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Compression testing spherical particles for strength: Theory of the meridian crack test and implementation for microscopic fused quartz

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ABSTRACT

We show that uniaxial compression testing of spherical particles can give unambiguous access to their tensile strength as governed by surface flaws if one uses pairs of elasto-plastic platens, tailoring their hardness in order to control the relative area of particle-to-platen contact during the test. This eliminates the development of contact microcracks that are typically found to govern particle fracture when hard platens are used. We show that, if the platen materials are well chosen, one can probe a range of stress states for which it is known that particle failure was initiated along the surface, under elevated hoop stress within a region situated remote from the points of load application. Specifically, platens must be chosen such that particles tend to fracture when the ratio of projected contact area radius to particle radius exceeds a specific value that depends on the Poisson ratio of the particles. With fused quartz of Poisson ratio 0.17, this specific ratio value equals 0.65. We demonstrate the approach using microscopic fused quartz spheres 40 ± 20 µm in diameter as a testbench material; with those particles hardened steel serves as an appropriate platen material. Their strength values are statistically distributed; this is addressed using several platen materials. The resulting bank of data is interpreted using established survival-analysis methods, namely the non-parametric product-limit estimator. We also give a maximum likelihood estimation of the particle strength Weibull distribution parameters derived from the ensemble of data after left-truncation and/or right-censoring of data points situated inside of the range of unambiguous surface fracture strength measurement for each platen material. This gives a Weibull modulus of 6.3 and characteristic strength of 890 MPa for the fused quartz particles. These values are significantly lower than what is produced in high-strength fused quartz fibers of comparable diameter; the difference is most likely a result of surface damage caused during powder storage and manipulation in the absence of a protective coating.

1. Introduction

The strength of brittle particles is important in many different fields of science or engineering. The strength of rock and mineral particles dictates the energy consumed in mining processes (Broch and Franklin, 1972). Particle strength also governs the mechanical behavior of many soils (Brzesowsky et al., 2011; McDowell and Bolton, 1998; Nakata et al., 1999) and is an important

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parameter in processes of powder granulation and compaction, which are present across a wide range of industries (including for example food and pharmaceutical industries) (Antonyuk et al., 2010, 2005; Khanal et al., 2008; Yap et al., 2008, 2006). In materials processing the strength of particles governs grinding (Lobo-Guerrero and Vallejo, 2006) and comminution processes and also the processing of ceramics (Tavares, 2007). In addition, the mechanical behavior of a wide range of composite materials, alloys or concrete, is governed by the strength of the microscopic particulate phases that they contain (Gammage et al., 2004; Hauert et al., 2009; Khanal et al., 2008; Miserez and Mortensen, 2004).

There is at present no well-established method to measure the strength of individual particles. Cutting out beam-like test specimens from the particle is generally impractical and might also lead to irrelevant result, since practically any cutting or machining procedure may introduce extraneous flaws (Kiener et al., 2007; Rice, 1979; Shim et al., 2009). Rather, the most common method used to measure the strength of individual particles is to compress the whole particle until it breaks. Two methods are generally used to this end. One is to impact accelerated particles against a hard substrate and record the velocity at which the particles break (Chau et al., 2000; Dean et al., 1952). The other is the quasi-static, uniaxial compression test, in which a particle is compressed between two parallel platens until it fails, recording the load and platen displacement during the test. The reason why both tests can generate meaningful strength measurements in brittle particles is that the stress distributions that they produce within the particle are so inhomogeneous that tension is often produced somewhere within the compressed particle.

For tractability, equiaxed particles are often assimilated to spheres in compression test data analysis. The solution for the stress field that is created within a sphere when it is compressed uniaxially across two points situated at either end of a diameter is well known; a frequently used treatment is that of Hiramatsu and Oka (Hiramatsu and Oka, 1967, 1966). These authors analyzed the stress within an isotropic linear elastic sphere of radius R compressed by a pair of uniformly distributed radial loads acting symmetrically over two equal spherical caps centered along the compression axis and of outer circle defining the *contact radius*, a. For (a/R) =0.04 to 0.13, Hiramatsu and Oka showed that the peak tensile stress is located along the compression axis, and is roughly equal to ~0.7 times the *nominal stress*, defined as $F/\pi R^2$, where F is the load acting on each spherical cap. This conclusion has been debated, notably because, depending on the precise value of a/R, the peak value of the tensile stress along the compression axis can deviate significantly from 0.7 (Darvell, 1990; Hiramatsu and Oka, 1967; Salençon, 1966; Wijk, 1978).

