Advanced Powder Technology xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Determination of plastic constitutive properties of microparticles through single particle compression

H. Assadi *, I. Irkhin, H. Gutzmann, F. Gärtner, M. Schulze, M. Villa Vidaller, T. Klassen

Helmut Schmidt University, Institute of Materials Technology, Hamburg, Germany

ARTICLE INFO

Article history:
Received 3 December 2014
Received in revised form 16 July 2015
Accepted 21 August 2015
Available online xxxx

Keywords:
Copper
Microparticles
Compression
Plastic properties
Cold spray

ABSTRACT

There is a need for information on plastic properties of microparticles for application in powder technology. This includes powder compaction processes, in which densification involves particle deformation. The present paper reports on the evaluation of yield strength, ultimate tensile strength and the entire stress–strain relationship of single metallic particles, by combining mathematical modelling and compression tests using a modified nanoindenter. Development and application of the method is initially demonstrated for the case of copper particles of 13–87 μm in diameter. The examined copper particles exhibited similar plastic properties to one another, but different to bulk copper. The method is further employed to work out plastic properties of a CoNiCrAlY powder, for which no bulk data exists. The findings are discussed in view of implications for cold spraying, where the deposition and coating characteristics are strongly influenced by plastic constitutive properties of particles.

1. Introduction

Powder technology often requires an understanding of the deformation behaviour of nano- or microparticles. Examples include powder compaction processes [1–3], where densification of powder involves both rearrangement and plastic deformation of particles, and cold spraying [4–6], where material deposition takes place through high-velocity impact of microparticles of the feedstock powder. Despite the existing demand, there is limited information available on the plastic behaviour of single microparticles [7]. For practical purposes, therefore, plastic properties of microparticles are often inferred from the available bulk data, assuming that particles are harder than the bulk in roughly the same proportion for all materials.

It is clear that inferring particle properties from bulk data has certain limits. First of all, particles are likely to exhibit different properties from the bulk material, because of reduced content and possibly different nature of material flaws and defects in the microstructure. These influences would clearly depend on the type of material, and hence, they are not constant for all materials. Moreover, different powders of the same bulk material may still exhibit significantly different properties. The difference in properties of different powders of the same nominal composition could result from possible differences in microstructure (e.g. different grain size or dendrite arm spacing due to different processing conditions during production), in level of impurities, and in oxygen content (e.g. due to different stocking conditions). There can also be size effects due to the so-called 'strain gradient' [8,9] and 'dislocation starvation' [10,11] effects, which are both expected to result in higher strengths for smaller particles. In addition, there are powder materials including most MCrAlY alloys, for which bulk data may not exist in the first place.

Thus, there is much interest in the evaluation of plastic properties, such as ultimate tensile strength (UTS) of particles of metallic powders. The main question is how to achieve this, i.e. how to evaluate the UTS of a deformable microparticle. For obvious reasons, cutting a cylindrical sample out of a microparticle and performing a miniature tensile testing would not constitute a technically practical answer. Thus, there remains the need for alternative solutions, which can be implemented conveniently and can provide reliable data. The main purpose of the present work is to seek and propose such a solution.

Nanoindentation [12] might be considered as a feasible method to evaluate plastic properties of a microparticle. This method is extensively used to evaluate mechanical properties of materials locally and on small length scales. For the present purpose, nevertheless, there would be some reservations. First, the measurement normally provides only a single value of hardness, which is a surface property. Special characterization techniques might be used to provide hardness as a depth-dependent property, but still they

* Corresponding author at: Helmut Schmidt University, Hamburg, Germany; Tarbiat Modares University, Tehran, Iran.
E-mail addresses: h.assadi@hsu-hh.de, ha10003@modares.ac.ir (H. Assadi).

http://dx.doi.org/10.1016/j.apt.2015.08.013
0921-8831/

would provide only local information from a selected volume of material. The deformation behaviour of particles can be influenced by 'global' size effects, e.g., due to strain gradient or dislocation starvation effects. Therefore, local methods such as hardness testing by nanoindentation does not comprehensively describe overall sample deformation – the latter being more relevant to applications in powder technology. There are also technical problems associated with sample preparation for nanoindentation of particles. Typical methods of preparing cross sections of particles involve using a mounting material, which deforms under nanoindentation loading and hence interferes with the results of measurements.

Mechanical properties of single particles can alternatively be deduced from the force–displacement data as obtained from single particle compression tests [7]. This is also not straightforward, mainly because the specimen is spherical, and hence the deformation is very much inhomogeneous, particularly at the initial stages of compression. Nevertheless, it does not have the drawbacks of standard nanoindentation as mentioned above. In principle, extracting constitutive properties from inhomogeneous deformation is a complex problem, which may require numerical modelling as a complementary tool for data analysis. An example is the determination of constitutive properties from spherical indentation data using a combination of experiments, numerical modelling and neural networks [13]. The present study focuses on a conceptually similar problem, but with a different geometrical setting. While the theoretical setting for the interpretation of particle compression in the elastic regime appears to be well established – dating back to the work of Heinrich Hertz on contact mechanics in 19th century [14] – comprehensive models of particle compression in the plastic regime are yet to be developed [7]. In the cases with plasticity, deduction of constitutive parameters from force–displacement data as obtained from particle compression may require elaborate numerical modelling. Even the elastic models are limited to special cases, e.g. Hertz law is only valid for non-adhesive spherical solids [14]. Overall, physically correct models to analyse particle compression data are available, but their accuracy is limited within certain boundaries depending on the deformation-dependent contact area [15,16].

