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Original Research Paper

Contact properties determination of macroscopic fine disperse glass particles via compression tests in normal direction



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ABSTRACT

The paper describes the deformation behavior of spherical, dry and non-porous particles during a single particle compression test in normal direction. Therefore a compression tester was built. Industrial used soda lime glass particles with two macroscopic fine disperse sizes $(d_{1,50,3} = 284.30 \,\mu\text{m}$ and $d_{2,50,3} = 513.20 \,\mu\text{m})$ were applied as model material to investigate the micromechanical contact behavior. In order to influence the elastic-plastic contact properties of particles, the surfaces were altered with chemical modification by means of silanization.

The determination of various micromechanical contact properties (e.g. adhesion force, modulus of elasticity and contact stiffness) was carried out model-based with the contact model 'stiff particles with soft contacts' by means of a back-calculation.

It could be shown that the model-based determination of material properties was a good alternative compared to the comprehensive tensile tests and pull-off force measurements.

In addition to the gained normal force-displacement data in normal direction, the friction limits for tangential loading and rolling with the load-dependent adhesion force were model-based determined. © 2016 The Society of Powder Technology Japan. Published by Elsevier B.V. and The Society of Powder

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1. Introduction

The elastic, elastic-plastic and plastic contact behavior of bulk solids and single particles were investigated theoretically and experimentally during the last decades. The detailed understanding of the compression, flow and handling behavior of cohesive powders and the interaction between the particles is still of special interest in many industrial branches, such as in pharmaceutics, chemical industry, agricultural or food technology [1,2]. For instance spherical glass particles are used in the production of light reflecting road signs and lane markings [3,4]. The interparticular adhesion forces between the particles depend on the particle sizes. On the one hand the adhesion between small particles increases because of an increasing ratio between adhesion force and weight force. The attractive adhesion force exceeds the weight force by several orders of magnitude. On the other hand the adhesion force increases with an increasing contact radius. This result leads to challenging handling problems. Especially for fine to ultrafine particle sizes, the van der Waals attraction forces exceed the weight

¹ Date of death: 24.11.2015.

forces. This leads to undesired (silo discharge or apparatus feed problems) or desired (tableting of cohesive powders to improve their handling) adhesion depending on the process [5].

In the recent years, most of the scientific investigations in the field of powder technology deal with the compression behavior of granules [6–8] or the compressive stress of single particle until breakage [6,9–12]. Thereby, a wide variety of contact models are used. Depending on the particle collectives, which have to be examined, an elastic [13], elastic-plastic [14–16] or plastic [17] contact model has to be chosen.

In the present work, contrary to this scientific papers, the elastic-plastic contact of a single particle to a wall in normal direction (up to a maximum normal force $F_{N,\text{max}}$) with a custom inhouse manufactured compression tester is studied. Thus, not only the micromechanical contact properties, but also values like the residual plastic deformation are determined for the particles [5].

In industry, not only the loading types play a decisive role, such as the compressive stress during the tableting process, but also the handling of single particles, for example the encapsulating of nanoand micro particles in tableting machines. Therefore, flow additives are applied to improve the flowability of bulk solids [18,19]. This means that nano particles are accumulated on the primary particle surfaces. To change the flowability of particles, it is also possible to

