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The effect of grinding mechanism on the preg-robbing of gold onto quartz

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This study investigates the effect of different grinding processes on the preg-robbing behavior of quartz. The adsorption of gold onto the quartz surface in a chloride medium is determined after grinding in a ball mill with steel media, and in a ring mill. The mechanical activation of the ground quartz is significantly influenced by the type of mechanical action in the mill; the compression–shear action of the ring mill seems to be more effective than the impact–attrition action in a ball mill. As a result, the grinding limit is lower in the ball mill, and agglomeration is not significant after the grinding limit. Grinding yields significant propensity towards adsorption of gold chloride complexes from solution, firstly due to an increase in surface area in material treated in both the ball and ring mills, and secondly due to the activation of the quartz surface, which mainly takes place in the ring mill. The preg-robbing of gold on the ground quartz was measured to be as high as 0.38 μmol/m^2 on the ring milled quartz, while the corresponding value did not exceed 0.15 μmol/m^2 after 360 min of ball milling. The rate of preg-robbing in ball milled products is also slower compared to that of the ring milled products, indicating geometrical or steric constraints on accessibility of the distorted (defect) quartz sites to gold complexes sorbing from the solution, which is consistent with the existing understanding of the different defect site locations induced by these two different grinding mechanisms.

1. Introduction

The effect of high-energy milling on the surface reactivity of quartz as it relates to the preg-robbing of gold chloride has recently been investigated using a combination of sorption testing and spectroscopic techniques (Mohammadnejad et al., 2011, 2013a,b). It has been shown experimentally that ring milling results in significant modification of the particle surface, such as amorphization of the surface, lattice distortions and defect formation, in addition to particle breakage, an increase in the surface area and agglomeration. The extent and type of surface alteration appear to be strongly dependent upon grinding conditions, but the interrelationship between the different grinding mechanisms and surface activation, and hence the extent of preg-robbing of gold by ground quartz, has not been investigated before.

Extensive work has been done on the mechanochemical activation of quartz and other silicates in laboratory mills (Bernhardt et al., 1974; Heegn, 1990; Rask et al., 2010; Tranfield et al., 2009; Zbik and Smart, 2005). However, these studies have not focused on the effect of mechanochemical activation on downstream operations. This paper compares the preg-robbing propensity of ground quartz, as an undesired result of mechanochemical activation in gold processing, in two different types of mills, i.e. a laboratory ring mill (vibratory disk mill or pulveriser) and a ball mill with steel media, representing two different mechanisms of grinding. The results are used to shed more light on the type and extent of mechanical activation generated by different types of grinding mechanisms.

2. Materials and methods

A quartz sample in the size range of 0.1–2.5 μm was supplied by Unimin Australia, containing 99.99% SiO_2. The sample was dried at 50 °C overnight before grinding for adsorption tests and surface area measurements. The sample was dry ground in a Labtechnics LM1 Standard series ring mill for intervals of 30 s, 1, 5, 15 and 30 min, as well as a steel Paul O. Abbe ball mill for intervals of 30, 120, 240 and 360 min. The specifications of the mills are provided in Table 1. Before grinding of any samples, the mills were loaded with coarse quartz sand (0.3–2.5 mm in size) to remove any oxide scale or other contamination from the surface, and then washed with water and fully dried. Batches of 20 g in the ring mill and 200 g in the ball mill were ground. During grinding, the temperature of the rings and bowl of the mill rose significantly (up to 82 °C after 30 min of grinding).

To investigate the nature of surface alteration by grinding, samples were characterized by nitrogen sorption, using an ASAP2010 instrument (Micromeritics, USA) and the BET method, after degassing at

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Table 1
Specifications of ball and ring mills used for grinding of quartz.

Parameters	Ball mill	Ring mill
Internal Diameter (mm)	213	140
Height (mm)	132	42
Volume (cm^3)	4700	650
Media	Total media mass = 9540 g	Ring = 75 mm
	Number of balls and size (mm)	Puck = 110 mm
	2 × 18	
	7 × 26	
	23 × 27	
	4 × 31	
	5 × 37	
	22 × 40	
Power (kW)	1.5 kW	1.1 kW

110 °C for 6 h. Preg-robbing experiments were carried out at room temperature and pressure, using an acidic chloride solution with 50 g/L of as-received or ground quartz, and 10 ppm gold solution (supplied as 1000 ppm $HAuCl_4$ solution, Sigma-Aldrich Australia, diluted with RO-grade purified water) immediately after grinding. The initial pH of the solutions with 10 ppm chloroauric acid was 1.9 and no pH adjustment was made at the beginning or during the tests. It rose up to 2.0–2.3 after dissolution of silicates and remained almost constant.

The sorption tests were performed in glass beakers with 500 mL of chloroauric acid per test, under dynamic conditions using a magnetic stirrer. Samples of 10 mL were taken at regular time intervals, without replacement, and finally at the end of the run. The specimens were then centrifuged, filtered with 0.2 μm polycarbonate membrane filters, and finally diluted (10×) with distilled water for analysis by inductively coupled plasma optical emission spectroscopy (ICP-OES; Varian Instruments). The concentrations of Si and Au in the diluted solutions were analyzed for all samples.

