International Journal of Mineral Processing 136 (2015) 56–65

Contents lists available at ScienceDirect

International Journal of Mineral Processing

journal homepage: www.elsevier.com/locate/ijminpro

Impact of mechanochemical effect on chalcopyrite leaching

Samayamutthirian Palaniandy *

Julius Kruttschnitt Mineral Research Center, University of Queensland, 40 Isles Road, Indooroopilly, QLD 4068, Australia

ARTICLE INFO

Article history:
Received 19 January 2014
Received in revised form 11 September 2014
Accepted 1 October 2014
Available online 7 October 2014

Keywords:
Mechanochemical effect
Stirred milling
Leaching

ABSTRACT

Stirred milling is an enabling technology to process low grade finely disseminated ore bodies. Besides size reduction, stirred mill has the ability to deliver high energy to particles which facilitate crystal structure distortion. High energy stirred milling experiments (up to 300 kWh/t) were carried out on chalcopyrite concentrate. The media size, mill speed and slurry % solids were kept constant in this experiment. The only operational variable is milling time that results different levels of specific energy. The feed and ground particles were characterized for particle size distribution and mineral phase analysis (XRD). Mechanochemical effect was quantified by calculating the degree of crystallinity, crystallite size and lattice strain. The feed and ground sample undergo leaching test in five different lixiviants. The lixiviants chosen were sulphuric acid, hydrochloric acid, nitric acid, ferric sulfate and ferric chloride. The minimum particle size and degree of crystallinity obtained were 3.7 μm and 42% respectively at 113 kWh/t. Ferric chloride exhibits the highest Cu dissolution (up to 75%) when it was ground at 113 kWh/t. Based on the results, this paper has suggested two circuit layouts that may assist mechanochemical effect in order to enhance leaching. Mechanochemical effect exhibited during high energy fine grinding enhances downstream processes i.e. leaching. This is not taken into consideration when evaluating the efficiency of the milling circuit. In the future the milling circuit efficiency should take into consideration the reduction in particle size and mechanochemical effect during milling circuit efficiency evaluation.

1. Introduction

The mining industry is seeking for improved processes to treat low grade finely disseminated ore bodies. This type of ores demands fine grinding to liberate the valuable minerals. Lately, there are many concentrators that engaged bulk fine grinding in the secondary and tertiary grind stages (Palaniandy et al., 2013, 2014). Furthermore, there are concentrators that adopted ultrafine grinding to further liberate the rougher concentrate. For example, the Mount Isa Mines (MIM), Australia requires fine grinding down to 7 μm to feed the zinc cleaner circuit. Conventionally, ball mill was used for this purpose but it has limitation to grind finer (below 10 μm) and the energy consumption is tremendous for this duty. The limitations of ball mills in the fine grinding regime affected the ability processing of fine grained ore bodies (Burford and Clark, 2007). Stirred mill has become a lucrative option in fine grinding circuit as it has ability to grind finer at lower energy consumption compared to ball mill especially below 100 μm. For example the McArthur River Mine (MRM) deposit was discovered in 1955, despite numerous effort by the mining companies to process the ore body none of them were successful until the introduction of stirred milling technology in 1989 (Burford and Clark, 2007). This technology was previously used for fine grinding of industrial minerals, food and paint (Jankovic, 2003). Stirred mills can be classified into two categories i.e. gravity induced (VERTIMILL® Grinding Mill and Tower Mill) and fluidized mills (IsaMill™, VXP mill, SMD™ and HIG mill).

Besides size reduction, the high power intensity in the stirred mills (see Table 1) causes intense mechanical stressing on particles that leads to rupture of the crystal structure (Urakaev and Boldyrev, 2000). This phenomenon is called mechanochemical effect. Balaz (2008) mentioned that the energy delivered by the media to the particles causes significant damage to both surface and bulk structure of minerals which leads to amorphism (decreases of mineral phase crystallinity). The degree of crystallinity is quantified using X-ray diffraction by measuring the peak intensity and base breadth. The assumption made in this calculation is the feed material is fully crystalline. The ground particles normally exhibit lower peak intensity and broader base which indicates that the measured phase has transform to partially amorphous phase. The discussion on impact of high intensity grinding that resulting mechanochemical effect has been reported in many publications (Achimovicov et al., 2006; Arbain et al., 2011; Balaz, 2000, 2008; Balaz and Dutkova, 2009; Jamil and Palaniandy, 2011; Palaniandy et al., 2007, 2008b; Welham, 2001a,c). This phenomenon is more pronounced when the particles were ground to below 10 μm. There are industrial stirred mills that produce product size below 10 μm. For example, MIM and MRM are regrinding rougher concentrate down to 7 μm to feed the cleaner flotation circuit (Burford and Clark, 2007). These facts have strengthened the needs to further study the impact of mechanochemical effect towards downstream processes. Besides its ability to grind finer, the stirred mills the capability of steep particle size

---

* Tel.: +61 7 3365 5854; fax: +61 7 3365 5999.
E-mail address: s.palaniandy@uq.edu.au.

http://dx.doi.org/10.1016/j.minpro.2014.10.005

Table 1
Power intensity of grinding mills.

Mill type	Power intensity (kW/m^3)
Regrind ball mill	19
Stirred Media Detritor (SMD)	150
IsaMill	400
VXP mill	240–765
HIG mill	100–300

distribution is essential for leaching (Burford and Clark, 2007; Palaniandy et al., 2014). This is an essential characteristic for good leach feed preparation. Minimal generation of ultrafine particles (below 1 μm) will avoid the increase in pulp viscosity which render leaching kinetics.

