STIRRED MILLING OF NICKEL LATERITES FOR SELECTIVE COMMINUTION

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ABSTRACT

A process involving pre-concentration, drum agglomeration and column leach was evaluated for nickel laterite ores from Western Australia. The result showed that pre-concentration by selective comminution followed by size classification has a potential to upgrade nickel laterites and to improve the leaching performance of agglomerated particles. Selective comminution of siliceous goethitic, saprolitic, and goethitic nickel laterites was investigated to upgrade nickel into the fine fraction using stirred milling. The breakage rates of minerals, grinding time, and the weight ratio of soft and hard minerals in the feed play an important role in selective grinding and nickel upgrade. When the grinding time and breakage rates were optimized, the weight ratio of the soft and hard minerals in the feed determine the potential for selective grinding and the nickel grade in the soft mineral determines the limit of nickel upgrade.

KEYWORDS

Nickel laterite, pre-concentration, mineral processing, selective comminution, size classification, agglomeration, heap leaching

INTRODUCTION AND THEORY

Processes involving pre-concentration, drum agglomeration, and column leaching were studied extensively on three nickel laterite materials referred to as siliceous goethitic (SG), goethitic (G), and saprolitic (SAP) that were selected from Western Australia. For pre-concentration, a selective comminution study was carried out on the nickel laterite ores to obtain a soft, nickel rich product through stirred milling. The nickel upgrade was achieved under the optimal grinding conditions and up to 77% nickel upgrade was achieved with 14% nickel recovery on a SG sample. Nickel was found to be distributed more in the soft mineral fraction that allowed the upgrading. In the SG sample, antigorite is the main soft mineral while quartz is the hardest one. It is interesting to know how the minerals with vary hardness played roles in the selective size reduction process (Nosrati, Quast, Xu, Skinner, Robinson, & Addai-Mensah, 2014; Quaicoe, Nosrati, Skinner, & Addai-Mensah, 2014; Quast, Xu, Skinner, Nosrati, Hilder, Robinson, & Addai-Mensah, 2013; Tong, Klein, Zanin, Skinner, Quast, Addai-Mensah, & Robinson, 2013, 2015; Xu, Liu, Quast, Addai-Mensah, & Robinson, 2013).

Selective Comminution

Selective size reduction processes have been investigated extensively in both coal and mineral systems. A successful example of selective comminution is the beneficiation of alumina in low-grade bauxite. Diaspore is the dominant alumina containing mineral, while the main silicate minerals are kaolinite, illite, and pyrophyllite. Kaolinite can be removed by selective grinding followed by size classification to get an enriched coarse diaspore concentrate. The mass ratio of Al_2O_3 to SiO_2 (A/S) was used to evaluate the effect of selective grinding. The mass ratio, A/S, increased from 5.9 to 9.0 with 43% Al_2O_3 recovery. Selective comminution of bauxite ore followed by flotation was developed and applied industrially (Ou, Feng, Chen, Lu, & Zhang, 2007; Yin, Han, Wei, Yuan, & Yu, 2004; Zhao, Miller, & Wang, 2010).

Many studies were carried out on the selective grinding of quartz containing ores to obtain a soft, fine product. Yusupov, Kirillova, & Denisov (2003) investigated the quartz-feldspar system to concentrate feldspar in the fine size fraction. Kim, Kim, & Cho (2007) studied the recovery of high-grade kaolin by selective grinding of kaolin from anorthite and quartz followed by size classification. Selective grinding of a pyrophyllite and quartz containing ore was studied in a stirred ball mill and the product with size smaller than 45 µm showed a higher value of alumina content (Kim, Kim, Lee, & Kim, 2012). A rare earth sample was investigated to upgrade bastnaesite by selective comminution. Bastnaesite was enriched in the fine size fraction while quartz was concentrated in the coarse size fraction (Ito, Takamatsu, Asakura, Nisikawa, Hiroyoshi, & Tsunekawa, 2013).

Improvements to selective grinding can be achieved by the modification of the feed particles before grinding through the following technologies including electrical comminution, cryogenic comminution, chemical comminution, and microwave pre-treatment. The SelFrag high voltage pulse technology was used to liberate coal, minerals, and recycling concentrates (Andres, 2010; Ito, Owada, Nishimura, & Ota, 2009; Wang, Shi, & Manlapig, 2012a). Numerical simulations show that high electrical field intensity was created around the boundary of the minerals with high conductivity/permittivity, resulting in selective fragmentation. The effects of feed size, sub-sieve classification, incremental breakage and energy input level on particle pre-weakening and mineral liberation were tested. A better understanding was achieved on the machine-related factors and ore-related factors that affect the electrical comminution performance (Wang, Shi, & Manlapig, 2012a, 2012b, 2012c; Van der Wielen, Pascoe, Weh, Wall, & Rollinson, 2013).

