles size class distribution with and without electric fragmentation of black shales

From these very preliminary results, it can be noted that after electric pulses treatment and a milling post-treatment (200 rotations in a Bond test jar), there is a shift towards finer particles and an increase of the production of particles below 63 µm. This corresponds to a decrease of 13 % of the BWI of shales.

3.2. AITIK PEBBLES

A second material was assessed by BRGM in the framework of PROMINE WP4 research on comminution. This material is described in chapter 2.1.2 and corresponds to AITIK mine hardest pebbles coming from the output of the primary mill of the beneficiation circuit. These pebbles are in the 50-80 mm range.

3.2.1. Microwaves embrittlement results

Following the tests performed for Lubin ROM and black shales, pebbles from the AITIK mine have been first microwaved so that to assess the efficiency of thermal embrittlement. Batches of 1 kg each were treated according to different sets of operating conditions aiming at assessing the evolution of BWIs with regards to the energy injected in the material.

First observations showed that this material strongly reacts to the microwave field and the quick rise in temperature goes with a thermal dilatation thatfragments the pebbles (see Figure 13).

Figure 13 – Pebbles fragmentation linked with fast temperature rise during microwaves treatment

Bond work index measurements were then performed for 3 different operating conditions corresponding to injected energies in the 15 to 80 kWh t^{-1} range. From the results obtained (see Table 8), it appears that the larger the amount of injected energy the lower the BWI. Besides, for the same amount of injected energy, the higher the power the lower the BWI. The maximum reduction of BWI obtained was however limited to 25% of the one of untreated ore and was achieved for an input power of 3kW and a treatment duration of 100 s (around 80 kWh/t).

Treated ore amount, kg	1	1	1
µwave power, kW	1	2	3
Treatment duration, s	100	150	100
µwaves energy consumption, kWh/t	15	80	80
Bond work Index reduction, %	< 1	14	25

Table 8 – BWI evolution with microwaves treatment of AITIK pebbles

3.2.2. Electric pulses embrittlement results

AITIK pebbles were then fragmented using the SelFrag lab equipment. For this purpose, two tests were performed on two batches of 5 kg each, as for Lubin black shales. For each batch a specific energy equal to 5.5 kWh t^{-1} was applied. After the fragmented ore pieces were recovered, dewatered, dried and then crushed in a roller mill so that the whole material passes through the 3.5 mm openings sieve. This recovered ore was then put in the Bond Jar and grinded (200 rotations). The particles size distribution of crushed pebbles before milling (green bars, -3.5 mm), Aïtik ore without fragmentation after 200 rotations (blue bars) and fragmented ore after 200 rotations (red bars) are given in Figure 14.

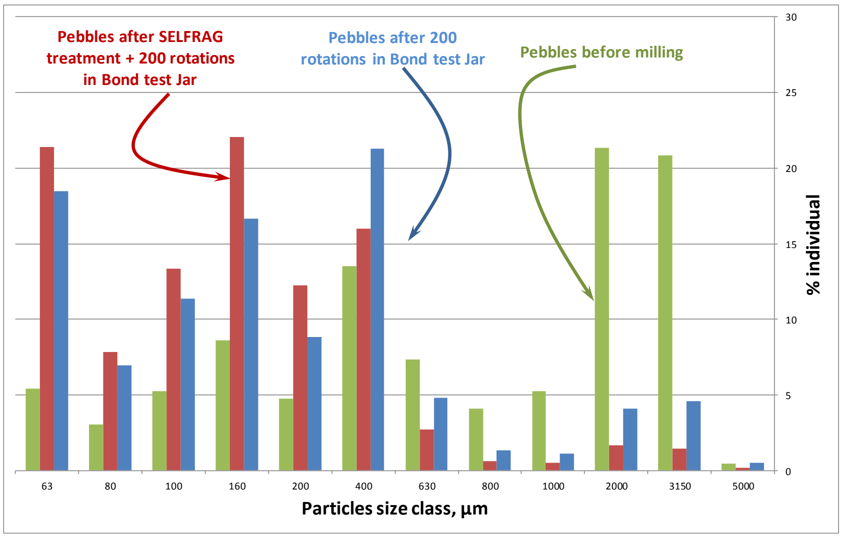

Figure 14 - – Evolution of particles size class distribution with and without electric fragmentation of AITIK pebbles

For both tests, the same results were obtained namely an enrichment of the under 200 µm gain size fractions for the weakened material compared to the un-weakened one. This evolution leads to a limited decrease of 8 % of the BWI of Aïtik ore. Moreover, unlike the conventional grinding, electrohydraulic embrittlement seems preventing from producing too much ultrafine particles. Indeed, very few particles under 10 µm were produced after electric embrittlement.

3.2.3. Microscopic observation of sulphides liberation

6 polished sections were prepared on Aitik grinded ore for 2 grain size fractions (+80-100 µm and +160-200 µm) and for the untreated material (2 sections) and weakened material (2 for µwaves embrittlement and 2 for electric pulses fragmentation). Each polished section was divided in subsectors (see Figure 15) (4 areas for each section) and a counting was performed on each particle together with a visual identification of minerals. Particles were then divided in 3 categories: the “ore” one (“no gangue” mineral part), the liberated one (totally liberated sulphides) and the “middling” one (partially liberated sulphides). The results of these microscopic observations and identifications are given hereafter.

