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Grinding to nano-sizes: Effect of media size and slurry viscosity

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1. Introduction

With the need to process low-grade finely dispersed ores the development of ultrafine milling processes has increased apace in recent decades (Lichter and Davey, 2002) and the rapid increase in power requirements and decrease in milling efficiency is well documented. Ball mills are being displaced by stirred media or centrifugal mills which use increasingly fine media and methods of increasing power input which are not limited by gravity considerations. For ultra-fine milling (e.g. down to product sizes less that $1-10 \,\mu$ m) these methods become essential, increasing grinding rate while at the same time exhibiting increased efficiency of power utilisation (Lichter and Davey, op. cit.).

Horizontal stirred mills such as the IsaMill have been manufactured with 3 MW power requirements, and vertical stirred mills such as the MetProTech and Deswik mills compete for market share. A lot of research has been done on the grinding kinetics in these mills. Yue and Klein (2004) and He and Forssberg (2006, 2007) studied the effect of slurry rheology on stirred milling kinetics with particle median sizes going down to about 4 μ m while Kwade (2010) has ventured down to a d50 of 0.6 μ m. Bilgili et al. (2004, 2006) have tested the population balance model down to 30 nm which is well within the so-called nano-size range (i.e. typically regarded as particle size less than 100 nm).

An exciting recent development is the Kelsey mill which uses centrifugal forces in a device which lends itself to continuous

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ABSTRACT

The growing trend towards ultrafine milling makes it worthwhile to study the effect of key variables on this process. The concept of the "grinding zone" is defined and analysed. It is found that (volume of grinding zone)/(volume of voids between media) is proportional to [(particle size)/(media size)]². The interaction between media travelling towards each other is analysed and the pressure developed between media is shown to be very high. The pressure acts on a small area of the media surface, so only affects media behaviour significantly when slurry viscosity is high. The analysis provides a basis for the development of a DEM contact model for interaction between media in a viscous slurry.

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operation and scale-up. Kelsey reported recently (2010) on his development of the device for producing slurry with 100% less than 10 μ m in a fully autogenous environment. This is a big step forward because generally mills using a centrifugal principle are difficult to operate continuously; and very fine media are difficult to separate from the product from the mill, so the opportunity to do without media is most welcome. A 1.5 MW Kelsey mill is in operation and larger mills are contemplated. It should be possible to exploit this approach for nano-milling.

Work done at Tata Consultancy Services (TCS) has as its objective the production of particles in the nano-size range. They have used a planetary mill for this purpose and have found an apparent grind limit at a d50 of 100 nm. Media size is an important variable but all four sizes tested (0.6, 1, 2 and 3 mm) appear to suffer this grind limit. Slurry viscosity is also a problem requiring operation at low percent solids and hence low capacity. It is of interest to examine media behaviour in the milling environment to establish why this is so.

2. Mill and media surface area considerations

Medium diameter used for nano-grinding is necessarily small in order to increase the number of impacts and the surface area for impact in the mill. Most of the surface area in the mill is thus the media surface area, A_{media} ; the mill surface area, A_{mill} , makes a small contribution as is shown below.

Consider a cylindrical mill of diameter *D*, length *L* filled to a level *J* (fraction of mill volume) with media of diameter *d*. If the voidage between the media in the load is ε then it can easily be shown that



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¹ The research for this work was done while Prof. Moys was on sabbatical with Tata from The School of Chemical and Metallurgical Engineering, University of the Witwatersrand, Johannesburg, South Africa.

$$A_{\text{mill}}/A_{\text{media}} = d(D + 2L)/(3DLJ(1 - \varepsilon))$$
(1)

which rapidly becomes small as *d* tends to small values. For example if for a typical lab planetary mill D = L = .1 m, J = .5 and $\varepsilon = .4$ then for d = .01 and .001 m the ratio is .33 and .033 respectively. In some cases media diameters as small as .00005 m are used! in which case the ratio is .0016. And remember that at any particular time less than half the mill internal area is in contact with the load.

3. Grinding zone volume

The grinding rate in a mill is necessarily limited by the volume of the grinding zones in the mill. What do we mean by "grinding zone"? Consider the interaction between two spherical media illustrated in Fig. 1.

The media are assumed to be a distance **a** apart (because of the presence of fine particles or a viscous slurry in the gap) and we assume that we are grinding particles which have a size ℓ_i . The grinding zone is then the circular bi-concave segment defined by $2\delta < \ell_i$. Particles outside this zone cannot be nipped by media and hence will not be ground. The volume of this segment can easily be calculated using elementary calculus:

Volume of grinding zone =
$$2\pi \left\{ rc^2 - 2[r^3 - (r^2 - c^2)^{3/2}]/3 + c^2 a/2 \right\}$$
(2)

Note that this volume is a strong function of the size of the particle being considered, since *c* is a strong function of $\delta = \ell_i/2$. Note also that as media separation **a** increases the volume of grinding zones for a particular particle size decreases because the radius of the grinding zone, *c*, decreases.

In order to calculate the maximum volume of grinding zones in the mill, we assume that each media particle has twelve nearest neighbours (upper limit for hexagonal close-packing (HCP); a lower number will apply in practice) and calculate the maximum total volume of grinding zones in the mill as a fraction of the void volume between the media. The data (for media separation a = 10 nm) is summarised in Fig. 2 which shows how the fractional grinding zone volume is related to media size and particle size. This is generally an overestimate given the HCP assumption above.

The data is very "well-behaved" and can be summarised using a very simple equation ($R^2 = 1.000$):

$$V_{\rm grinding\ zones}/V_{\rm voids} = 13.4(\ell_{\rm max}/d_{\rm media})^2$$

Note that when trying to grind 100 nm particles using 0.3–1.0 mm media the ratio is between approximately 10^{-6} and 10^{-7} ! If one starts with 10 µm particles then at *t* = 0 the ratio is



Fig. 2. Effect of media and particle size on the fractional volume of grinding zones in a mill (for media separation a = 10 nm). The points in the graphs are the points at which the pressure was calculated; they are not experimental points.

between .01 and .001 for these media diameters respectively. The volume of the grinding zones has decreased by a factor of 10^{-4} by the time the particles are smaller than 100 nm! We might expect that the grinding rate would decrease by a similar fraction. However this is not the case.

Note that the rate of grinding of particles of size ℓ_i in a grinding mill is $S_i m_i$ (kg/s) where S_i is the Selection function (s⁻¹) and m_i is the mass of particles in the mill. Typically for fine grinding $S_i = al_i^{\alpha}$ where α has a value between 0.8 and 1.5 (Austin et al., 1984). This implies that particles become easier to mill as they get smaller. If this were not true then the value of α would be 2 (assuming that grinding rate is proportional to the volume of the grinding zones).

4. Slurry viscosity effects

Viscosity of the slurry plays a major role in milling in general, but is particularly important in ultrafine grinding. In particular a viscous slurry will tend to reduce the relative velocity between media as they approach each other thereby reducing their momentum and hence their milling effectiveness. We therefore invested some time in exploring this effect.

Consider two media approaching each other at a relative velocity u_m , using Fig. 1 again for illustration. Assume that the space between them is filled with a viscous slurry. Since the slurry is incompressible it will be expelled from the grinding zone in a well-defined manner governed by $dV_{sl}/dt = 0$, where V_{sl} is the volume of slurry in the circular zone of diameter *c* in the figure. *c* will increase as the media move towards each other in a manner which



Fig. 1. Grinding zone for particles of size ℓ_i , between two media which are a distance *a* apart and are travelling towards each other at a velocity u_m .

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