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ScienceDirect
Int. J. Miner. Process. 82 (2007) 195–202
INTERNATIONAL JOURNAL OF MINERAL PROCESSING
www.elsevier.com/locate/ijminpro

Study on mechanochemical effect of silica for short grinding period

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Received 30 May 2006; received in revised form 16 October 2006; accepted 19 October 2006
Available online 29 November 2006

Abstract

Silica was ground in an oscillating mill at various grinding period to study the mechanochemical effect in fine grinding process. The ground particles exhibited massive size reduction where the volume moment diameter of 5.56 μm was reached within 600 s. Aggregation of fine particles was very pronounced when it was ground for 600 s due to high surface energy. Aggregation of fine particles caused the ground particles to exhibit poly-modal particle size distribution. Line broadening and reduction of diffractogram peak intensity were observed. Amorphization rate up to 16.9% was exhibited by the particle ground for 600 s. Preferential breakage of plane was observed where (101) and (111) was easily distorted compared to (110) and (200). Rapid reduction of crystallite size was observed at early stage of grinding until it reached a plateau at 5 nm at 600 s whilst the change in lattice strain was 0.5%.

Keywords: Mechanochemical; Oscillating mill; Fine grinding; Short grinding period

1. Introduction

Severe and intense mechanical actions on the solid surface are known to have physical and chemical changes on the near surface region where the solids come into contact under mechanical forces (Venkataraman and Narayanan, 1998). This phenomenon is termed as mechanochemical effect. Mechanochemical effect is very pronounced in the high intensity grinding mills such as planetary mill, oscillating mill, vibration mill and jet mill. These grinding mills deliver huge amount of energy for particle breakage and it is normally used to produce fine particle below 10 μm and submicron sizes. During mechanical stress on the solid particles, some energy will be used for particle breakage whilst part of the energy will be stored in the material (Iguchi and Senna, 1985). Most of the non-metallic minerals have low thermal conductivity characteristics so the energy delivered will not be stored as thermal energy but will be applied to bending or breaking of crystal which leads to structural alterations by loss of regularity in the crystalline network (Aglietti et al., 1986a,b). Besides loss of crystalline network regularity, the mechanochemical effects also bring about loss of crystallinity (amorphization), increase in surface energy, dislocation, stacking faults, non-uniform vacancy concentration, phase transformation and chemical reactivity (Aglietti et al., 1986a,b; Kanno, 1985; Begin-Colin et al., 1999). Juhasz and Opoczky (1990) suggested that 3.7% of the

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0301-7516/$ - see front matter
doi:10.1016/j.minpro.2006.10.008

energy supplied to vibration mill is used for phase transformation besides the major portion which are lost as heat and mechanical losses.

The study on mechanochemical effect on fine particles has created much interest among the researchers for the past 20 years as it has several advantages to the downstream processes such as reducing the annealing and sintering temperature, surface tension, accelerate the rate of densification in ceramics powder, increase disintegration time and dissolution rate of ethenzamide of tablets in pharmaceutical products, production of porous minerals, increase the reactivity of cementitious waste materials, reduction in phase transformation temperature, enhance leaching process, decrease thermal decomposition temperature, increase in particle reactivity and in environment (Venkataraman and Narayanan, 1998; Kanno, 1985; Begin-Colin et al., 1999; Terada and Yonemochi, 2004; Hu et al., 2003; Benezet and Benhassaine, 1999; Fernandez-Bertran, 1999; Gonzalez et al., 2000; Zhang et al., 1997; Kano et al., 2000; Ryau, 2004; Yang et al., 2005; Sanchez et al., 2004; Wu et al., 1996). Although there are several advantages of mechanochemical process, agglomeration of particles is a major problem when dealing particles below 10 μm especially those particles with high surface energy. Juhasz and Opoczky (1990) suggested that there are three stages of interaction between the particles which are adherence, aggregation and agglomeration. At adherence stage, the particles will coat on the lining and the grinding bodies. At aggregation stage, the particles associate weakly by Van der Walls type adhesion and it is a reversible reaction. Agglomeration is defined as a very compact, irreversible interaction of particles with occurrence of chemical bonding between the particles. Agglomeration is detrimental for the quality and activity of the ground product (Juhasz and Opoczky, 1990). Currently grinding aids is used to avoid particle agglomeration during the production of fine particles.

Forces acting during fine grinding process are impact, attrition, shear and compression. The combination of these forces induces particle breakage and mechanochemical effect. Impact plays an important role in the rupture of particles, and attrition acts between the line and the grinding media. The shear and compression forces play an important role in inducing mechanochemical effect (Gonzalez et al., 2000). In oscillating mill, the compression and the shear forces are the main forces acting on the particles.

Silica is a favorite model substance of researchers who deal with comminution and mechanical activation. Silica is a material with simple chemical and crystal structure, obtainable in high purity, crystallizing readily and having favourable mechanical properties. Therefore SiO_2 has been recognized at an early date and has been thoroughly investigated (Juhasz and Opoczky, 1990). Juhasz and Opoczky (1990) reported that size reduction was dominant in the early stage, but after prolonged grinding, the main process was amorphization accompanied by agglomeration and recrystallization. After prolonged grinding process, quartz maintain its amorphism due to high requirement of energy for crystallization. The amorphous layer developed on the surface of the particles. Based on the amount of the amorphous materials and the value of specific surface area, the thickness of amorphous layer on coarsely ground silica was 2 nm. However this value increased up to several tens of nm and finally after prolonged grinding, the entire particle would become amorphous. Benezet and Benhassaine (1999) suggested that silica structure can't be modified by grinding process, whilst Kanno (1985) showed that SiO_2 changed to amorphous phase after 120 h of grinding in vibrating mill. This paper will focus on the mechanochemical effect on silica particles in terms of its crystallinity, crystallite size, lattice strain and aggregation of fine particles which take place during very short grinding period in an oscillating mill.

2. Experimental

High purity silica from Johor supplied by Sibelco Asia, which was well crystalline with an average particle size of 13.38 μm was used for the experiment. The dry grinding tests were carried out in an oscillating mill which consisted of three steel ring as the grinding media. This grinding machine worked through friction and impact caused by movement of the rings and a concentric cylinder, placed within a casing containing the material to be ground. The dimension of the mill is shown in Table 1. Mechanochemical process was carried out in air atmosphere under closed condition where the grinding mill was kept closed during grinding process. The amount of material used for each run was

Table 1
Experimental milling condition

Parameter	Value
Grinding media weight (kg)	5.92
Media filling (%)	42.7
Media to powder ratio (%)	12.8
Amplitude (mm)	9
Media density (kg/m^3)	7450
Mill diameter (cm)	22
Mill height (cm)	4.8
Grinding media diameter (cm)	18.5, 13.5 and 8.0