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Online rheological monitoring of stirred media milling

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ABSTRACT

Authors had developed a tube rheometer to supplement a Netzsch MiniCer (Netzsch GmbH, Germany) stirred media mill in order to monitor online the rheological characteristics of the suspension and therefore the result of grinding during the process. It was demonstrated that the above measuring system was working appropriately based on the preliminary tests. The product related stress model was applied for the evaluation of the grinding results. It was found that the rheological behaviour of the suspension strongly affects the grinding result, i.e. specific surface area, median particle size as well as specific grinding energy. The tested model material limestone – water suspension behaved Newtonian initially, then after a given grinding time their properties had changed to Bingham plastic, occurred more or less at the same produced specific surface area at about 10 m^2/g for each tested concentration. A separate off-line validation test with a rotational rheometer had been also implemented to verify the Bingham approximation; correlation was very good.

1. Introduction

Stirred media milling now is a common practice to produce fine and ultrafine granular materials for different purposes. With an increasing demand of ultrafine powders for industries [1], wet and dry ultrafine comminution has been increasingly used in various fields, such as minerals [2,3], ceramic materials [4], pigments [5], chemical products [6], microorganisms [7] and pharmaceutics [8]. It is well known that suspension rheology affects the efficiency of wet comminution of materials [9]. The rheological behaviour of the suspension containing the ground particles might indicate the level of interparticle interactions or aggregation and therefore it is an important variable factor of the process control [10]. The influence of the rheological behaviour of mineral suspensions on their grindability has attracted more and more interests and attention, especially on ultrafine grinding performance [11,12]. Knieke et al. [13] carried out laboratory scale stirred media milling experiments with off-line rotational rheometer testing. Based on the experimental results and theoretical considerations Kniecke et al. [13] concluded that the primary reason of grinding efficiency decrease is the viscous dampening on the motion of the grinding media caused by the apparent viscosity increase of the ground suspension. “At high solids concentrations and/or small particle sizes, a drastic increase in suspension viscosity occurs, which leads to a dampening of the grinding media motion and to a reduction in the transferred stress energy. Hence, the rheological behaviour can limit the grinding process, and a viscous dampening-related grinding limit can be reached prior to the true grinding limit.” However, there are still many issues concerning the machine and the process for the different applications. When grinding is effective and the particle size range decreases significantly, the circulating suspension (in closed circuit) is becoming typically non-Newtonian. The changed rheological behaviour and the increasing rheological material parameters can negatively affect the grinding efficiency; therefore, it is an important issue.

Concerning rheology, the three measuring pipes tube rheometer was developed decades ago [14,15]. So far, many different solid materials from all over the world had been tested in this rheometer [14]. Main aim of these tests was to gain basic data for large scale hydraulic transport applications. The goal of the research presented in this paper is to develop an online tube rheometer for the Netzsch MiniCer stirred media mill and to carry out grinding and rheological testing simultaneously followed by the investigation of the correlation of the results as well as to validate the developed system.

2. Theory

2.1. Tube rheometer

Grinding specialists are very well aware of suspension rheology and their change during milling because the increasing viscous resistance can seriously inhibit the motion of the grinding media and therefore grinding efficiency decreases. However, the scientific field of fluid dynamics of multi-phase media is far from their everyday routine, therefore related fundamentals of suspension rheology are described in detail in the following. The viscous flow of single phase fluids (liquids and gases) is really well described in the literature [16]. The rheological

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behaviour of a fluid can be described by the constitutive equation. Generally, the constitutive (shear) equation is the relationship between the stress tensor and the rate of deformation tensor. One dimensional constitutive equations (shear stress τ as function of the shear rate γ) are commonly used in the engineering practice. Viscous rheology can be characterised as time dependent or time independent and as Newtonian or non-Newtonian. Non-Newtonian rheology cannot be described by the well know Newtonian linear constitutive equation. Test data shown later imply that the Bingham plastic rheological model should be described in detail.

The most appropriate equipment for measuring the rheology of fine suspensions (1–100 μm typical particle size range) is the tube rheometer. The principal of the measurement is that in straight horizontal pipes, stable laminar flow has to be established and the pressure loss as a function of the flow rate has to be measured [14]. The laminar flow of Newtonian incompressible fluids in straight horizontal pipes is really well-described in the literature [16]. According to the well-known Hagen – Poiseuille model, the velocity profile is parabolic and the shear stress profile is linear because shear stress is proportional with the first derivative of the velocity (Fig. 1). The Hagen – Poiseuille equation for laminar Newtonian flow can be written in the following form:

$$\frac{\Delta p \cdot D}{4 \cdot L} = \mu \cdot \frac{8 \cdot v}{D} \quad \tau_w = \frac{\Delta p \cdot D}{4 \cdot L} \quad \gamma_w = \frac{8 \cdot v}{D} \tag{1}$$

In every viscometer or rheometer there is a defined surface and the instrument can indirectly measure the speed difference of the neighbouring layers (shear rate) and the shear stress between them. With some instruments, only the relative change of the viscous resistance compared to a reference fluid can be measured. These instruments should be called viscometers, because only Newtonian fluids can be tested with them. If the constitute equation can be measured by an instrument they should be called rheometers, because with them non-Newtonian fluids can be tested as well. In the case of tube rheometers, the sheared layer is the cylindrical shape layer of fluid molecules just right at the inner pipe wall. In the case of rotational rheometers with cylinder – cylinder geometry this layer is the cylindrical shape layer of fluid molecules just right at the wall of the revolving bob. The so called wall shear stress (τ_w) is determined from the measured pressure loss and the wall shear rate (γ_w) is determined from the measured cross sectional average velocity (Eq. (1)) for tube rheometers. The wall shear rate is the tangent of the velocity profile at the pipe wall (Fig. 1).

