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Measuring the strength of irregularly-shaped fine particles in a microcompression tester

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ABSTRACT

Characterizing the response of individual particles to stresses is of great relevance in a number of fields. In the field of grinding, important advances in mill modeling by decoupling material from machine contributions to the outcome of the process have been made in recent years that require direct information on the distribution of strengths of particles being ground. Although a number of methods and experimental devices have been proposed to measure the breakage strength of individual particles, only recently alternatives have become commercially available for testing particles in the fine size range. The paper demonstrates that a micro compression testing machine allows measuring the distribution of strengths and fracture energies of non-spherical fine particles, although great care should be taken while doing the measurements. It then shows that the mean strength of particles contained in a narrow size range is closely related to the rate of breakage of particles in the same size range in a planetary ball mill, thus demonstrating the validity of the measurements.

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1. Introduction

Characterizing the response of individual particles to stresses is of great relevance in a number of fields. In the case of chemical and pharmaceutical powders, as well as lump ores and pellets it is of relevance to understand their likelihood to lose physical integrity or fines due to abrasion during pneumatic or mechanical handling and transport (Yan et al., 2009; Nguyen et al., 2009; Rahmanian and Ghadiri, 2013). In the case of aggregates and cement constituents it is of interest to understand their behavior in pavements, mortars and concrete (McDowell and Bolton, 1998), whereas for a number of materials, including ores and cement clinker, it is of relevance to understand the material contribution to the effort required in mechanical size reduction (Schönert, 1991; Tavares, 2007).

In the field of grinding, important advances in mill modeling by decoupling material from machine contributions to the outcome of the process have been made in recent years (Tavares and Carvalho, 2009; Crespo, 2011; Breitung-Faes and Kwade, 2013) that require direct information on the distribution of strengths of particles being ground to predict the performance of different mill types.

A number of methods and experimental devices have been proposed to measure the breakage strength of individual particles, varying in size range that can be treated and in the rate of application of stresses (Steier and Schönert, 1972; Yashima et al., 1979; Schönert, 1991; Tavares and King, 1998; Unland and Szczelina, 2004; Antonyuk et al., 2005). In the context of comminution, probably the most challenging and important within these is testing of fine and ultrafine particles, since these are the sizes in which energy consumption in size reduction is the highest. In the past, the breakage strength of particles contained in these size ranges could only be measured using special custom-made presses (Steier and Schönert, 1972; Yashima et al., 1979; Sikong et al., 1990). More recently, with the development of a number of micro compression testing machines it became possible to determine the strength of nearly-spherical individual particles loaded under slow compression (Antonyuk et al., 2005; Nguyen et al., 2009; Yan et al., 2009).

The present work demonstrates that a commercial micro compression testing machine allows measuring the distribution of strengths and fracture energies of non-spherical fine particles. It then shows that the mean strength of particles contained in a given size range is closely related to the rate of breakage of particles in that same size range in a planetary ball mill, thus demonstrating the validity of the measurements.

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2. Experimental

2.1. Materials

Six materials were chosen for the investigation, given their widely different mechanical properties: silicon carbide, quartz, blast-furnace slag, limestone, coal shale and rice husk ash. Their chemical compositions was measured using an X-ray fluorescence spectrometer (EDX-720 from Shimadzu) and results are presented in Table 1. The specific gravity of the samples was determined using a Helium pycnometer (AccuPyc 1340 from Micromeritics). Indentation hardness and Youngs modulus measurements were carried out using an Ultra-micro Hardness Tester (DUH-21 from Shimadzu) using a Vickers indenter, at a loading rate of 44 mN/s, holding at the maximum load for 5 s. A summary of the results is presented in Table 2.

Samples were previously ground dry in a tumbling ball mill and wet-sieved in the 45 × 37 μm size range for the tests, having a maximum allowance of 5% of out-of-size material, which corresponded in its majority to the material passing 37 μm. After sieving, samples were oven-dried for testing.

