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Comprehensive numerical modelling of the hot isostatic pressing of Ti-6Al-4V powder: from filling to consolidation

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Abstract

Hot Isostatic Pressing (HIP) is a manufacturing process for production of near-net-shape components, where models based on Finite Element Method (FEM) are generally used for reducing the expensive experimental trials for canister design. Researches up to date implement in the simulation a uniform powder relative density distribution prior HIPping. However, it has been experimentally observed that the powder distribution is inhomogeneous after filling, leading to a non-uniform tool shrinkage. In this study a comprehensive numerical model for HIPping of Ti-6Al-4V powder is developed to improve model prediction by simulating powder filling and pre-consolidation by means of a two-dimensional Discrete Element Method (DEM). Particles' dimension

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has been scaled up in order to reduce the computational cost of the analysis. An analytical model has been developed to calculate the relative density distribution from powder particle distribution provided by DEM, which is then passed in information to a three-dimensional FEM implementing the Abouaf and co-workers model for simulating powder densification during HIPping. Results obtained implementing the initial relative density distribution calculated from DEM are compared with those obtained considering a uniform relative density distribution over the powder domain (classic approach) at the beginning of the analysis. Experimental work has been carried out for validating the DEM (filling) and FEM (HIP) model. Comparison between experimental and numerical results shows the ability of the DEM model to represent the powder flow during filling and pre-consolidation, providing also a reliable values of the relative density distribution. It also highlights that taking into account the non-uniform powder distribution inside the canister prior HIP is vital to improve numerical results and produce near-net-shape components.

Keywords: Hot Isostatic Pressing (HIP), Discrete Element Method (DEM), Finite Element Method (FEM), Non-homogeneous powder distribution, Relative density calculus

1. Introduction

Hot Isostatic Pressing (HIP) is a manufacturing process widely used in powder metallurgy (PM) to produce near-net-shape components. It is used to produce near-fully dense parts from loose powder through a consolidation process due to a simultaneous application of high temperature and pressure. It is generally employed for parts made of materials that present high costs and difficulty if produced by conventional machining processes, e.g. components made of titanium and nickel-based superalloy. In HIPping the capsule, which undergoes shrinkage, becomes an essential factor in providing dimensional accuracy. Since a sacrificial tool is used, HIPping presents high cost associated with tooling design, which requires an iterative approach in order to obtain the desired dimension of the final component. Numerical simulation can be used to reduce the number of iterations and to help in studying the process reducing the costs, providing also additional information that could be difficult to obtain experimentally (e.g. distribution and evolution of the relative density during HIP over the model domain and force at the tool-powder interface). Numerical models are particularly helpful for tooling design when parts having complex shapes have to be produced [1].

The finite element method is generally used for simulating the HIP pro-

cess [2, 3]. Over the past years, several constitutive material models have been used to simulate the powder compaction during HIP [4–7]. Regarding titanium alloy powder, Xue *et al.* [8] and Kim and Yang [9] simulated the densification mechanism during HIP using two different models: hybrid and Abouaf’s model. The hybrid model was obtained combining the model developed by Fleck *et al.* [10] with that obtained by Tvergaard [11, 12], which modified the yield function for porous metal proposed by Gurson [13]. In particular, the first model was used for relative density (D) values $D < 0.9$, while the latter for $D > 0.9$. A transition phase between the models was also considered. The results showed that both hybrid and Abouaf and co-workers [14] models were in good agreement with experimental observations [8, 9], with the latter slightly overestimating the experimental results in terms of relative density. The Abouaf and co-workers model was also used by Kim and Yang [15]. The numerical results showed good agreement with the experimental results in terms of shape change and density contour plot of titanium alloy powder during HIP.

Usually, independently of the material model used, a homogeneous powder distribution inside the tool before HIP is considered [16, 17]. However, an improvement in the model capability in predicting the tool shrinkage was

highlighted by Van Nguyen *et al.* [18] when implementing a powder distribution obtained by experimental observation of particles' configuration after filling. In fact, experimental results showed that the powder inside the canister (cylindrical tool) is generally distributed inhomogeneously [19], and the relative density gradient prior HIP strongly depends on the filling and pre-consolidation process [20]. This usually contributes to a non-uniform tool shrinkage or high distortion of the final component [21]. The importance to determine the initial distribution of the relative density in the canister was also affirmed by Raisson [22], who refers to it as one of the key points necessary to take into account for improving the prediction of the numerical model, contributing to the production of near-net-shape parts. Recently, also Samarov [23] highlighted the necessity to develop new modelling accounting for filling, and the growing capability to produce near-net-shape parts using the support of numerical models.