Selective grinding is achieved based on the differences in brittleness of two materials. The brittle and ductile properties of a substance can change at a low temperature and the technique is referred as cryogenic comminution. Liquid nitrogen is normally used in the process and sometimes carbon dioxide can also be used (Daborn & Derry, 1988). Cryogenic comminution was used to separate metals, plastics, and metal/plastic systems. Kumar, Lee, Jeong, Jha, Kim, & Singh (2013) reported that the metallic values were recovered from PCBs by selective grinding followed by size classification. Experimental results show that the metallic grade reached 95% for an optimum size range. Many studies were carried out to recycle metals or plastics to find the optimum operational conditions. Compared to comminution processes performed at room temperature, the cryo-comminution improved the effectiveness of size reduction of metals or plastics, promoted liberation of constituents and increased the specific surface size of comminuted particles (Froelich & Maris, 2010; Gente La Marca, Lucci, Massacci, & Pani, 2004; Guo, Guo, & Xu, 2009; Koyanaka, Endoh, & Ohya, 2006; Liang & Hao, 2000; Wilczek, Bertling, & Hintemann, 2004).

Chemical comminution followed by separation methods based on density or surface chemistry provides a way of crushing coal to enhance liberation of mineral matter without excessive size reduction. Chemical comminution consists of the treatment of coal with a chemical, usually ammonia gas or a concentrated aqueous ammonia solution. Selective breakage of the coal takes place at the boundaries of coal and non-coal material, such as pyrite (Howard & Datta, 1977). The reason for chemical comminution is either due to the swelling of coal after adsorption of chemicals or due to the disruption of the bonding between the major components of coal by the chemical agents (Wheelock & Markuszewski, 1981).

Microwave treatment was studied on the selective fragmentation of oolitic iron ore (Song, Fabian Campos-Toro, & López-Valdivieso, 2013). The selective heating on different minerals leads hematite to expand much more than quartz, resulting in the formation of micro-fractures in the hematite and gangue mineral interfaces. The selective fragmentation could increase about 20-30% of relative liberation of hematite and gangue minerals from the tested oolitic iron ore samples. The application of the microwave energy in mineral processing was reviewed by several researchers (Haque, 1999; Jones, Lelyveld, Mavrofidis, Kingman, & Miles, 2002; Kingman & Rowson, 1998).

The above selective liberation processes are suitable for the following separation techniques, such as re-grinding, leaching, flotation, and gravity separation. Selective comminution, including cryogenic comminution, is suitable for size classification to obtain an upgraded product. Usually, the frequency of breakage events and the size distributions of the fragments produced in each event are used to describe a comminution process. The grinding rate and the product size distribution are determined by the above two factors (Hogg, Dynys, & Cho, 2002). In the present study, the first-order equation is the applied to show how selective comminution is achieved.

Modeling Selective Breakage Rates

As summarized by Quast & Hicks (1998), the breakage rate is rarely constant, for the following reasons: accumulation of harder minerals in the unbroken fragments in a mixed mineral system; a cushioning action of fines around coarse material; and/or decreasing efficiency of the grinding equipment with decrease in particle size. To model the non-first-order breakage behavior of a mixed mineral system, a two-component mechanistic model has been proposed by Gardner & Rogers (1975). In their study, the feed was composed of soft and hard particles, A and B, and there were no interactions between soft and hard minerals. The two-component mechanistic model has since been updated by incorporating the interactions between soft and hard minerals (Austin, Shoji, & Bell, 1982; Austin, Trimarchi, & Weymont, 1977). It is assumed that both the slow-speed and fast-breakage materials produce daughter fragments which in turn could break slow and fast. The two-component mechanistic model has been successfully applied in the grinding kinetics of coal in the Szego mill (Koka & Trass, 1985).

As suggested by Sudário & Luz (2012), two methods were used to model the selective comminution. The first method is to use the population balance approach to quantify the product size distribution (Bilgili & Scarlett, 2005; Fuerstenau, Phatak, Kapur, & Abouzeid, 2011). The second approach to model selective comminution was also based on population balance model (Ray & Szekely, 1973). An objective function based on maximization of the benefit that a selective comminution gave to the subsequent process with a penalty cost function for comminution. The difference between the mean sizes of the two components in the grinding product was used as quantification criterion. In the Sudário & Luz's study (2012), the evaluation of binary mixture differences was realized by sieving analyses and the estimation of Rosin-Rammler sharpness and median diameter. An objective function was conceived stressing the relationship between the expended energy in the grinding process and the optimum residence time.

