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An alternative to the conventional triaxial compression test

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Abstract

A new test for measurement of the mechanical properties of granular powders is proposed, consisting of upsetting the powder inside a metal tube. Varying the tube material as well as its wall thickness allows superimposing of a variable radial pressure. By pre-compacting the powder inside the tube in a closed die it is possible to test the powder at different densities. The radial pressure is found by correlating measurements of radial bulging of the tube with numerical analysis of tube bulging. Estimates of the error on the determination of the radial pressure are given and it is found that this error may be kept less than ±4%. The coefficient of friction between powder and tube for a specific case is evaluated and found to be between 0.05 and 0.1 at $p_c > 400$ MPa. Data from the test with axial pressures up to 1100 MPa and radial pressures up to 500 MPa are presented. Data on the yield surface for BSCCO (ceramic powder $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ for manufacturing of superconductors) at 74% density are given and found to be in good agreement with previously published data determined by closed die compaction and fracture tests.

The results obtained show that the new test may be a good alternative when high pressures are required or when pressurizing by fluid is impractical.

Keywords: BSCCO; Constitutive equations; Mechanical testing; Drucker–Prager; Powder metallurgy

1. Introduction

This paper suggests a new way of making a cylindrical triaxial compression test for granular or geo-technical materials.

Manufacturing superconductors, such as Fe/MgB_2 or Ag/$(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (Ag/BSCCO), by the OPIT or Oxide-Powder-In-Tube method, implies triaxial loading with variable hydrostatic pressures during mechanical processing. The maximum hydrostatic pressure may exceed 500 MPa. When modelling these processes by e.g. the finite element method, it is required to test the MgB_2 or BSCCO precursor powder under similar conditions.

A large amount of research on triaxial testing of powder has been reported in literature. In the following examples are given on this literature and general principles of triaxial testing.

In a true triaxial test two of the three principal stresses should be independently adjustable, which makes the device very complex to build and use. These designs generally rely on multiaxial piston, flat-jack or fluid-bag loading of cubical samples (Hojem and Cook [1], Mogi [2], Gau et al. [3], Amadei and Robinson [4], Handin et al. [5]). The equipment used for this kind of test is usually designed for pressures smaller than 100 MPa. However, in a true triaxial cell designed by Esaki and Kimura [6], cubic samples were subjected to pressures in the Giga–Pascal range.

Smart et al. [7] succeeded in designing a true triaxial cell using a simpler test procedure than that used in ordinary square true triaxial cells. Radial stresses reached a magnitude of 55.2 MPa (8000 psi); the author's ambition being to reach 68.9 MPa (10,000 psi) in the future.

An alternative way of obtaining true triaxial data consists of subjecting a hollow cylinder to varying internal and external pressures along with torsion (Alsayed [8], Handin et al. [5]). As with the tests involving cubical samples, equipment used to test hollow samples is complex to build and use. The pressures applied are generally smaller than 100 MPa.

Since the typical pressures reached in the true triaxial tests are too low for the testing purpose in the present work, one may look at simpler tests.

An example of a simpler test is the typical rotational symmetric triaxial test (Hoek and Franklin [9]) which only controls radial stress σ_r and thus the hydrostatic pressure by a

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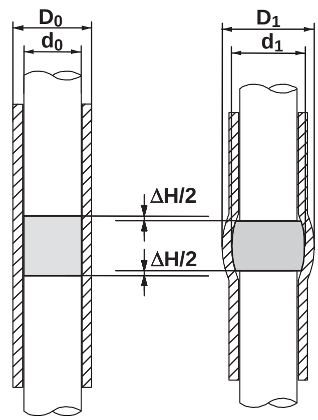

Fig. 1. Outline of PFD test: tube with powder before and after loading.