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Nomencl	ature
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a_0	minimum molecular surface distance (nm)	$h_{K,total}$	displacement of both particle-wall contacts (µm)
a_1	slope of linearized elastic normal force-displacement	$h_{K,Y}$	displacement at yield point (µm)
	function (N·mm ^{-1})	k _{N,Abl}	contact stiffness at detachment ($N \cdot mm^{-1}$)
a_2	slope of elastic-plastic yield limit (N·mm ⁻¹)	k _{N.el}	elastic contact stiffness (N·mm $^{-1}$)
<i>b</i> ₂	Y-intercept of elastic-plastic yield limit (N)	$k_{N,el-pl}$	elastic-plastic contact stiffness (N·mm ⁻¹)
$C_{H,sls}$	effective Hamaker constant (J)	k _{N,Entl}	contact stiffness for unloading (N·mm ⁻¹)
d_{50}	mean particle diameter (µm)	$M_{R,C,H}$	friction limit of the rolling moment (N·m)
Ε	modulus of elasticity (GPa)	р	pressure (p)
E^{*}	effective modulus of elasticity (GPa)	p_f	characteristic micro-yield strength (MPa)
F_G	weight force (N)	$r_{1,2}$	effective particle radius (µm)
F_H	adhesion force (N)	r_K	contact radius (µm)
F _{H0}	characteristic adhesion force of the unconsolidated con-	$r_{K,el}$	elastic contact radius (µm)
	tact (N)	δ	tangential displacement (μm)
F_N	normal force (N)	$\delta_{C,H}$	limit of tangential displacement (µm)
$F_{N,\max}$	maximum normal force (N)	κ	elastic-plastic contact consolidation coefficient (-)
$F_{N,Y}$	normal force at yield point (N)	κ_A	elastic-plastic contact surface ratio (–)
$F_{R,C,H}$	critical load dependent rolling resistance (N)	κ_p	plastic repulsion coefficient (-)
F_T	tangential force (N)	μ_i	internal friction coefficient (–)
$F_{T,C,H}$	Coulomb friction limit (N)	μ_R	coefficient of rolling friction (-)
G	shear modulus (GPa)	v	Poisson ratio (–)
G*	effective shear modulus (GPa)	$\sigma_{ m sls}$	effective surface energy (mJ·m ⁻²)
h_K	displacement (µm)		
$h_{K,\max}$	maximum displacement (μm)		
$h_{K, pl}$	residual plastic contact deformation (µm)		

modify the particle surfaces chemically. In the present work, the particles were chemically surface modified through the method of silanization [20,21]. This kind of functionalization has a fundamental impact on the micromechanical contact properties of the treated cohesive powders [21,22].

The goals of this study were to answer two questions: What is the role of contact stiffness and effective particle radius with regard to the load-dependent adhesion force before loading and during contact flattening? Is it possible to determine the micromechanical contact properties of macroscopic fine disperse particles with the used contact model?

2. Materials and methods

In this section, the used soda lime glass particles and the surface functionalization will be discussed (2.1). Subsequently, a description of the in-house manufactured compression tester is shown (see Section 2.2). Following this, the contact model used for the model-based back-calculation of the micromechanical contact properties is annotated (2.3).

2.1. Materials

In the present work, smooth, spherical and dry soda lime glass particles (SiLibeads Type S, Sigmund Lindner GmbH) were used (Fig. 1).

It is worth to note that two size fractions (SiLibeads 200 and 400, Type S) were chosen to investigate the dependency of particle size in the presented results. Initially, the particle sizes had the following mass fraction based size distribution $Q_3(d)$, measured with a laser diffraction spectrometer (Mastersizer 2000, Malvern Instruments), SiLibeads 200 $d_{1,50,3}$ = 284.3 µm, SiLibeads 400 $d_{2.50.3}$ = 513.2 µm, see Fig. 2. The used particles contain approximately 72.30% silicon dioxide, 13.30% sodium oxide, 8.90% calcium oxide, 4.00% magnesium oxide and 1.50% other additives according to the manufacturer, are non-porous and have a smooth surface with a mean surface roughness (RMS) of 73.92 nm ± 52.28 nm for $d_{1,50,3}$ = 284.3 µm and 41.54 nm ± 25.68 nm for $d_{2,50,3}$ = 513.2 µm. The evaluation of the surface roughness was performed using an atomic force microscope (JPK NanoWizard I AFM, JPK Instruments AG) in tapping mode. The surface roughness was determined three times for each particle size in an area of $4 \times 4 \mu m$. The sphericity is \approx 1 according to the SEM image processing.



Fig. 1. Scanning electron micrographs of the soda lime glass particles (SiLibeads Type S, Sigmund Lindner GmbH), *d*_{2,50,3} = 513.20 μm; (a) 50× magnification; (b) 100× magnification; (c) 500× magnification.

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