In the selective comminution of nickel laterite ores, the interested parameters are grinding time and breakage rates with respect to various minerals and elements. Nickel upgrade was achieved on a SG sample by selective grinding of antigorite and quartz (Tong et al., 2013). Antigorite is one the main minerals in the SAP ore, so it was interesting to know how the mineral play the role in the selective grinding of SAP ore. Extensively test work has been done on the selective comminution of nickel laterites. In this paper, the nickel laterite samples are simply considered a mixture of soft and hard minerals. The effect of grinding time, breakage rate, and grinding feed was discussed through the first-order equation and the recent achievements were summarized.

EXPERIMENTAL

Minerals and Materials

<u>Sample Preparation</u>

Low grade SG, G and SAP nickel laterites from Western Australia were used in this study. For each nickel laterite type, sub-samples were prepared from the -15 mm Run-of-Mine (ROM) sample by crushing or wet screening.

<u>Chemical and Mineralogy Analysis</u>

SG, G and SAP ore samples were characterized by the quantitative X-ray powder diffraction (QXRD) and QEMSCAN to establish the mineralogical compositions (Swierczek, Quast, Addai-Mensah, Connor, Li, & Robinson, 2011, 2012). As a nickel containing mineral asbolane was detected through QEMSCAN. QEMSCAN method was not suitable to obtain accurate mineralogy information on nickel due to the low concentration and wide distribution of nickel in the host mineral. So, selective comminution was not discussed on asbolane. In this study, QXRD results on the samples are shown in Table 1. Quartz and antigorite are the major minerals in SG sample. Goethite and hematite are the major minerals in the G sample. Amorphous material, antigorite, chlorite, and quartz constitute the major minerals phases in the SAP sample. The chemical compositions of the samples are listed in Table 2.

Batch Grinding Tests

Grinding tests were carried out in a Netzsch LME4 horizontal stirred mill, under the following conditions: 1000 rpm, 50% charge volume, 150 g feed. Ceramic balls, Keramax MT1 (3 mm), were used as grinding media. After grinding, the products were separated into two size fractions, referred to as screen oversize and screen undersize. The screen undersize product was screened using the 400 mesh screen to get a -38 µm product. Selected grinding products were sent out for chemical and mineralogy analysis.

Table 1 – QXRD results on the SG, G, and SAP feed samples: wt%

Nickel laterite	Minerals
SG 1.68-1.18 mm	35.7% quartz, 34.9% antigorite, 18.1% goethite, 9.6% amorphous material, 1.2% chlorite, 0.4% trevorite
G 1.68-1.18 mm	45% goethite, 15.1% hematite, 11.8% amorphous material, 7.6% gibbsite, 4.5% kaolin, 3.6% antigorite, 4.5% anorthite, 1.4% quartz, 1.3% alunite, 0.7% trevorite, 3.1% talc, 0.6% $Mn(OH)_2$, 0.7% sepiolite
SAP 2.38-1.68 mm	35.4% amorphous material, 32.2% antigorite, 14.4% chlorite, 10.2% quartz, 3.4% hematite, 2.3% goethite, 1.4% trevorite, 0.6% chromite, 0.1% smectite-chlorite

Table 2 – Chemical composition of the SG, G, and SAP ore samples with various size fractions

Element	SG 1.68-1.18 mm	G 1.68-1.18 mm	SAP 2.38-1.68 mm
Si (%)	27.9	4.64	22.3
Ni (%)	0.73	0.77	0.66
Fe (%)	20.0	45.8	15.8
Mg (%)	1.38	0.17	7.95
Cr (%)	0.54	1.10	1.03
Al (%)	0.67	4.91	2.72
Co (%)	0.07	0.131	0.041

Data Treatment Method

When the breakage rate is a constant value with grinding time the breakage behaviour is called first-order (Equation 1). Where, $w_1(t)$ and $w_1(0)$ are the weight of the top size mineral at time t and time 0, respectively. The first-order equation leads to the rate-mass balance equation, which is also the base of the two-component mechanistic model (Austin, Bagga, & Celik, 1981; Austin & Luckie, 1972; Gardner & Rogers, 1975). In the study, the first-order equation was applied on the size reduction of both the soft mineral and the hard one (Equations 2 and 3). Where, w_{s-p} is the weight of the soft mineral in the grinding product; w_{s-f} is the weight of the soft mineral in the feed; s_s is the breakage rate of the soft mineral; w_{h-p} is the weight of the hard mineral in the grinding product; w_{h-f} is the weight of the hard mineral in the feed; s_h is the breakage rate of the hard mineral.

$$w_1(t) = w_1(0)\exp(-S_i t) \tag{1}$$

$$w_{s-p} = w_{s-f}[1 - \exp(-s_s t)] \tag{2}$$

$$w_{h-p} = w_{h-f}[1 - \exp(-s_h t)] \tag{3}$$

RESULTS AND DISCUSSION

The selective grinding of nickel laterites study was summarized based on both experimental tests and the first-order breakage model. The representative test results were introduced here aims to introduce the major findings in the investigation.