3. Results and discussion

Fig. 1 shows the evolution of specific surface area of quartz with time, on a logarithmic scale. The alteration of surface area in comparison with the average particle size has been discussed before in the case of a ring mill (Mohammadnejad et al., 2013b). Particles undergo brittle breakage in the first stages of grinding (first 1 min in the ring mill and 120–240 min in the ball mill). After approaching a certain particle size (the size, or surface area, defined as the grinding limit), agglomeration as the result of surface activation starts, which is reflected by a steady or decreasing trend in the surface area.

The energy intensity of a mill depends on the grinding mechanism as well as the operating conditions. The mechanical load in a mill can be delivered by mechanisms including compression, shear, attrition, stroke and impact (Baláž, 2008; Tarján, 1981). It is shown here that the mechanical activation of ground products is significantly influenced by the type of mechanical action in a mill, and hence by the mill type. A combination of different types of grinding mechanisms takes place in

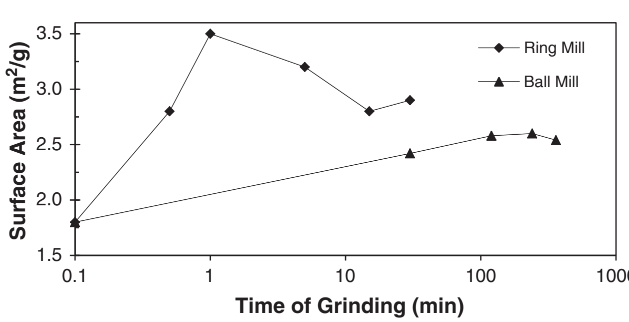

Fig. 1. Surface area of original quartz, and products ground in a steel ball mill and a ring mill.

most mills, while the ratio between different actions varies depending on the structure and operating regime of the mill (Prokof'ev and Gordina, 2012). According to Tarján (1981), the key operating mechanism in a ball mill is impact–attrition, while in a ring mill it is compression–shear. A ring mill has been reported to be more effective and energy intense in comparison to a ball mill (Andrić et al., 2012; Prokof'ev and Gordina, 2012).

The power intensity in terms of the ratio of installed motor power to volume of the grinding chamber has also been calculated for both mills. The disk ring mill with a power intensity of 1700 kW/m^3 (650 cm^3 grinding chamber and 1.1 kW power) is much more intense than the laboratory ball mill with a power intensity of 320 kW/m^3 (4700 cm^3 grinding chamber and 1.5 kW power).

The grinding limit is lower in the ball mill compared to the ring mill (2.6 against 3.5 m^2/g), which is a result of the lower energy intensity in the ball mill; this is where transition from fine grinding to mechano-chemical activation happens. In the ring mill, because of the higher energy intensity, the milled particles undergo significant surface activation which is reflected in the negative trend of surface area. This is where agglomeration of the particles is the dominant mechanism. In the ball mill, even after the grinding limit, the surface area trend levels off and agglomeration does not seem to be significant, and extending the time of grinding from 120 to 360 min does not change the surface area significantly. The low power intensity in the ball mill is probably not enough for surface activation of the quartz grains compared to the significant agglomeration effect observed in the ring mill.

The results of sorption tests demonstrate interaction between dissolved gold, and the original and ground quartz surfaces (Fig. 2). The ring milled samples adsorbed between 57 and 98% and ball milled samples 12 and 37% of the gold from the 10 ppm solution, while the un-milled sample adsorbed less than 3% of the gold in the solution after 480 min of contact. The specific adsorption onto the quartz milled using a ball mill was considerably lower compared to that of the ring mill even after extensive grinding for 360 min. Also, prolonging the grinding of quartz in the ball mill from 240 to 360 min did not result in higher surface activation, which is in agreement with the surface area results as well as the previous studies (Mohammadnejad et al., 2013b). As explained, this appears to be due to the role of fines in protecting the larger particles.

The preg-robbing is measured to be between 0.01 and 0.38 $\mu mol/m^2$ on quartz ground in the ring mill, while preg-robbing by ball milled products does not exceed 0.15 $\mu mol/m^2$. Preg-robbing is also observed on the unmilled quartz (0.01 $\mu mol/m^2$); however the sorption is much lower in comparison to the milled samples, especially the ring mill products. The gold uptake per surface area of the unmilled sample is noticeably lower than what was measured in the previous work (Mohammadnejad et al., 2011), which was in the range of 0.04–0.28 $\mu mol/m^2$. This is probably because of the lower pH of 1.9–2.3 for the 10 ppm chloroauric acid solution used here in comparison to the pH of 2.5–3.0 for the 5 ppm solution used in the previous experiments. The zero point charge of quartz is reported to be at a pH of 1.8 (Fuerstenau and Han, 2003; Tikhomolova et al., 2001), so the present quartz surface will have less negative deprotonated silanol groups (≡SiO−) which attract less protons, resulting in less electrostatic adsorption of $AuCl_4^-$ anions. Mohammadnejad et al. (2013a) also showed that hydroxyl groups on the surface of quartz reduce Au^{3+} to Au^0 via a complex mechanism, so that a less negative surface will result in less hydroxyls and hence lower overall gold adsorption. Grinding results in the formation of new surface defects including low valence silicon and non-bridging oxygen centers (Mohammadnejad et al., 2013a) which is reflected in higher gold uptake per surface area for both types of mills.

An additional consideration is the kinetics of the reactions in two different types of ground products. Fig. 2 shows that in the unmilled sample, as well as ring milled products, the adsorption process is fast, as equilibrium conditions are reached within the first 30–60 min of running the experiment for almost all samples, consistent with the previous