There are evidences of mechanochemical effect that enhance the hydro and pyro-metallurgical processes (Palaniandy et al., 2008a). Among the hydrometallurgical processes that have combined fine grinding and mechanochemical effect are Lurgi-Mitterberg (vibration mill), Activox (stirred mill), Melt and Albion (IsaMill) processes (Balaz and Dutkova, 2009). Although these advantages have been observed in downstream processes, to date there are no relationship that has been developed to relate particle size, mechanochemical effect and its impact towards downstream processes such as metal dissolution and leaching kinetics.

The gaps in this area include:

- Identifying the key operational variables that influences mechanochemical effect,
- Ability to apportion the amount of energy used for particle breakage and mechanochemical effect,
- Practical methodologies for easily and quickly measuring mechanochemical effect,
- A standard methodology to measure milling performance in terms of mechanochemical effect, and
- Potential circuit layout that has mechanical activators.

If the above mentioned gaps can be addressed, the mill operator will have more flexibility in controlling the stirred mill to balance the particle size and mechanochemical effect for recovery enhancement.

Although fine grinding and mechanochemical effect have exhibited many advantages towards metal recovery, there are other operational issues that need to be tackled such as aggregation of fine particles. Juhacz and Opoczky (1990) define aggregation as particles weakly associated by a reversible Van der Waals type adhesion. Aggregation of ultrafine particles to form large particles is detrimental towards leaching. It is not the intension of this paper to address this issue but there are alternative steps that can be taken to minimize this effect.

This paper will discuss the impact of fine grinding in a pin type vertical stirred mill and its impact on mechanochemical effect and Cu dissolution.

2. Materials and method

A series of batch mode grinding experiments were carried out in a pin type stirred mills over a range of specific energy. The energy range was chosen based on the current industrial mill operation and extended into severe mechanochemical effect regime. The media size, filling, slurry density and mill tip speed were kept constant at 10 mm, 40%, 40% and 15 m/s respectively. The energy was varied by grinding at 15, 30, 45, 60 and 90 min intervals. Chalcopyrite concentrate obtained from a mineral processing plant in Chile was used in this experiment. Fig. 1 shows the X-ray diffraction of concentrate. The main mineral phases contained in this sample are chalcopyrite, pyrite and bornite. Table 2 shows the main elements present in the sample.

The feed and ground samples were characterized for particle size distribution, mechanochemical effect and morphology. The particle size analysis was carried out in a laser diffraction sizer. The chemical composition of the sample was determined by performing X-ray fluorescence (XRF) analysis using Rigaku X-ray Spectrometer RIX 3000. The phase analysis and mechanochemical effect quantification (i.e. degree of crystallinity (DOC), crystallite size and lattice strain) were determined based on the changes observed in the X-ray diffractogram. XRD patterns were evaluated using a BRUKER powder diffractometer with Bragg–Brentano geometry equipped with a curved graphite monochromator in the diffracted beam arm and using Cu Ka radiation (k = 0.15406). The XRD patterns of the samples were recorded in the range 2θ = 10–70° using a step size of 0.043° and a counting time of 107.4 s/step. Peak positions (2θ) and the full-width at half maximum (FWHM) area under peak (A) were obtained from the XRD spectra to characterize the microstructure such as crystallite size, D_m using Scherrer equation, lattice strain, and degree of crystallinity (DOC), as shown in Eqs. (1)–(3) respectively (Arbain et al., 2011; Pourghahramani and Forssberg, 2006a). The APD Version 4.1 g software was used to obtain these parameters, of which the (112), (204) and (312) planes were selected for the profile analysis (Palaniandy and Jamil, 2009).

$$D_v = \frac{K\lambda}{\mathrm{FWHM}} \cos\theta \tag{1}$$

where D_v is the volume weighted mean of the crystallite size, K is a constant, h is the Bragg angle of (h k l) reflection, and k is the wavelength of X-rays used (Palaniandy and Azizli, 2009; Pourghahramani and Forssberg, 2006b).

$$\varepsilon = \frac{\beta}{4\tan\theta} \tag{2}$$

where ε is the lattice strain, and β is the integral breadth profile (Palaniandy et al., 2008a; Pourghahramani and Forssberg, 2007).

$$\mathrm{DOC} = \frac{A_t}{A_0} \tag{3}$$

where DOC is the degree of crystallinity, while A_0 and A_t are the areas under the peak for feed and ground sample, respectively (Palaniandy and Jamil, 2009).

Morphology analysis was carried out by a scanning electron microscope (Model ZEISS SUPRA 35VP). The leaching experiment was carried out in bottle rolled for 5 days. Cu and Fe content in the pregnant solution was determined using atomic absorption spectroscopy (AAS).

3. Results and discussion

3.1. Fine grinding

Fine grinding experiments was carried out in a range of specific energy by varying the milling time as shown in Table 3. The current industrial scale stirred mill is being operated up to 100 kWh/t. This experimental plan covers that range and extended up to 299 kWh/t in order to evaluate the mechanochemical effect and particle size.

Fig. 2 shows the particle size distribution of the feed and ground product as a function of specific energy. Severe particle size reduction was observed at the beginning stage of grinding. The grinding rate reduces after 80 kWh/t. The increase in particle size distribution after 80 kWh/t indicates that the ground particles are experiencing aggregations and it re-breaks at 299 kWh/t. Welham (2001a) mentioned that the particles are subjected to two processes i.e. breakage and re-welding. At the initial stage of grinding, the breakage rate is higher and the re-welding is minor. However, as the milling progresses the fraction of fines generation is small until the re-welding is greater