Effect of Grinding Time

As shown in Figure 1, the breakage behaviour with respect to sample mass and various elements indicates the non-first-order breakage behaviour mainly due to the slow breakage rate of hard minerals and the fast breakage of the soft minerals. Short grinding time is optimum for selective grinding and nickel upgrade. For the SG ore, there is great difference on the Mg recovery and Si recovery which is due to the selective grinding of antigorite and quartz. The Ni grade was upgraded from 0.73% in the feed to 1.22% in the -38 µm grinding product with 12% Ni recovery (Table 3). For the SAP ore, there is great difference on the Ni recovery and Si recovery due to the selective grinding of amorphous material and quartz. The Ni grade was upgraded about 49% with 33% Ni recovery in the -38 µm product. The G ore shows the least selective grinding achievement because it contains very little quartz.

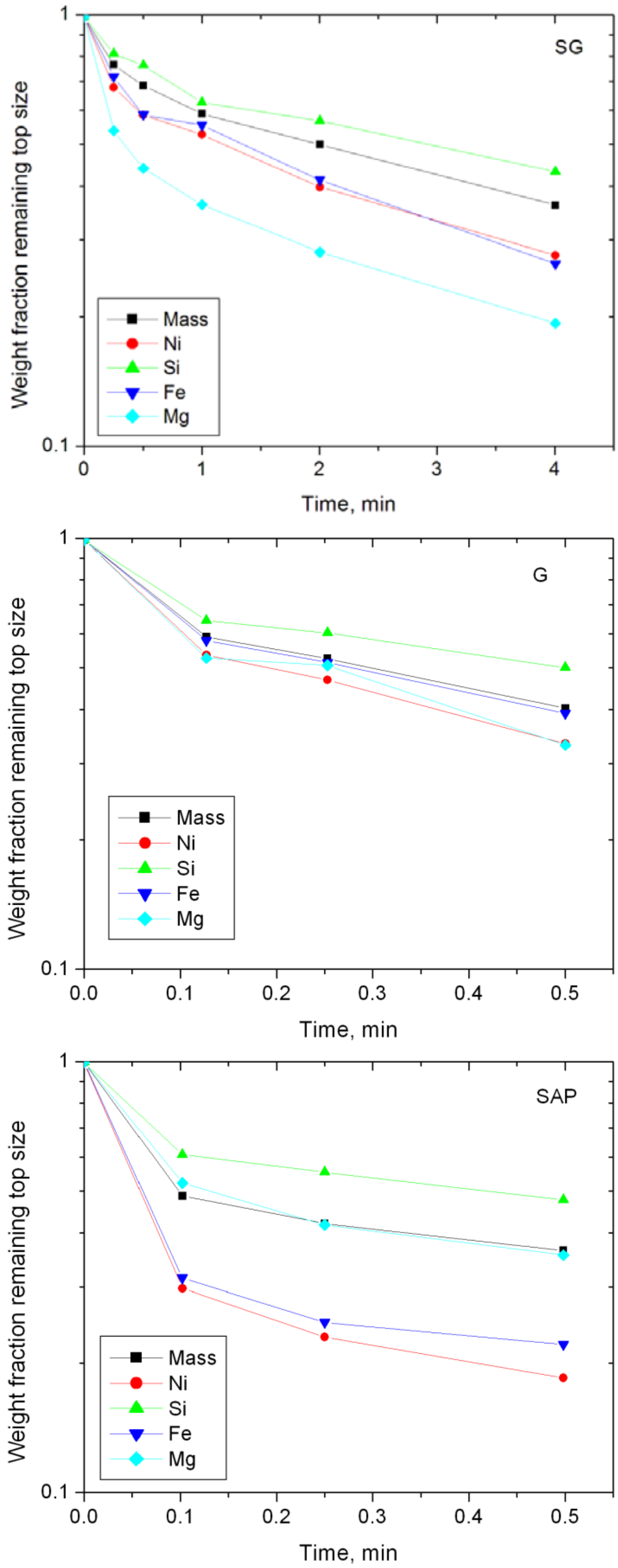

Figure 1 – Grinding behaviour with respect to the sample mass and various elements

Table 3 – Summary of the selective grinding results for the -38 µm product and screen undersize product

Nickel laterite	-38 µm product	Screen undersize product
SG	68% Ni upgrade, 12% Ni recovery	37% Ni upgrade, 32% Ni recovery 85% antigorite and 2% quartz recovery
G	43% Ni upgrade, 23% Ni recovery	13% Ni upgrade, 46% Ni recovery Selective grinding of minerals with various hardnesses
SAP	49% Ni upgrade, 33% Ni recovery	37% Ni upgrade, 70% Ni recovery 83% amorphous material, 6% quartz and 33% antigorite recovery

