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Original Article

A validation procedure for numerical models of ceramic powder pressing

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ABSTRACT

This paper describes an experimental procedure to validate numerical models used to simulate powder pressing. It consists mainly of two steps: closed die uniaxial pressing followed by isostatic pressing. The uniaxial pressing causes a non-homogeneous density distribution in the pressing direction as a consequence of friction between die walls and powder. In the isostatic pressing, less compacted regions have a larger volumetric strain, resulting in a non-trivial shape of the re-compacted part, which computes indirectly the previous density distribution. Experimental data from both steps are compared to the results from finite element models. The Drucker-Prager/Cap constitutive model was used to represent the compaction of alumina powder. Several simulations covering a range of parameters obtained from the literature were performed to calibrate the model, through an inverse analysis. The developed procedure sheds a light in the methods to calibrate and/or validate constitutive models used for powder pressing.

1. Introduction

Closed-die and isostatic pressing are widely used techniques in industry for green forming ceramic parts. These processes are generally used in industry due to the relatively low cost, the high speed and the possibility to produce complex-shaped parts [1]. To obtain a compact with uniform properties, e.g. density distribution, and to produce parts that do not exceed a certain critical damage during ejection and handling up to firing are well-known challenges in industry [2]. These challenges have required a better understanding of the powder pressing.

Finite element (FE) simulations have been used by several authors [1,3–11] to predict the shape, the density distribution and to prevent crack formation in the green compact powder. Although the FE method is an excellent design tool, it should be emphasized that the accuracy and reliability of the results obtained through simulation are strongly dependent on the model parameters and their calibration, which include, in particular, the ones related to the constitutive model and contact conditions (friction), and not least important, the whole model validation.

The Drucker-Prager/Cap (DPC) model, implemented in ABAQUS™FE code, is an example of a constitutive model generally used for powder pressing. Nevertheless, the calibration of the DPC parameters is complex and requires several tests. DPC yield surface is mainly composed of two segments: Drucker-Prager failure surface and cap surface. The Drucker-Prager surface is defined by two parameters: material cohesion, d, and angle of internal friction, β. The cap surface is defined mainly by a shape parameter, R, and Drucker-Prager parameters. Usually, not less than two experiments are performed to calibrate the cap: an isostatic pressing test to determine the evolution of yield pressure as a function of volumetric plastic strain, and another loading path that touches the cap yield surface, either a triaxial test or an oedometric test. Drucker-Prager surface requires at least two more experiments [12], and commonly, uniaxial and diametral compression tests are used for this surface calibration [13].

The DPC constitutive model has been thematic of several studies [1,3,5,6,8,10,11,14] and many experimental procedures have been developed to aid the calibration/validation of DPC parameters. Some validation techniques rely on density measurements through images analysis. For example, Wu et al. [14] applied X-ray computed tomographic images taken of pharmaceutical green compacts to evaluate density distribution and damage induced in their ejection. Qualitative comparisons between density patterns observed in tomographic images and the FE results were used as a validation tool for DPC constitutive model. Aydin et al. [3] proposed a closed die compaction whose die filling was performed layer by layer, alternating alumina powder and lead spheres. After pressing, the relative position of the lead spheres was measured via X-ray diffraction and used as an indirect measure of the density gradient formed during closed die pressing. Foo et al. [15] used SEM images taken from the diametric fractured cross-section of alumina green compacts discs to identify high-density and low-density regions on the compact.

Measurements of hardness is another method to indirect evaluate density gradients, as used by Sinka et al. [16] to obtain density maps on

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cross-sections of the pharmaceutical tablets and validate the constitutive model calibration, which was done using a die instrumented with radial pressure sensors.

Kadiri et al. [17] developed an interesting experiment to evaluate density gradient induced by closed die pressing, the experiment consists in pressing five homogeneous pre-compacted piled up slices of microcrystalline cellulose in a closed die. After pressing, the density of the slices was measured with a mercury porosimeter and the density gradient was evaluated.

Aydin et al. [18] studied the dimensional variation of die-pressed ceramic green compacts. The study was applied to a high purity α-alumina, a closed die pressing was performed aided by a standard universal testing machine. After ejection, a measurement of the external overall shape of the green compact was made using a non-contacting laser scanning profilometer. Then, the experimental data obtained from compaction and from shape analysis were compared to the results obtained from a finite element analysis.

Although the literature shows some procedures to validate numerical models for powder pressing, these are generally complex and eventually require equipment that is not available in general powder-pressing laboratories. This work aims at establishing a simple experimental procedure for the validation of numerical models for powder pressing. Particularly, a DPC constitutive model applied to an alumina powder was studied. However, the procedure can be applied to other constitutive models as [19–21], including newer developments as Piccolroaz et al. [22,23] and Stupkiewicz et al. [9,24]. Two mechanical tests were performed: a uniaxial pressing performed with axial pressure sensors in the lower and upper piston of a cylindrical closed die, allowing the analysis of normal stress difference applied at the top and bottom of the green compact. Then, after extraction, the green cylinder is re-compacted by an isostatic pressing, resulting in a shape similar to a truncated cone due to the induced density gradient in the first step. So, this second step is a practical way to indirectly measure the remaining porosity from the first step. In order to calibrate the numerical FE model parameters, several parameter sets based on the literature [1,3,5,6,10,11,18] were scanned using an automatized FE model, until minimizing the difference between the normal stresses measured via pressure sensors and their corresponding numerical results (Eq. (3), Section 3.3). The final green compact shapes after two steps of isostatic pressing were then compared to the FE results to validate the numerical model.

