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Effect of mill type on the size reduction and phase transformation of gamma alumina

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HIGHLIGHTS

- We study the effect of stress modes from three mills on structure of gamma-alumina.
- Extent of size reduction and mechanochemical effects are analysed.
- Jet milling is effective in size reduction and does not initiate mechanochemistry.
- Shear-induced phase transformation is observed in planetary ball mill.
- Transformation is by slip on alternate close packed oxygen layers from ccp to hcp.

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ABSTRACT

The influence of stress modes and comminution conditions on the effectiveness of particle size reduction of a common catalyst support; γ-Alumina is examined through a comparative assessment of three different mill types. Air jet milling is found to be the most effective in reducing particle size from a d_{90} of 37 μm to 2.9 μm compared to planetary ball milling (30.2 μm) and single ball milling (10.5 μm). XRD and TEM studies confirm that the planetary ball mill causes phase transformation to the less desired α-Alumina resulting in a notable decrease in surface area from 136.6 m^2/g to 82.5 m^2/g as measured by the BET method. This is consistent with the large shear stresses under high shear rates prevailing in the planetary ball mill when compared to the other mill types. These observations are consistent with a shear-induced phase transformation mechanism brought about by slip on alternate close packed oxygen layers from a cubic close packed to a hexagonal close packed structure.

1. Introduction

Milling is a widely used industrial operation common for cases where size reduction of particles is required (Reid et al., 2008). It can also be known as grinding and involves the size reduction of particles smaller than 10 mm. There is a vast range of mill types available commercially and the choice of mill is based on a variety of factors, such as properties of the material to be milled, e.g. failure mode, and the required product particle size (Angelo and Subramanian, 2008). Fig. 1 shows an array of size reduction equipment available for different combinations of feed and product particle sizes (Neikov et al., 2009).

Ball mills, vibratory mills, rod mills and jet mills can be used to achieve particles less than 1 mm in diameter (Rosenqvist, 2004) but for ultrafine dry milling, e.g. particles ($d_{90} = < 10$ μm), vibratory ball milling, planetary ball milling (Kano et al., 2001) and air jet milling (Midoux et al., 1999) are commonly used methods. In these mills particle size is reduced by impact, shear, attrition or compression or a combination of them (Balaz et al., 2013). The stresses may affect product attributes in different and often 'unexpected' ways through mechanochemical activation, so an understanding of the mill function on the product characteristics is highly desirable for optimising product functionality. The material investigated in this paper, γ-Al_2O_3, is a versatile material used in many applications, including catalysis for the petroleum and automotive industries (Oberlander, 1984; Wefers, 1990). It is widely used for catalytic applications due to its favourable properties which include a high surface area and porous morphology for good dispersion of metal catalysts as well as thermal and chemical stability for use in different catalytic reactions (Trueba and Trasatti, 2005; Rozita et al., 2013). However, in order for the γ-Al_2O_3 to be fit for use as a catalyst support, it has to be reduced in size by milling. γ-Al_2O_3 is derived from the dehydration of Boehmite (γ-AlOOH) as one of the transition aluminas according

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to the sequence given in Eq. (1)(Liu and Zhang, 2005).

$$\text{Boehmite} \overset{450^{\circ}C}{\Longrightarrow} \gamma - Al_2O_3 \overset{750^{\circ}C}{\Longrightarrow} \delta - Al_2O_3 \overset{900^{\circ}C}{\Longrightarrow} \theta - Al_2O_3 \overset{1100^{\circ}C-1200^{\circ}C}{\Longrightarrow} \alpha - Al_2O_3 \quad (1)$$

From Boehmite, γ-Al_2O_3 can be produced at temperatures of between 450 °C and 750 °C. This is followed at higher calcination temperatures by a series of transformations to the δ and θ phases, whilst at temperatures between 1100 °C and 1200 °C α-Al_2O_3 is formed as the final thermodynamically stable phase with a structure based on a hexagonally close-packed oxygen sub-lattice structure

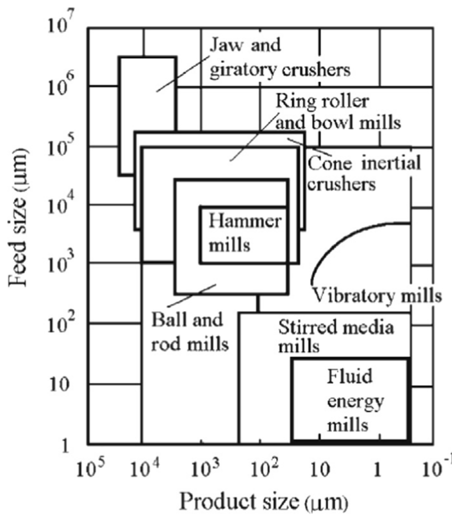

Fig. 1. Size reduction equipment available for different combinations feed size and product particle size (Neikov et al., 2009).