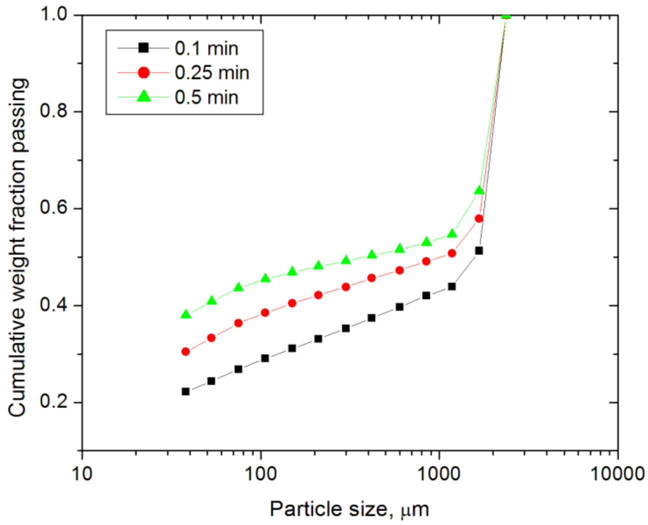

Figure 2 – Particle size distribution of the SAP nickel laterite grinding product: feed size 2.38-1.68 mm

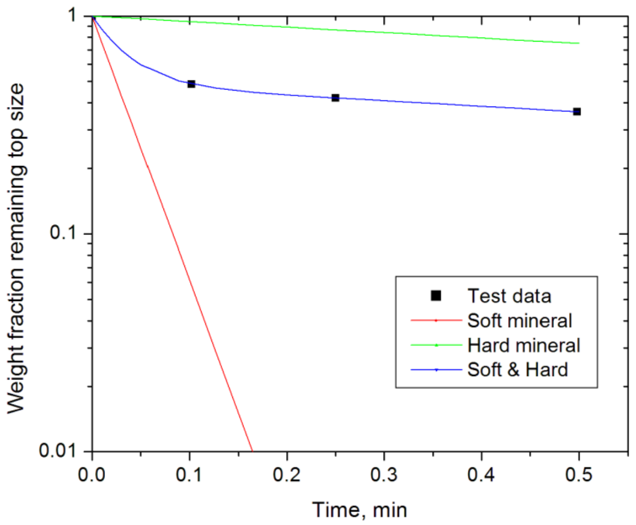

Figure 3 – Grinding behavior with respect to sample mass: SAP nickel laterite, assume the feed composed of soft and hard minerals, feed size 2.38-1.68 mm

Figure 2 shows the effect of grinding time on the particle size distribution of grinding products. It shows that about 50 wt% of the particles is not break after stirred milling for 0.1 min. Selective grinding requires the breakage of soft mineral particles and un-broken of hard ones. Figure 3 indicates that it is possible to simply assume that the feed is composed of relatively soft mineral particles and relatively hard ones.

The effect of grinding time on the grade of the soft mineral in the grinding product can be modelled through the first-order equation and the result is shown in Figure 4. The soft and hard mineral weight ratio is 1 in the feed and when there is no difference on the breakage rates, the grade of the soft mineral in the grinding product remains 50%. When the breakage rate of the soft mineral is a constant value, decrease the breakage rate of the hard mineral results in the improved grade of the soft mineral. Generally, the grade of the soft mineral in the product decrease with time.

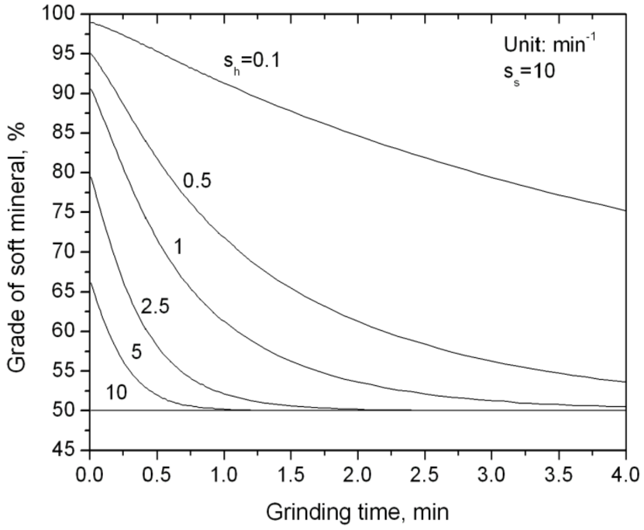

Figure 4 – Effect of grinding time on the grade of soft mineral in the screen undersize product

Effect of Breakage Rates

According to the test results shown in Figure 1, the breakage rates with respect to sample mass and the major elements were calculated (Figure 5). Generally, the breakage rates decrease with time and the optimal selective size reduction was achieved at the initial grinding stage. The test work proves the finding shown in Figure 4.