Following Hiramatsu and Oka's analysis, the "tensile strength" of particles tested in uniaxial compression is therefore often computed as:

$$\sigma_T = \kappa \frac{F_f}{\pi R^2}, \tag{1}$$

where F_f is the peak (failure) load and κ is a constant near unity. Experimental data of Jaeger (Jaeger, 1967) and analysis by several authors suggest that $0.7 \le \kappa \le 1.4$. Many experimental studies have used this expression to evaluate the strength of spherical or irregular particles tested in uniaxial compression (McDowell and Amon, 2000; McDowell and Bolton, 1998; Nakata et al., 1999; Ogiso et al., 2007; Pitchumani et al., 2004; Portnikov et al., 2013; Ribas et al., 2014; Rozenblat et al., 2011; Verrall, 1976; Yap et al., 2008, 2006; Yoshida et al., 2005; Zhao et al., 2013).

In nearly all uniaxial particle compression tests to date, platens are used that are much harder than the particle, such that $(a/R) \le 0.1$. This is convenient from a practical point of view, because the particle will then not damage the platens. Also, Hertzian contact theory can then be used to calculate a/R (Huang et al., 2014) and the stress state is relatively well defined, with its peak tensile value given by Eq. (1) and reached along the particle diameter parallel to the compression axis, generally near the load application points and with $\kappa \ge 1$. There are, though, disadvantages to the use of hard platens. First, when a particle is compressed between two platens much harder than itself, the high stress concentration that develops at and near the small area of contact between the platens and the particle can cause the nucleation of extraneous cracks that may then govern the measured particle fracture stress, obscuring the detection of intrinsic particle flaws (Khanal et al., 2008; Majzoub and Chaudhri, 2000; Schönert, 2004; Swab et al., 2011). Moreover, in many strong brittle particles, the largest defects are located not within the particle, but along its surface (Lawn, 1993), which is at best poorly sampled when the site of peak tensile stress is located, not at the surface, but deep within the particle.

Those limitations, and a solution thereto, were identified by Shipway and Hutchings in a 1993 contribution that we consider to be a significant, but so far underexploited, advance in the state of the art of particle testing (Shipway and Hutchings, 1993a, 1993b). These authors reanalyzed the particle stress field solution for the problem of compression of a linearly elastic sphere compressed by symmetric pressure uniformly distributed over the surface of the spherical caps at either end of a diameter. They pointed out that there can be a significant tensile hoop stress, σ_ϕ acting along the equatorial belt of the particle surface, due to which the particle could fail. Over the range $0 \le (a/R) \le 0.8$ this tensile hoop stress remains roughly equal to

$$\sigma_\phi \approx 0.4 \frac{F}{\pi R^2}. \tag{2}$$

In addition, Shipway and Hutchings showed that as the ratio a/R increases, i.e., if the particle is allowed to sink more deeply into the platen material, then the location of the peak tensile stress shifts from the compression axis to the equatorial belt region. For a material with Poisson's ratio ν=0.25, this transition happens when $(a/R) \approx 0.6$. Past that point, the hoop tensile stress σ_ϕ in the equatorial belt exceeds the tensile stress anywhere else within the spherical particle.

Shipway and Hutchings also put their conclusions to practice, testing spheres, roughly 700–800 μm in diameter, of lead glass or sapphire using a variety of platen materials. They showed that more consistent particle strength values are obtained using Eq. (2) as compared to Eq. (1), implying that fracture was more likely initiated from the particle surface than from its interior.

At the microscopic and nano- scales quasi-static uniaxial compression tests become even more challenging. There are only a few