(Liu and Zhang, 2005). For maximum catalytic effectiveness, it is essential to maintain the desired physical and chemical properties of γ-Al_2O_3 in the milled product, e.g. high specific surface area and the absence of any phase changes (Trueba and Trasatti, 2005). Mechanochemical activation can cause microstructural changes to materials (Sopicka-Lizer, 2010). The planetary ball mill has been reported to induce mechanochemical phase transformations and reactions, the conditions of high stresses during milling are envisaged to play a major role in such phase transformations (Šepelák et al., 2007). Zielin´ski et al. (1993) reported on the phase transformation from γ to α Al_2O_3 by the use of this mill (Zielin´ski et al., 1993). Kostic et al. (2000) also reported on the phase transformation from γ to α by the use of a vibrating disc mill (Kostic et al., 2000). Additionally, evidence of phase transformation due to milling, similar to that achieved by thermal dehydration of boehmite, has been reported in various works (Duvel et al., 2011; Wang et al., 2005). However Bodaghi et al. (2008) observed no phase change in γ-Al_2O_3 after 30 h of milling in the Fritsch Pulverisette 7 planetary ball mill but reported the occurrence of phase change of γ-Al_2O_3 to α-Al_2O_3 only after the addition of α-Al_2O_3 seeds into the mill (Bodaghi et al., 2008). According to Bodaghi et al. (2008) the α-Al_2O_3 seeds act by reducing the transition temperature and activation energy for α-Al_2O_3 to nucleate (Bodaghi et al., 2008). It is therefore still necessary to carry out an in depth investigation into the effect of size reduction mechanisms brought about by different stress modes such as shear, impact, and compression on the surface and morphology or structure of the material. This is an essential starting point to understanding the initiation of the mechanochemical phase transformation that occurs in γ-Al_2O_3 during milling. The present work aims at exploring the size reduction of γ-Al_2O_3 using three mill types; single ball mill, air jet mill and planetary ball mill. In a similar manner to previous studies (Zhou and Snyder, 1991; Knozinger and

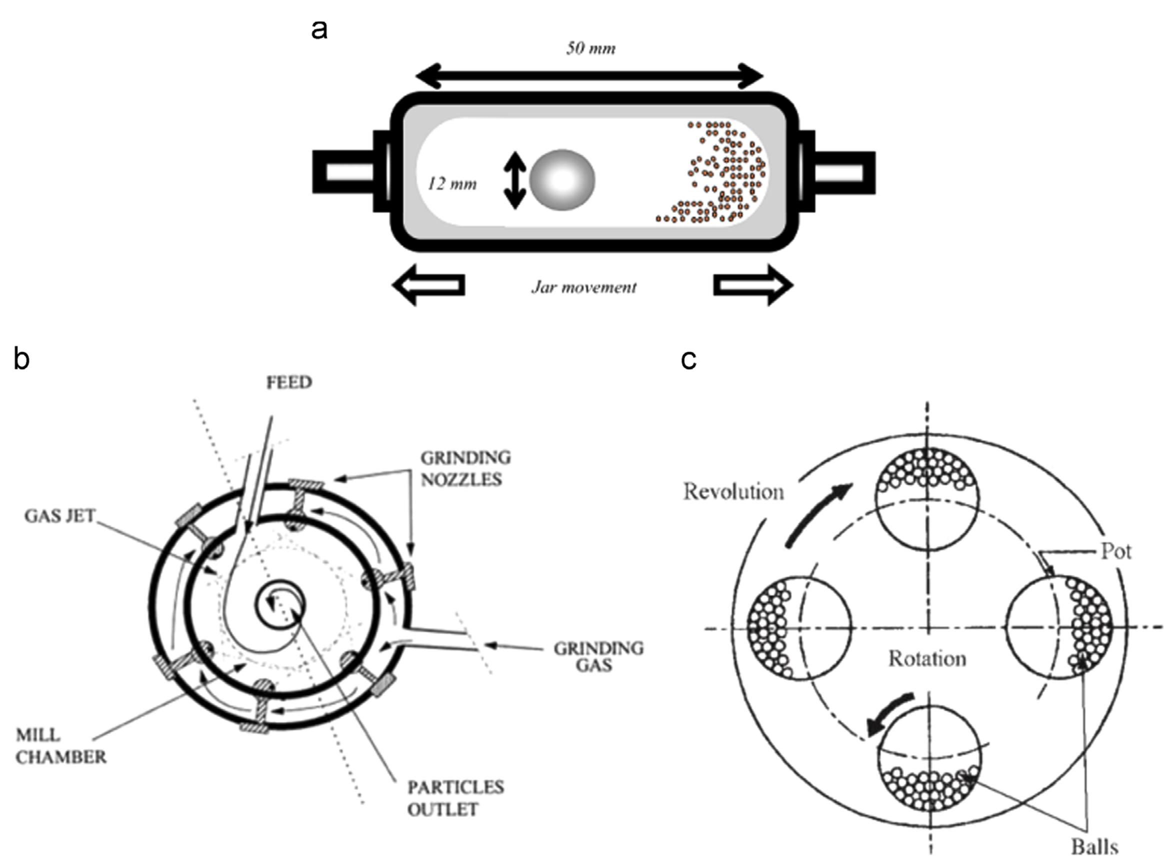

Fig. 2. Schematic diagram of milling chambers for the three mill types used showing (a) ball and powder motion in the single ball mill (Kwan et al., 2005) (b) movement of powder through milling chamber in the spiral jet mill (Kano et al., 2001), and (c) pot motion in the planetary ball mill (Neikov et al., 2009).