The effect of breakage rates on the soft mineral grade in the grinding product was discussed through the first-order equation (Figure 6). At a constant grinding time (0.25 min), the effect of selective grinding requires the slow breakage rate of hard mineral and the great difference between the breakage rates of soft and hard minerals. When reaches the maximum value, further increase the breakage rate of soft mineral has no effect on selective grinding.

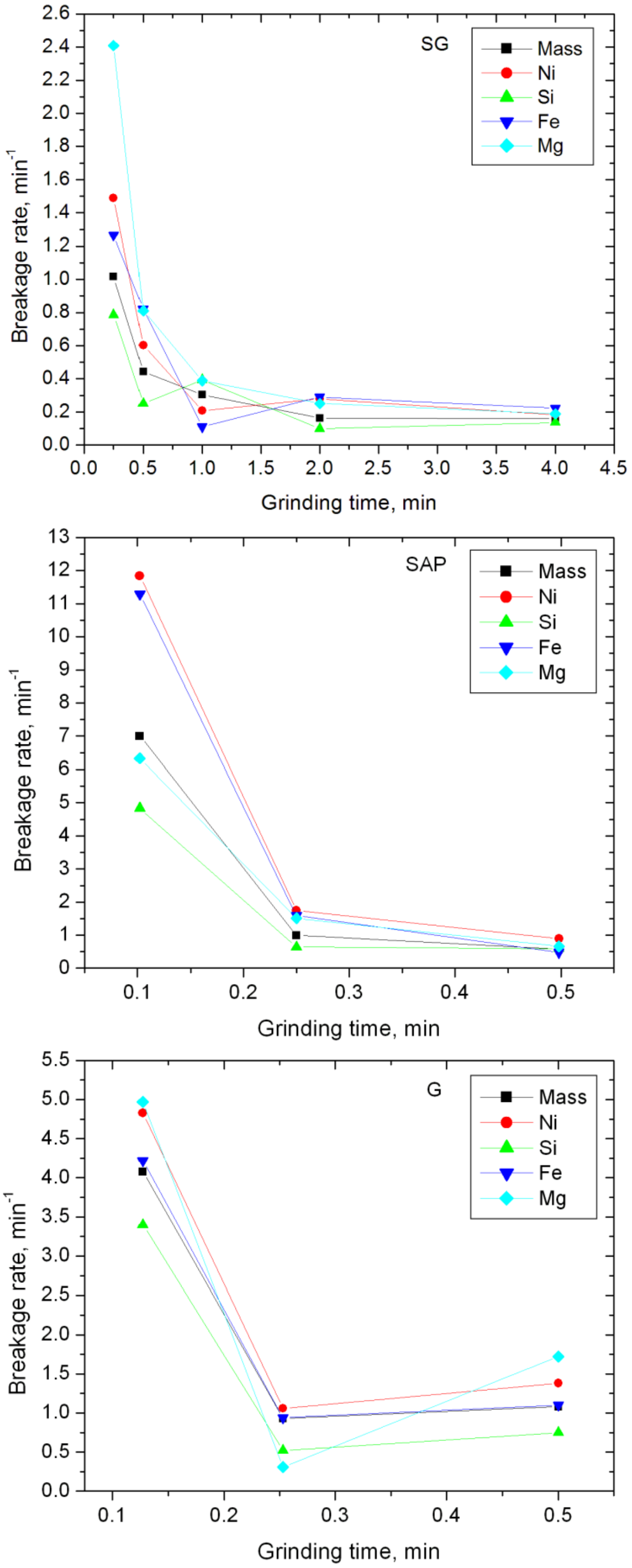

Figure 5 – Effect of grinding time on the rate of breakage of nickel laterites

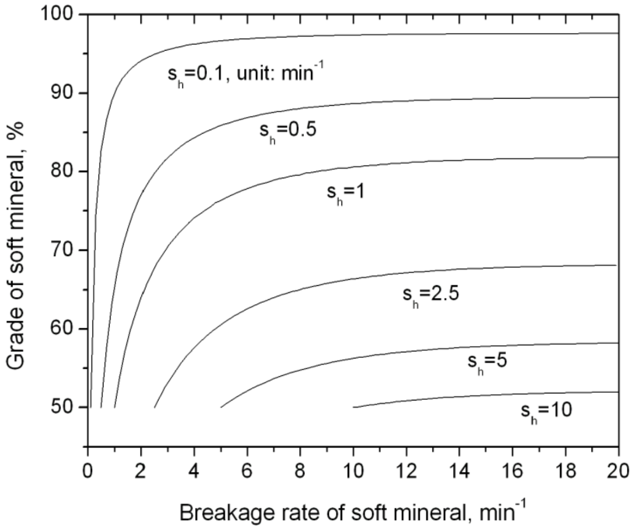

Figure 6 – Effect of breakage rates on the grade of soft mineral in the product: grinding time 0.25 min,