The main aim of the present work is to develop a comprehensive numerical model that simulates the filling and powder consolidation in hot isostatic pressing. A two-dimensional model implementing DEM will be developed to predict the relative density distribution of gas atomised Ti-6Al-4V powder. This relative density distribution will be passed to a three-dimensional elasto-

viscoplastic FE model (based on Abouaf model) to predict the powder densification and shape changes during hot isostatic pressing. Two different geometries will be studied: a simple-shaped demonstrator (cylindrical geometry) for calibrating and validating the DEM and FEM numerical models, and a complex-shaped demonstrator to study the influence of the inhomogeneous powder distribution on shrinkage deformation during HIPping. Finally, the numerical models will be validated against results obtained by in-house experiments.

2. Powder: morphology and chemical analysis

Titanium Ti-6Al-4V gas atomized TLS Technik powder was used. The chemical composition of the material is shown in Table 1.

The powder morphology was studied by means of a scanning electron microscope (SEM) using a magnification of X100 and a working distance of 8 mm. Some particles were found having an irregular shape, and a large amount of satellites was observed (Figure 1).

The particle size distribution was measured by laser diffraction using a Sympatec Suceil Automated Wet Dispering Unit. It was carried out in a liquid dispersion, using water as liquid and adding some drops of dispersant.

Particle size was found mainly in the range 45-180 μm (Figure 2), while the tap density was found to be 59.1%.

Finally, a shear ring tester was used for measuring the flowability (ffc) of the powder as the ratio of consolidation stress to unconfined yield strength [24]. The flowability resulted to be $ffc=18$, and it is affected by the large amount of satellites present.

Schulze Dietmar [24] defines the flow behaviour of the powder based on the value of the flowability ffc as it follows:

- $ffc < 1$ not flowing;
- $1 < ffc < 2$ very cohesive ;
- $2 < ffc < 4$ cohesive;
- $4 < ffc < 10$ easy-flowing;
- $ffc > 10$ free-flowing.

The flowability measured for the considered powder is $ffc=18$, therefore powder flow behaviour is classified as free-flowing.

3. DEM modelling of powder filling

The discrete element method is well known to be a powerful mean for simulating powders [25–27]. It was employed to simulate the powder filling in Abaqus/Explicit for a two-dimensional model. The model is composed of particles representing the titanium powder and of a rigid surface. The latter is used to simulate a hopper containing the powder prior filling and a cavity to fill (canister). Each particle representing the powder was simulated as perfectly spherical and rigid. The diameter of the particles was implemented according to the particle size distribution shown in Figure 2.

During filling a particle can collide with another particle (p-p), or with a wall of the cavity (c-p). A schematic of a collision model between two particles is shown in Figure 3. A similar scheme could be drawn for a cavity-particle interaction. The forces considered acting on a particle during a collision were: repulsion, friction and dissipation. The formula for each contribution is reported in Table 2.

The Coulomb friction model was used for the cavity-particle (c-p) and particle-particle (p-p) interaction. However, the exponential decay friction model was implemented for the particle-particle contact. This requires setting the kinetic friction coefficient (μ_k), the static friction coefficient (μ_s)

and the decay coefficient (d_c). The rotational degree of freedom was suppressed for all particles to match experimental results in terms of powder configuration during filling.

In the simulation of the contact among particles, a cohesive force could also be taken into account. Its implementation causes a significant increase of the computational cost of the model, so it should be considered only if needed. The magnitude of the cohesive force, and therefore the need or not for its implementation, mainly depends on the powder material and morphology. Direct observation of the powder behaviour during flowing can also provide useful information. In fact, presence of powder agglomerates usually highlights that cohesive forces are significant. For the titanium powder considered in the present work, the flowability was measured using a shear ring tester and, according to Schulze Dietmar [24], powder flow behaviour is very far from being classified as cohesive. In fact, it can be definitely classified as free-flowing. In addition, no agglomerates were observed during powder flow, with the powder nicely flowing through the filling tube and filling the cavity. In the present work, filling and preconsolidation processes are studied. These are highly dynamic processes where powder is shaken using high frequency and amplitude. A significant amount of energy is transferred

to particles, which predominates over a very weak cohesive force eventually present between particles. For these reasons, a cohesive force was not implemented into the contact.