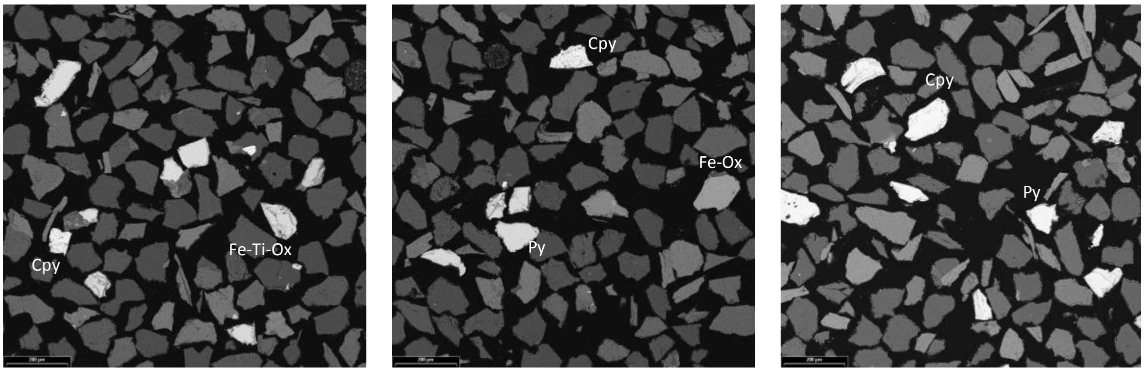

Figure 15 – Photographs of polished sections of the +160-200 µm size fraction of Aitik ore

Microwaves treatment

The liberation assessment results (counting) for sulphides in the case of a µwaves embrittlement are given in Table 9.No clear difference appeared for sulphides liberation in the 80-100 µm size. The ratio is equal to 1.3 in the case of the untreated material and is equal to 1.4 in the case of the µwaved one. The difference is a bit more marked for the 160-200 µm size (0.8 for the untreated ore to be compared to 1.2 for the treated one). On the whole, a tiny improvement of µwaves embrittlement with regards to sulphides liberation was pointed out.

	total particles				
	nb analyzed particles	% ore	% liberated	% middling	ratio liberated/middling
UNTREATED 80-100 µm	437	3.7	2.1	1.6	1.3
UNTREATED 160-200 µm	407	4.4	2.0	2.5	0.8
µwaves 80-100µm	393	3.1	1.8	1.3	1.4
µwaves 160-200µm	387	6.7	3.6	3.1	1.2

Table 9 – Liberation assesment of sulphides for µwaved Aitik ore

Electric pulses treatment

The same analyses were performed on the electro-fragmented Aitik ore. In that case however, the ratio totally liberated particles and partially liberated particles, was far better than in the case of µwaves treatment. For the +80-100 µm size, the ratio reached a value of 3.3 and for the +160-200 µm, this ratio reached a value of 5.3.

These results show that notwithstanding the energy consumption gain during grinding, the pre-weakening can improve the liberation of sulphides upon post-grinding and thus improve the whole beneficiation process.

	total particles				ratio
	nb analyzed particles	% ore	% liberated	% middlings	liberated/middling
UNTREATED ORE 80-100 µm	437	3.7	2.1	1.6	1.3
UNTREATED ORE 160-200 µm	407	4.4	2.0	2.5	0.8
SELFRAG 80-100µm	325	4.0	3.1	0.9	3.3
SELFRAG 160-200µm	349	5.4	4.6	0.9	5.3

Table 10 - Liberation assesment of sulphides for electro-fragmented Aitik ore

4. Conclusion

In the frame of Promine project, a specific research action was carried out, aiming at evaluating the capabilities of two ores embrittlement technics to reduce the energy consumption during comminution. Weakening was performed through µwaves heat treatment and electro-hydraulic fragmentation (SelFrag technology).

Lubin ROM and especially LBS were the first target samples for embrittlement tests because of the difficulties they induce during grinding and flotation operations. Black shales strongly reacted during µwaves treatment because of their high organic carbon content(9%) and because of the presence of sulphides that heat-up very quickly when exposed to a microwaves radiation. Incandescent red spotswere observed on shales blocks during µwaves treatment and the presence of calcinated grains (microscopic observations) was noted. According to Bond procedure, once µwaved, LBS grindability appears to be worsening when microwaved but a comminution energy gain of 13 % was measured when electro-fragmented.

Faced with these first results a second material wasselected for study and pebbles from AITIK mine were sent to BRGM by Boliden. The behaviour of AITIK ore towards µwaves treatment appeared different from the one of LBS. BWI of AITIK harder rocks decreases when pebbles are µwaved. Subsequently, a second embrittlement technic was tested: the electrohydraulic fragmentation. SelFrag Company kindly offered to perform pre-weakening tests on pebbles and LBS. Even with rather low energy consumption (5 kWh t^{-1}) electric-fragmentationallowed improving Aitik ore grindability.As well, sulphides liberation of AITIK ore revealed to be improved thanks to this pre-weakening stage.

In conclusion, microwaves embrittlement appeared more or less efficient, depending on the material to be treated. However, from the performed tests it has been observed that even in the case of a beneficial effect, the energetic gains in BWI never cover the consumed energy during micro-waves embrittlement. Electro-hydraulic fragmentation looks promising as it brought gains both in energy consumption reduction and in sulphides liberation allowing further optimisation of the whole beneficiation process.

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