Nickel Recovery

Nickel recovery is an evaluation of the selective grinding through summarizing the breakage rates and grinding time. The nickel recoveries both in the screen undersize and the -38 µm product were summarized in Table 3. To achieve the operating targets, the nickel grade and the nickel recovery has to be balanced through controlling the grinding time.

Figure 7 shows the modelling results of the soft mineral recovery based on the first-order equation. The soft mineral recovery is determined by the breakage rate of soft mineral and grinding time which indicates that the nickel recovery is determined by both the selective grinding and nickel distribution in the feed.

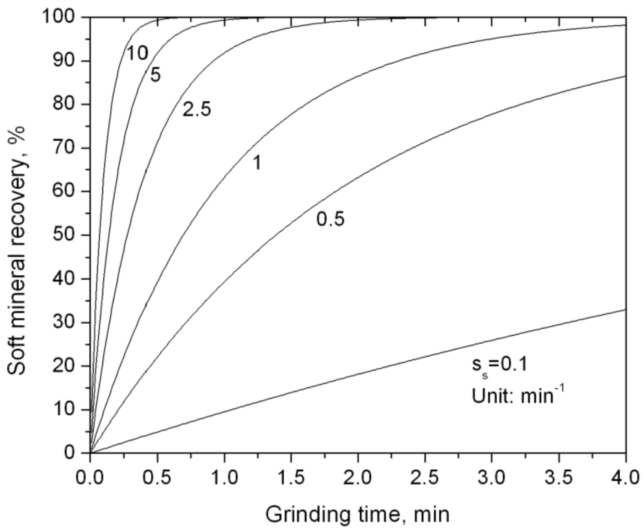

Figure 7 – Effect of grinding time on the soft mineral recovery

Effect of Weight Ratio of Soft and Hard Minerals

The test work shows that better selective grinding and nickel upgrade results were achieved on both the SG and SAP ores. It indicates that the mineralogy of the grinding feed has dramatic effect on selective grinding.

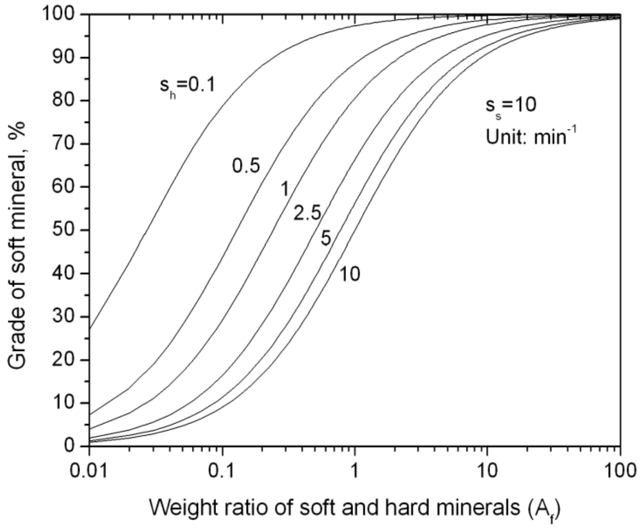

Figure 8 – Effect of minerals weight ratio on the grade of soft mineral in the screen undersize product

The effect of the grinding feed on the grinding effect was modelled through the first-order equation (Figure 8). In Figure 8, when the weight ratio is 1 and both the soft and hard minerals share the same breakage rate, 10 min^{-1}, the grade of soft mineral is 50% which indicates no selective grinding. No selective grinding line was drawn when the weight ratio ranges from 0.01 to 100. The lines above the no selective grinding line are all selective grinding lines which were determined by the breakage rate of the hard mineral. The area above the no selective grinding line and below the selective grinding line shows the limit of selective grinding. For example, for a soft mineral rich feed, there is only a small potential to upgrade it.

Effect of Nickel Grade in the Feed Minerals

The effect of nickel distribution on the feed nickel grade was discussed and the result is shown in Figure 9. The potential of nickel upgrade can be discussed through the Figure. For example, if the weight ratio of soft and hard mineral increases from 1 to 10, the effect of nickel upgrade can be read directly. Clearly, the nickel grade in the soft mineral determines the limit of nickel upgrade.