In addition to the forces acting during a collision, the gravitational force was also applied to all particles in the model. Because of the nature of the process, gravity is very important during filling, transforming the potential energy of particles contained in the hopper into kinetic energy, accelerating particles to high velocity through the filling tube.

Numerical models simulating powder flow are very expensive in terms of computational cost. Even if a two-dimensional model can be developed, it is not possible to take into account the large amount of particles involved in the reality, which leads to a prohibitive computational cost of the analysis. This is particularly true in the case of powders composed of very fine particles. In a two-dimensional model, each particle can be considered as a circle representing the cross-section at the centre of a particle and having a diameter equal to the diameter of the particle considered. In order to reduce the computational cost of the analysis, it is a common practice in the scientific community to scale up the size dimension (diameter) of the particles in order to reduce the number of particles involved in the simulation

[28, 29]. The magnitude of the scaling up factor that can be used depends on the specific problem addressed. The lower value that the scaling up factor can assume is linked to the maximum number of particles that is possible to implement in the simulation: higher is the particles number, lower the scaling up factor can be. The maximum value that the scaling up factor can assume depends on the geometry of the system in which the powder flows. Bierwisch [28] considered the steady state flow rates through a slit opening during a feeding shoe passage at constant velocity to evaluate the effect of the scaling up. Discharge rates were observed to decrease for bigger particles since boundary effects become more predominant, i.e. the particles see an effective smaller opening. This phenomenon was also observed by Beverloo *et al.* [30] in hopper discharge experiments. Finally, limitations arise only if length scales of the system (e.g. the orifice diameter) become comparable to the particles size. The scaling up factor can then be chosen as any value in a range limited by the above described limits. For each value of the mass scaling, coefficients reported in bold in Table 2 have to be calibrated based on experiments. In particular, the calibration has to lead to a model able to simulate the powder behaviour during flow. Therefore, a set of coefficients calibrated against experimental results exists for each scaling up factor in

the range above described.

In the present work, a very fine powder is considered (Figure 2). In addition, filling of large cavities (when compared with particles dimension) is studied. This leads to a very high computational cost, so a scaling up factor as high as possible is desirable. The size dimension (diameter) of the particles was scaled up four times. The value of the scaling up factor allowed to carry out simulations with an affordable computational cost, while containing the particles size when compared to the length scale of the systems studied. In addition, the scaling up factor used allowed to implement in the simulations a number of particles which is statistically representative of the particle size distribution experimentally measured. In order to obtain a DEM model able to represent powder flow during filling as close as possible to that experimentally observed, the coefficients reported in bold in Table 2 were calibrated studying the filling of a simple-shaped demonstrator.

4. FEM modelling of HIPping

The finite element method was employed to simulate the hot isostatic pressing process. A three-dimensional model was developed in Abaqus/Standard, where the tool and the powder parts were obtained by means of partitioning.

The aim of the present work is to compare, against experimental findings, two numerical models for simulating the HIP process: a classic approach implementing FE method where a uniform relative density equal to the experimental tap density is considered in the tool prior HIP; and the proposed approach where a DEM method is implemented for predicting the non-uniform relative density prior HIP, followed by a FEM model for simulating HIP process. Therefore, in order to make a comparison, the same model has to be used to simulate powder densification in both approaches. Different densification models are available in the literature. However, as highlighted in the introduction, the model of Abouaf and co-workers has been implemented in several published researches for different materials, proving to be able to predict tool shrinkage and final relative density for the hot isostatic pressing process when compared with experimental results. For this reason, the titanium powder was simulated using the viscoplastic densification model suggested by Abouaf *et al.* [14]. The creep strain rate for porous material can be written as following:

