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Invited review paper

A review of models for single particle compression and their application to silica microspheres

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ABSTRACT

We report on the deformation behavior of single silica microspheres. For the first time a detailed discussion on the quantitative evaluation of force–deformation data in the elastic and plastic regime is given. The microspheres are compressed between two flat plates of a custom built manipulation device supported by a scanning electron microscope. The device allows a high sample throughput enabling full statistical evaluation of force–deformation data of single microspheres. Existing theories describing the deformation behavior of single spheres are discussed and applied to the elastic and plastic deformation regime of silica microspheres. The results obtained from the theories are compared between each other and values reported in literature. The silica microspheres exhibit a significantly different deformation behavior than expected from bulk fused silica, i.e. a distinct plastic deformation behavior. Furthermore, a significant decrease in Young's modulus and hardness was observed caused by high porosity and reduced crosslinking of silicon atoms, respectively, that is inherent to silica produced by Stöber synthesis. However, the calculated contact pressures exceed the compressive strength of bulk fused silica by at least a factor of two as an effect of a reduced amount of material flaws in microstructures.

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1. Introduction

One of the grand challenges in particle technology is the development of physical models for systems where particles are in contact. An important precondition in designing, modeling and understanding of particulate processes is the availability of predictive models of individual unit operations and whole systems comprising several unit operations. The challenge is to predict energy consumption, throughput and the dynamic evolution of particle properties in dependence of operational parameters. Great progress in all fields of particle technology was made in the last decades. Nevertheless, we have to admit that the systematic determination of particle properties beyond particle size is still in its infancy. Information on surface, composition and mechanical properties of complex particles is very limited.

Unit operations of powder processing, e.g. distinct types of mills, tableting machines or conveying systems, provide a particular stress type, stress distribution and stress number to the particles. The particles can be stressed by impact, compression and shear or combinations of these. The operation function can be defined as the functional dependency between the operational parameters and the resulting stresses applied to the particles. In principle, this complex function can be determined by a proper modeling of the underlying two-phase flow, e.g. by means of CFD and particle trajectory simulation or by DEM modeling. However, for many unit operations this function is still unknown. The particles react to the stress conditions provided by the operation function according to their material properties. This is called the material function. The material function depends on the particle size, shape and on the intrinsic material properties such as the complex modulus due elastic and inelastic deformation. Systematic approaches where operation and material functions are clearly separated are rare (see e.g. Toneva et al. [1,2], Vogel and Peukert [3]). In general, mechanical particle properties are related to normal and shear stressing conditions. In this contribution we focus on the compression of spherical particles under small normal compression rates. Rate-dependent processes as well as shearing, translational and rotational friction are not covered.

Powders of micron sized particles are applied in various technical processes such as powder tableting, powder flow, fluidization, comminution, additive manufacturing or ceramics processing. In all these fields the mechanical properties of the single particles have a significant effect on the macroscopic properties of the powder and on the final product properties. However, despite their fundamental importance the mechanical properties of single micron sized particles are mostly unknown. In particular, distributions of mechanical particle properties have never been measured and discussed. The experimental challenges to characterize the mechanical particle properties are: localizing the position of individual particles, the well-defined stressing of small objects, measuring small forces ($<100\ \mu N$) and deformations of a few nanometers. The last two requirements are fulfilled by nanoindentation devices that are commonly used for testing the mechanical behavior of bulk materials. During nanoindentation experiments a rigid probe is pressed in a deformable flat surface while the applied load and the depth of indentation are observed. Nanoindenters are usually unable to test single particles systematically due to the lack of an imaging technique enabling the localization of single particles. For this purpose we developed a manipulation device for in situ compression of single micron sized particles supported by a scanning electron microscope [4]. The device enables not only the localization of single particles but also the observation of the loading process and the imaging of the deformed particles after compression providing additional information and thus facilitating the interpretation of the recorded force–deformation data.

Another challenge is the evaluation of the recorded force–deformation data. Nanoindentation has been extensively studied since it forms the basis of hardness testing on the nanoscale where the model of Oliver and Pharr is widely applied for evaluation [5]. However, compression of single particles is a quite different case: particles tend much more to expand perpendicular to an applied load than bulk materials for simple geometrical reasons [6]. Furthermore, boundary effects caused by the finite volume of the particles as well as their geometrical shape influence the deformation behavior significantly [7,8]. Because of its high symmetry a sphere is the simplest geometry to deal with. Several theories to model the deformation behavior of spheres are known yielding partially quite different results. It is an important question which theory is the most accurate. Especially in the case of microspheres it is unknown, weather these theories are directly applicable.

While much experimental research has been conducted in the field of nanoindentation of bulk material there has been comparatively little on micron sized structures, especially single particles because of the aforementioned experimental difficulties [4,9–26]. The conducted studies were predominantly of qualitative nature or focused on individual material properties. No statistically relevant data set was presented so far due to the small number of tested particles.

The objective of this paper is to review the existing models for quasi-static elastic and plastic deformation of particles under compression. These models provide material properties like Young's modulus, hardness and yield strength which all have been extracted from our measured force–deformation data. These data are most relevant for studies on adhesion (e.g. Zhou and Peukert [27]), fracture (Toneva and Peukert [28]) and DEM modeling of particulate systems. Note that these models are generally valid in case of hard ductile materials. In case of soft, brittle and viscous materials or very adhesive contacts the validity of the models might be limited and the reader is referred to additional literature of Lui [29], Hiramatsu and Oka [30] and Tomas [31,32].

The models were applied to the experimental data of compressed single silica microspheres with a radius of 418 nm and 261 nm. Silica microspheres were chosen as they are considered as an ideal model system because of their amorphous structure, eliminating any isotropic effects of the mechanical properties and their perfectly spherical shape. The spheres were compressed between two flat plates using the new custom built manipulation device. It is capable of measuring with a comparatively high throughput so that a total