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Mechanochemical effect of dolomitic talc during fine grinding process in mortar grinder

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ABSTRACT

Dolomitic talc was milled in a mortar mill by varying the milling time, solid content, and vertical stress. The milled samples exhibited massive particle size reduction and came to a threshold value around 4 µm, with wider particle size distribution. The breakage mechanism of the dolomite and talc phase was influenced by hardness, crystallographic structure, and Young's Modulus. The milled particles underwent massive mechanochemical effect where the degree of crystallinity ranged from 40% to 50%. The variation of lattice parameters of both phases was influenced by mill operational parameters and breakage mechanism of the particles in the mill.

1. Introduction

Talc is an industrial mineral composed of hydrated magnesium-sheet silicate with theoretical formula of $Mg_3Si_4O_{10}(OH)_2$. It is widely used in many products such as paint, paper, plastic, polymer, rubber, fertilizer, insecticide, ceramic, and cosmetics as filler material or raw material due to its inertness, whiteness, low thermal and electrical conductivity, and absorption of organic substances capacity. Due to its unusual combination of properties, brought about by the high degree of anisotropy of its crystal structure, it has created much interest in technological applications. For most of these applications, talc is used as a high-purity fine powder. World production of talc in 2007 was 7.62 million tonnes. Ultrafine talc is mainly used in paint, paper, ceramic, and plastic. The occurrence of gangue minerals such as carbonates, magnesite, dolomite, serpentine, chlorite, and calcite in talc deposits contribute to the production of undesirable characteristics. Currently, this problem is being overcome through the floatation or air classification techniques. It would be an advantage if low-grade talc with impurities can be used without purification as the impurities also have certain advantages when used as fillers. Dolomite is widely used as filler in the paint, paper, and adhesive industries. On the other hand, Gai et al. (2005) incorporated and prepared composite calcium carbonate and wollastonite powders polypropylene and found that the mechanical properties of the composite mineral powders were improved compared to those containing the original mineral powders. This finding created a new avenue to use naturally-occurring composite minerals as filler material in various industries.

Among the industrial processes, grinding is used in a broad range of industrial applications to produce fine particles. Lately, the product size specifications for advanced materials have become extremely fine, sometimes approaching nanosize range. However, it is very difficult to produce particles in this size range through mechanical milling. Moreover, milling is expensive due to low capacity (kg/h) and high energy consumption (Cho et al., 2006). Thus, this area is regarded as a major area for development (Wang and Forssberg, 2007). The fine milling methods that have been intensively studied include stirred media mill, jet mill, planetary mill, vibratory mill, and mortar mill. Recently, much attention was given by various researchers in evaluating the possible changes of its main properties during processing because specific treatments such as grinding and sonication could modify intrinsic characteristics (Sanchez-Soto et al., 1997; Perez-Maqueda et al., 2005). Mechanochemical effect resulting from structural distortion of crystal lattice during fine grinding has also created much attention among the researchers due to several exhibited advantages. These advantages include reduction in sintering temperature, increase in pozzolonic properties of cement filler, enhancement of leaching, production of nanocrystalline materials, improvement of reactivity of waste materials to be used as construction materials, transformation of phases, and production of a new phase (Sanchez et al., 2004; Benezet and Benhassainne, 1999; Zhang et al., 1997; Begin-Colin et al., 1998). Antasari et al. (2006) mentioned that milling mechanism plays an important role on the mechanochemical effect, especially with layered-type minerals. For instance, in graphite processed in planetary ball mill, where the shear

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component in the applied stress is dominant, highly anisotropic particles with relatively well-preserved structure are obtained, whilst amorphous and nanoporous carbon often resulted from milling with shock-type ball impacts (Antasari et al., 2006). The morphological and structural properties of layered silicate minerals appear to be relevant for their technological applications. It has been reported that particles with well-preserved crystal structure and a high geometrical anisotropy with nanometer range particle thickness improves the mechanical properties of polymer composites compared to nanometer-scale powder (Li et al., 2005). Therefore, it is essential to choose the milling mechanism that will most likely result in the above-mentioned characteristics. Comminution takes place in mortar grinding mill, thus, it should be interesting to further explore processing routes based on plastic deformation, where an increase of the specific surface area of layered silicate is obtained while preserving some integrity of the crystalline structure. Considering that the shear mode of deformation appears more suitable to produce talc nanoplatelets and that talc does not require a particularly high stress for plastic deformation, pure shear stress was carried out in this work on the microstructural evolution of talc submitted to prolonged milling under low intensity. The main aim is to enhance the contribution to plastic deformation of the easy gliding (001) lattice planes, reducing the effect of other deformation mechanisms, which require higher stress for activation and can induce a larger crystal disordering of dolomatic talc.