The fracture strength of particles contained in the 37–45 μm was measured using a Micro Compression Testing Machine (MCT-W Series 500 from Shimadzu). This high-resolution press uses a 50 μm-diameter flat-ended indenter that is capable of applying loads that range from as low as 10 mN to about 5 N, measuring displacements that range from 100 μm to 10 μm with 0.01 μm and 0.001 μm of resolution, respectively. The device is equipped with a camera installed sideways, which allows observing the specimen status during the entire compression event.

The procedure used in single-particle testing consisted of first dispersing a small amount of material in ethyl alcohol and letting a drop of the suspension fall on the lower compression plate, which is fixed to the equipment table. After evaporation, particles were appropriately dispersed, allowing the selection of individual ones for testing. Particle size (average of length and width) was measured using the specimen dimension measurement function which is used at 50 times magnification. From these two measurements, the aspect ratio of each material was estimated. The rate used in compression of the particles was 8.3 mN/s for quartz, rice husk ash, silicon carbide and blast-furnace slag. A lower rate of 1.5 mN/s was required for coal and limestone, given their lower strength. The machine applies loads at a constant rate using an electromagnetic system, in which the force is proportional to the intensity of the electric current, whereas deformations are measured. Fracture of the particle under loading is identified as a plateau in the force–displacement profile (Fig. 1), which results from a rapid increase in displacement at a constant load. From this load the particle compressive strength σ_p is calculated by (Hiramatsu and Oka, 1966; Tavares, 2007):

$$\sigma_p = \frac{2.8F_c}{\pi d^2} \tag{1}$$

where d is the particle size and F_c is the load at failure. For each material 50 particles contained in each size range were tested.

The mass-specific particle fracture energy E_m was calculated by integrating the force–deformation response

$$E_m = \frac{1}{m_p}\int_0^{\varepsilon_c} F\,d\varepsilon \tag{2}$$

where ε_c is the deformation at failure, m_p is the particle mass, estimated using $\beta\rho d^3$, so that ρ is the specific gravity of the material and β the shape factor. In the present work, given the difficulty of measuring shape factors of particles in such fine ranges, it was assumed that their value was equal to 0.43, which is equivalent to the average value found for a number of materials (Tavares and King, 1998).

Table 1
Chemical composition of the materials.

Oxides	Blast furnace slag	Coal shale	Limestone	Quartz	Rice husk ash	Silicon carbide
SiO_2	18.79	47.68	0.55	99.27	77.96	96.45
Al_2O_3	9.29	26.77	0.25	0.13	–	2.19
SO_3	1.73	4.94	–	–	1.97	1.03
CaO	27.64	0.67	55.43	0.03	0.91	–
K_2O	0.28	1.49	0.05	–	1.66	–
SrO	0.11	0.03	–	–	–	–
P_2O	–	–	–	–	1.23	–
P_2O_5	–	0.43	–	–	–	–
MnO	0.51	–	–	–	0.29	–
Fe_2O_3	0.29	1.08	0.25	0.07	0.15	–
MgO	2.60	–	0.29	–	–	–
CuO	–	0.02	–		–	–
Rb_2O	–	0.02	–	–	–	–
TiO_2	0.33	1.28	–	0.01	–	–
BaO	0.10	0.25	–	–	–	–
ZrO_2	–	0.05	–	–	–	–
L.O.I.[a]	38.31	15.28	42.8	0.26	15.80	0.30

[a] Loss on ignition.

Table 2
Summary of data from additional characterization testing (mean values, with standard deviations from 10 measurements in parentheses).

Material	Specific gravity (g/cm^3)	Young's modulus (GPa)		Vickers hardness (GPa)	
Blast furnace slag	2.04	37.7	(16.2)	539.8	(154.3)
Coal shale	2.28	16.4	(5.2)	70.9	(31.4)
Limestone	2.67	12.2	(3.1)	117.9	(45.1)
Quartz	2.67	64.1	(5.7)	1306.2	(138.5)
Rice husk ash	2.29	8.3	(1.6)	22.4	(3.5)
Silicon carbide	3.21	210.3	(48.8)	3283.3	(649.7)

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