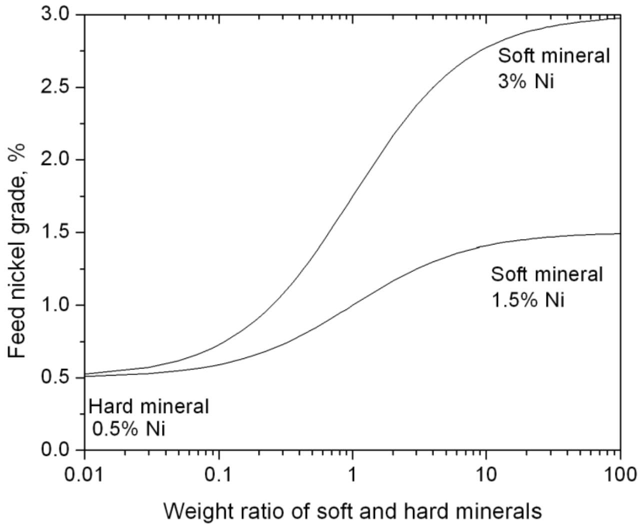

Figure 9 – Effect of nickel distribution on the feed nickel grade

<u>Process Development</u>

Batch grinding tests suggested a two-step nickel laterite upgrade process as shown in Figure 10. The grade/recovery relationships were summarized in Table 4. The first step in the upgrade process was size classification of the -2000 µm nickel laterite feed to recover the -38 µm fraction. The second step involved upgrading the nickel from stirred mill grinding of the +38 µm size fraction and then size classification to recover the -38 µm fraction. The combined fine fractions constituted the undersize product and the oversize was considered waste.

Selective comminution not only increased the Ni grade of the product but also had great impact on the following agglomeration and heap leaching processes. The nickel laterite agglomerates formed from SG sample are robust enough to withstand a heap height up to 3.5 m without collapse. The satisfied agglomerates formed from SAP and G samples can also be achieved by optimizing particle size, binder content, moisture content, and drying conditions. The Agglomeration of the fine mineral particles obtained through selective grinding results in the enhanced leaching rates and metal recoveries due to the increase in primary particles' surface area.

Table 4 – Size classification of the -2,000 µm nickel laterites and selective grinding the screen oversize fraction

Sample	Feed	Size classification				Selective comminution				Total product		
	-2000 µm	-38 µm	-38 µm	-38 µm	+38 µm	-38 µm	-38 µm	-38 µm	+38 µm	-38 µm	-38 µm	-38 µm
	Ni (%)	Ni (%)	Wt (%)	Rec (%)	Ni (%)	Ni (%)	Wt (%)	Rec (%)	Ni (%)	Ni (%)	Wt (%)	Rec (%)
SG	1.15	1.50	46.0	60.0	0.85	1.37	17.3	27.8	0.79	1.48	55.3	71.1
G	1.02	1.13	56.2	62.6	0.87	1.05	21.1	25.4	0.83	1.12	65.4	72.1
SAP	0.92	1.11	56.3	67.8	0.68	1.00	15.2	22.4	0.57	1.10	62.9	75.0

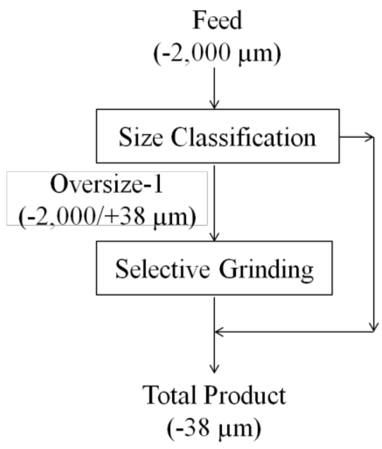

Figure 10 – General nickel upgrade processes

CONCLUSIONS

It is possible to upgrade nickel through selective comminution of siliceous goethitic and saprolitic nickel laterite ores. Batch grinding tests suggested a two-step nickel laterite upgrade process involves size classification and selective comminution.

The conditions that influence selective grinding include the breakage rate of hard minerals, the breakage rate of soft minerals, grinding time, and the weight ratio of soft and hard minerals in the feed. The conditions that influence nickel upgrade include both the selective grinding conditions and the nickel distribution in both the soft and hard minerals.

The effect of selective grinding requires a slow breakage rate of hard mineral and the great difference between the breakage rates of soft and hard minerals. When reaches the maximum value, further increase the breakage rate of soft mineral has no effect on selective grinding. Both the effect of selective grinding and nickel upgrade decreases with the grinding time.

The weight ratio of the soft and hard minerals in the feed determines the effect of selective grinding. When the selective grinding is achieved, the effect of nickel upgrade in selective comminution of nickel laterite is determined by the nickel grade in the soft mineral.

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