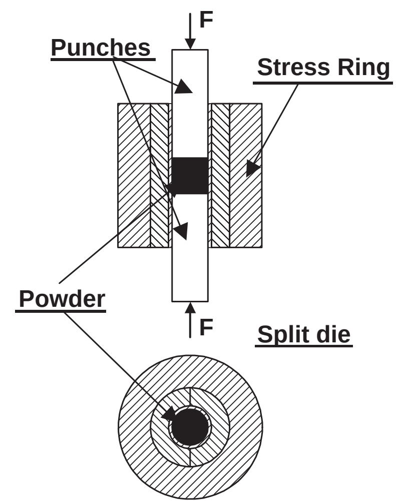

Fig. 2. Set-up for pre-compaction of powder in the PFD test.

superimposed liquid pressure. Using this principle, special test equipment for characterization of powders for powder metallurgy may be designed for fluid pressures as high as 200–700 MPa (Park and Kim [10], Doremus et al. [11], Sinka and Cooks [12]) and 1000 MPa (Massat et al. [13]). These devices are, however complex and expensive to build and they cause limitations due to sealing and friction problems.

Even simpler tests may be used to evaluate the powder yield surface: Axial compression (see, e.g. Brown and Abou-Chedid [14]), diametral compression of pre-compacted samples (see, e.g. Fahad [15]) along with data from closed die compaction (see, e.g. Kim et al. [16]) may provide three points on the yield surface as described in Bech [17]. This strategy has also been used by Allais et al. [18] although with a different set of tests.

In order to obtain a test method of moderate complexity the PFD (Powder in Flexible Die) test was proposed by Bech [17]. In this test the idea is to induce the radial pressure passively by a plastically deformable metal tube. This makes the experimental set-up quite simple and easy to operate (see Fig. 1). The axial and the radial pressure are determined by measuring corresponding values of the axial load, displacement and degree of bulging and calculating the relationship between bulging and radial pressure by FE analysis.

2. Powder in Flexible Die — basic testing method

Before testing, the powder is pre-compacted according to the following procedure, see Fig. 2: 1) lubrication of the inner tube wall, 2) inserting of the tube in a split die clamped in a stress ring, 3) filling the tube with a weighted amount of powder, and 4) loading to a pre-determined density in a 600 kN hydraulic press. The die is mounted in such a way, that it is floating in the axial direction to minimize axial density variation caused by die friction. Lubrication is applied to minimize friction and decrease scattering compared to dry friction conditions. When testing BSCCO a liquid polymer is applied by stippling and when testing MgB_2 a thin film of ZnS grease is used or no lubrication is applied at all. All tests are carried out with tubes having the bore diameter ϕ10 mm. In order to vary the superimposed radial pressure the tube wall thickness and the tube material are varied. Nominal outer diameters of the tubes are chosen as ϕ11, ϕ12 or ϕ13 mm, respectively, using three sets of split dies with corresponding bore diameters. The tube materials applied were steels of types: AISI M3:2, A2, P20 modified (denoted P20m), H13, see furthermore Table 1. The punches are designed to withstand a maximum pressure of 2000 MPa.

After pre-compaction of the powder the split die is disassembled to take out the tube/powder test specimen. The test specimens are subsequently loaded stepwise with the same punches recording corresponding values of axial load and tube diameter with PC based data acquisition. The maximum diameter of the tube bulge is measured with two opposing electronic length transducers (Tesa gauges) with flat heads. The resolution of the gauges is 1 μm.

A combined experimental and numerical analysis applies the measured tube bulge and axial punch displacement to calculate the radial stress $\acute{o}_r$ on the powder billet by FE analysis and the measured axial load to determine the axial pressure $\acute{o}_z$ on the powder billet. Assuming a uniform radial stress exerted by the powder sample on the tube wall during the PFD test, an FE model is set-up calculating the radial stress as a function of the tube bulging. This is explained in detail in Section 3.

Fig. 3 illustrates the data handling. Following the arrows with one, two and three arrowheads indicate the sequence of the procedure. Curve I, showing the axial pressure p_z as a function of the axial strain $\varepsilon_z = ln(1+\Delta H/H)$, is determined experimentally. The load is taken as the mean value of the signal from two force transducers on which the punches are mounted; the axial displacement ΔH is measured by a length transducer.

Table 1
Tube material properties

AISI code	DIN W. Nr.	E/[Gpa]
M3:2	1.3344	230
A2	1.2363	190
P20 mod.	1.2738 mod.	205
H13	1.2344	210