2. Methodology

Commercial dolomitic talc powder from Liaoning, China with particle size d_{50} 9.38 μm with span value of 0.835 was taken in as raw material. X-ray fluorescence analysis was done using Rigaku X-ray RIX 3000 to determine the chemical composition of the dolomitic talc. Table 1 shows chemical composition of dolomitic talc.

Fine grinding was performed in wet mode in a Retsch mortar grinder model RM 200. The milling mechanism in this mill is pressure and friction action. The grinding pressure is achieved by the weight of the pestle combined with the adjustable spring pressure acting on its axis. The pestle rotates simultaneously with the grinder bowl causing friction of material to the grinder. Motor gear rotates the mortar bowl and pressure is applied in both horizontal and vertical directions towards the mortar wall, imposing friction-causing particle sliding. A standard integrated scraper turns the sample and guides it back to the pestle. Fine grinding was carried out in wet mode by varying percent solid, milling time and pressure. Table 2 shows the range of operational parameters chosen in this testwork.

The $d(4.3)$ was calculated with Sympatec Rodos laser particle size analyzer with x_k number percentage of detected diameter d_k, as shown in Eq. (1) (Lecoq et al., 1999).

$$d(4.3) = \left(\sum x_k d_k^4\right) / \left(\sum x_k d_k^3\right) \quad (1)$$

The span value ψ showed the particle size distribution of the samples, calculated using Eq. (2), from the values obtained from the particle size analysis data where diameter indicating that i% is smaller than d_i ($i = 10\%$, 50%, and 90%) (Nakach et al., 2004).

$$\psi = (d_{90} - d_{10}) / 2d_{50} \quad (2)$$

The mechanochemical effect was determined through X-ray diffraction (XRD) whilst the density was determined using MICROMETRICS pycnometer. Surface morphology of the particles was determined using Scanning Electron Microscopy (SEM) Model ZEISS SUPRA 35VP.

Table 1
Chemical composition of dolomatic talc.

Composition	Weight %
SiO_2	51
MgO	34
CaO	15
Fe_2O_3	0.2
Al_2O_3	0.051
P_2O_5	0.019

Table 2
Operational parameters of mortar mill.

Operational parameter	Level		
	Low	Medium	High
Milling time (min)	30	90	150
Solid content (%)	10	30	50
Pressure (kN/m^2)	10	15	

3. Results and discussion

3.1. Effect of grinding to particle size distribution and span value

The particle size distribution of untreated and milled samples at three levels of solid content, and milling time at two levels of vertical stress, are shown in Fig. 1. It could be seen that the distribution is essentially polymodal, which permits the use of volume moment diameter to characterize the results and consequently to define the product fineness criteria based on volume moment diameter. Fig. 1 shows there are two groups of milled samples which are being influenced by milling time. Furthermore, the particle size distribution becomes wider as the milling progresses with the variation of the above-mentioned operational parameters. The milled sample exhibits two types of population: the first type has a mode at around 12 μm region, whist the second type exhibits mode around 5 μm region with a much wider distribution. Prolonged milling up to 50 min with lower solid content results in wider particle size distribution, as shown in Fig. 1, whilst short milling time with higher solid content produces narrower distribution. In addition, the milled samples exhibit increase in the amount of submicron particles. The generation of submicron particles increases as the milling time and solid content rise. Enhancement of vertical stress is only influenced at lower solid content. The milling mechanism in the mortar mill has an influence on the particle size distribution of milled particles and generation of submicron particles. The size reduction is induced in solid materials through various modes of stress application, including slow compression, fast compression, impact, or abrasion. Effective size reduction depends on the strength of materials and type of milling machine. The size reduction mechanism in this mill is facilitated by pressure and friction. Pressure by the mortar and pestle will result in compression breakage and larger particles, while friction produces finer particles due to abrasion breakage. Larger generation of fine particles in polymodal particles size distribution is due to abrasion breakage, which usually takes place at higher solid content in wet milling. At higher solid loading, the sharp edges of the particles will be trimmed off, producing ultrafine particles in submicron range, while the larger particle will be more rounded.

Fig. 2 shows the particle size, quantified through volume moment diameter, as the milled particles exhibit polymodal distribution. Generally, increase in milling time significantly decreases the particle size. The size reduction is induced to particles through various modes of stress application among which are: slow compression, fast compression, and impact or abrasion. It also depends on the strength