If a non-Newtonian fluid is pumped in the tube rheometer the velocity profile might be different from the one described by Hagen – Poiseuille, therefore the $8 \cdot v/D$ term might not be equal to the tangent of the velocity profile at the wall. Therefore, the $8 \cdot v/D$ term is called as pseudo shear rate, the $\Delta p \cdot D/4 \cdot L$ term is called as pseudo shear stress and the function is called as pseudo shear curve (Fig. 5b). If this function of the measured laminar pipe flow data is a straight line from the origin, then the fluid is Newtonian and the absolute viscosity is the tangent of the angle of the line. If the pseudo shear curve is not a straight line from the origin then the tested fluid is non-Newtonian. If the pseudo shear curve is a straight line but the starting point is not the origin the tested fluid is Bingham plastic. The evaluation of non-Newtonian tube rheometer test data is rather difficult, in every case the rheological model must be known or be estimated and then the evaluation protocol of the given non-Newtonian model can be applied. In the following let us examine the pipe flow of Bingham plastic fluids (Table 1).

The Buckingham equation correlating the pressure drop of laminar flows of Bingham plastic fluids was deduced from the Bingham constitutive equation (Eq. (3)) for a pipe flow [16,17].

$$\tau_w = \eta \cdot \gamma_w \cdot \left[1 - \frac{4\tau_0}{3\tau_w} + \frac{1}{3}\left(\frac{\tau_0}{\tau_w}\right)^4\right]^{-1} \quad \tau_w = \frac{\Delta p \cdot D}{4 \cdot L} \quad \gamma_w = \frac{8 \cdot v}{D} \tag{5}$$

The Buckingham equation can be expressed in terms of the so called Bingham plastic Reynolds number and the Hedström number. The Fanning friction factor (f) can be calculated by iteration, and then the pressure loss can be calculated by the well-known Darcy and Weisbach Eq. [18].

$$\frac{1}{Re_B} = \frac{f}{16} - \frac{He}{6 \cdot Re_B^2} + \frac{He^4}{3 \cdot f^3 \cdot Re_B^8} \quad Re_B = \frac{v \cdot D \cdot \rho}{\eta} \quad He = \frac{D^2 \tau_0 \rho}{\eta^2} \tag{6}$$

Re_B is not a real Reynolds number because η in the denominator does not describe fully the shearing force for Bingham plastic fluids and therefore another dimensionless parameter (He) is needed. However, these terms make the Buckingham equation to be easier solvable. In case of turbulent flows of Bingham plastic fluids in smooth pipes the Torrance equation is an alternative [16].

$$\frac{1}{\sqrt{f}} = 4.53 \cdot \log(1 - x_0) + 4.53 \cdot \log\left(Re_B \cdot \sqrt{f}\right) - 2.3 \quad \text{where,} \quad x_0 = \frac{\tau_0}{\tau_w} \tag{7}$$

The laminar-turbulent transition occurs at the critical value of the Bingham plastic Reynolds number and it can be calculated from the Hanks Eq. [16].

$$(Re_B)_C = \frac{He}{8x_{0C}}\left(1 - \frac{4}{3}x_{0C} + \frac{1}{3}x_{0C}^4\right) \quad \text{where,} \quad \frac{x_{0C}}{(1 - x_{0C})^3} = \frac{He}{16800} \tag{8}$$

2.2. Rheological measurements during grinding in stirred media mill

In the literature there are many paper dealing with the rheological testing of suspensions carrying ground particulate materials [14,19]. Common feature of these rheological testing methods is that they are typically off-line measurements. During an off-line measurement, some samples are taken from the ground material and tested

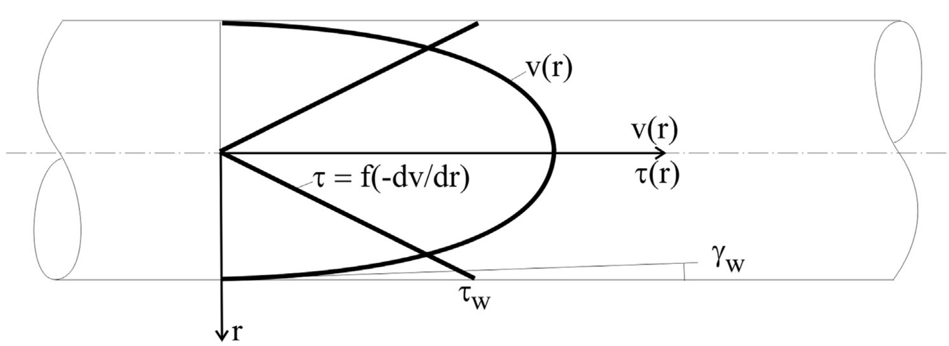

Fig. 1. Velocity and shear stress profiles of laminar Newtonian pipe flow.