2. Material and experimental procedure

An atomized alumina (Al_2O_3) powder was studied in the present paper. The average granule size was 75 μm and the weight loss on ignition was 2.7% on 500 °C for two hours. The apparent density of the powder is 1.28 g cm^{-3} and it was obtained by direct measurements of its mass and volume inside the die cavity. The fully dense density, obtained from Montilha et al. [25], is 3.74 g cm^{-3}. A stereo microscopy view of the powder is shown in Fig. 1. The material was supplied by the company Ceraltec Cerâmica Técnica Ltda, from Brazil.

The experimental procedure consists in two steps: closed-die uniaxial pressing (Fig. 2a) followed by isostatic pressing (Fig. 2b). In the first step, loose powder is introduced into the die cavity (I). The powder is then compacted in a single-acting pressing (II), resulting in a cylindrical specimen with non-homogeneous density distribution (III) as a consequence of friction between unlubricated die walls and powder [26]. The ejection is made removing the lower piston and moving down the upper piston (IV). In the second step, the compacted part (V) is isostatically pressed (VI and VII). In the isostatic pressing, less compacted regions have a larger volumetric strain, resulting in a non-trivial shape of the re-compacted part (VIII).

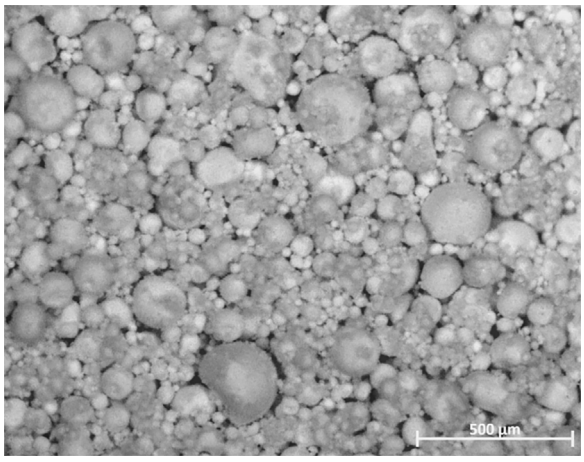

Fig. 1. Image of the studied ceramic powder taken on a Zeiss Stemi 2000-C stereo microscope [25].

2.1. Uniaxial pressing

A metal rigid die with a cylindrical internal cavity and upper and lower pistons instrumented with pressure sensors was designed and constructed as shown in Fig. 3, the die is 300 mm high and 50 mm of internal diameter, the pressure sensors were designed according to Canto [27]. In Fig. 4, photographies highlighting the apparatus details are shown.

The uniaxial pressing step (Fig. 2a) was performed using an INSTRON standard universal testing machine, model 5500R, with a 250 kN capacity load cell. The die cavity was filled with 500 g of alumina powder, resulting in an initial height of powder in the die cavity of ≈199 mm. The test proceeded with a loading followed by an unloading program. A maximum load of 150 kN (equivalent to approximately 76 MPa in the inner cross-section area of the die) was reached during loading and the cross-head speed was 1 mm min^{-1}, as shown in Fig. 5. After the axial unloading, the green compact was ejected.

A polytetrafluoroethylene (PTFE) gasket was used to prevent the powder from entering the gap formed between the matrix and the upper piston. This PTFE gasket was formed in the closed-die, using a PTFE powder, resulting in a 5.5 mm high disc. This procedure ensures a perfect coupling of the gasket with the die. All the system compliance (including the PTFE gasket) was measured with a previous mechanical test without alumina powder, and discounted from the presented results. The aim of this first experimental step was to obtain a non-homogeneous green compact in terms of relative density, and to measure the difference of the applied pressure between the top and the bottom of the compact during uniaxial pressing.

2.2. Isostatic pressing

This step was performed in an isostatic press AIP CP360, applying increasing pressures in two sequential stages: 25 and 200 MPa. At the end of each stage, a caliper (ABSOLUTE Digimatic, Mitutoyo Ltd, accuracy of 0.02 mm) was used to determine the non-regular cylindrical shape dimensions of the alumina compact (Fig. 2b – VIII).

3. Numerical analysis

3.1. Drucker-Prager/Cap constitutive model

The constitutive model used in this study is the DPC model implemented in the Abaqus™ FE code [12]. Its yield function is defined by three surfaces in the p–q plane as shown in Fig. 6a.

The shear surface, F_s, provides a pressure-dependent yield, defined by