Based on the above motivation, a method has been developed to work out plastic constitutive properties of individual microparticles of deformable materials. This is done through a combination of experimental and analytical methods whereby flow stress of a microparticle is determined over a wide range of plastic strain. The development and application of the method is initially demonstrated for the case of copper particles of a gas-atomised powder. The method is also employed subsequently to determine plastic properties of a CoNiCrAlY alloy, for which no bulk data exist. The results are discussed mainly with regard to applications in powder technology, though they are considered to be relevant for other purposes, e.g. for studies in the broader topic of size effect in plasticity.

The method and the obtained information are envisaged to be particularly useful for cold spray applications. Cold spraying is a rapidly expanding technology, which can be used to produce dense coatings of metallic materials on a wide range of substrate materials. A main advantage of cold spraying is alleviation of oxidation. It can also be used to produce bulk components, i.e. as an alternative to conventional powder metallurgy, but with the advantage of eliminating the sintering stage [4–6]. A specific application of cold spray is in high-power electrical components, where oxygen-free high-conductivity deposits of copper and aluminium can be obtained at relatively low costs [17,18]. In cold spraying, the final properties of the deposits are governed by thermal and mechanical properties of particles of the feedstock [19]. Most notably, the critical velocity – a main powder characteristic for cold spray applications – is considered to be a strong function of the ultimate tensile strength of particles [19]. As a general rule, higher values of strength result in higher critical velocities for bonding. Cold spray deposition is also influenced by particle ductility, in the sense that materials with limited ductility are generally difficult to be deposited using cold spray. Thus, knowledge of plastic constitutive properties of the spray material becomes an essential part of understanding and hence optimising bonding and deposition in cold spraying. To estimate critical velocity for cold spray applications, the mechanical properties of particles are so far assumed to be the same as those of the bulk material. Incidentally, this rough assumption seems to have worked reasonably well for some materials such as copper. This is so because the difference between the bulk and particle properties is compensated to some extent by adjusting the fitting parameters in the critical velocity formula [19,20]. For the reasons mentioned above, nevertheless, there is a growing need for methods that would allow direct evaluation of plastic properties of microparticles, particularly for cold spray applications.

2. Experimental method

Compression tests of single particles of a gas atomised copper powder – oxygen-free high conductivity (OFHC) from TLS Technik GmbH, Germany – were carried out under quasi-static conditions using a nanoindentation tester – NHT^2 from CSM Instruments, Switzerland – with a flat diamond head of 200 μm diameter. A schematic of the method is shown in Fig. 1. Initially, 38 copper particles of diameters in a range of 10–100 μm were selected for the test. The range of particle size was deliberately chosen to be wide, in order to investigate possible effect of particle size on plastic constitute properties. However, the focus was given to the particles smaller than 40 μm in diameter, for which relatively large plastic strains could be achieved with the utilised nanoindenter (load limit of 0.5 N). The particles were divided into 6 size groups (bins) as follows:

Size (μm)	10–20	20–30	30–40	40–50	50–60	>60
Number	7	5	13	3	6	4

Thus, despite the wide overall range of particle size studied, the range of particle size in the individual 'bins' was restricted, in order to ensure statistical reliability of the measurements. The particles had spheroid geometry with about 0–15% difference between minor and major axes. They were placed individually on a 'hard metal' substrate (WC-Co composite, hardness 1780 HV2) and compressed under a constantly increasing load from zero up to a maximum value of 500 mN within 120 s. The results of compression tests were logged as force–displacement data with 0.1 mN resolution on the force scale. The copper particles were investigated before and after deformation using confocal microscopy – 3D Laser Scanning Microscope VK-X200 series from KEYENCE GmbH, Germany – to reveal the geometry and so, to provide additional information for cross checking with the force–displacement data obtained from the nanoindenter. A cross-section of pristine copper particles was prepared and investigated using standard optical microscopy to reveal the grain structure. Compression tests were also performed under the same conditions on some 15 particles of a gas-atomised CoNiCrAlY powder with a mean composition of 30 wt.% Ni, 21 wt.% Cr, 11 wt.% Al, 0.5 wt.% Y, and balance Co. The results obtained with the initially selected number of particles for both copper and CoNiCrAlY, already showed a reasonably low standard deviation (see e.g. Fig. 13), so that experiments with more particles were not necessary.

Please cite this article in press as: H. Assadi et al., Determination of plastic constitutive properties of microparticles through single particle compression, Advanced Powder Technology (2015), http://dx.doi.org/10.1016/j.apt.2015.08.013