$$\dot{\epsilon}_{ij} = A\sigma_{eq}^{n-1} \left(\frac{3}{2}c(D)S_{ij} + f(D)I_1\delta_{ij} \right) \quad (1)$$

$$\sigma_{eq}^2 = f(D)I_1^2 + 3c(D)J_2 \quad (2)$$

$$c(D) = 1 + 0.6 \left[\frac{1 - D}{D - D_0} \right]^{0.87} \quad (3)$$

$$f(D) = 0.29 \left[\frac{1 - D}{D - D_0} \right]^{0.78} \quad (4)$$

where D_0 is the initial relative density, σ_{eq} is the equivalent Mises stress for a porous material, J_2 and I_1 are the second invariant of the deviatoric Cauchy stress tensor and the first invariant of the Cauchy stress tensor ($I_1 = \sigma_m$). The coefficients A and n represent the creep constants of the solid titanium alloy, which were determined from uniaxial compression creep tests at different temperatures (750 °C, 850 °C and 950 °C) by Kim and Yang [15]. The coefficients $c(D)$ and $f(D)$ are function of the current relative density (D). When the material reaches the fully dense stage ($D=1$) then $f = 0$ and $c = 1$, hence the Equation (2) reduces to the Von Mises criterion of the dense material. The constitutive equations, Equation (1) - Equation (4), were implemented by means of the user-defined CREEP subroutine [31, 32].

The material model also includes the implementation of the thermal conductivity, thermal expansion and specific heat coefficients as function of the temperature [33, 34]. In addition, the Young’s modulus was considered being dependent on the temperature and relative density, while the Poisson’s ratio only on the relative density [34]. This was implemented by means of a USDFLD subroutine.

The tool, made of 304 stainless steel, was simulated using a temperature dependent elasto-plastic material model [20, 35], where the plastic region was defined by means of the Von Mises yield criterion and isotropic hardening. The thermal conductivity, thermal expansion and specific heat coefficients were implemented as function of the temperature [16, 20].

A variation of the temperature and pressure was applied to the tool during the analysis according to the HIP cycle described in Section 5.4. Finally, a coupled temperature-displacement analysis was carried out.

5. Experimental validation of DEM and FEM models

In-house experiments of powder filling and hot isostatic pressing were carried out on a demonstrator having a simple cylindrical geometry. The aim is the calibration and validation of the DEM and FEM models described

in Section 3 and Section 4, respectively. Models validated will then be used to study the powder filling and the hot isostatic pressing process for a more complex geometry of the demonstrator in Section 6.

5.1. Experimental set-up

The Experimental set-up used for studying the powder filling in a simple cavity is shown in Figure 4. It includes a system composed by a hopper, a die and a slider. A high-speed camera was also positioned in front of the system to record the filling for validation purpose.

The hot isostatic pressing was carried out in a HIP machine at the University of Birmingham, which presents a heating zone of 300 mm in length and 145 mm in diameter, and can reach a maximum temperature of 1450°C and a maximum pressure of 200 MPa. Prior HIPping, the tool was filled with titanium powder, the air removed and the filling tube sealed by hot crimping. After HIPping the FARO Edge Scanner was used to scan the component. The CAD model obtained was then used for validating the FEM numerical model in terms of tool shrinkage.

5.2. Tooling design

The study of the filling was carried out using a hopper and a die made of plexiglass and having a geometry of half cylinder (Figure 4). The transparent material used allowed recording the process using an high-speed camera obtaining information on the powder configuration during filling, and on the total filling time. The cavity was 34 mm in diameter and 29 mm high. The dimension was chosen big enough to observe the powder filling at macroscale level, but not too big in order to contain the number of particles involved and so the computational cost of the DEM model developed.

The tool used for the hot isostatic pressing presented a simple cylindrical geometry with an external diameter of 24.5 mm, a height of 96 mm and a wall thickness of about 3 mm.

5.3. Filling condition

The system configuration prior filling is shown in Figure 4. The slider was positioned to close the connection tube between the hopper and the cavity. After, the titanium powder was poured to fill the connection tube and part of the hopper. Then, the slider was moved to open the connection tube allowing the passage of the powder. At the same moment the high-speed camera was activated to record the filling of the cavity.

5.4. HIP condition

The HIP cycle consisted of a simultaneous increase in temperature and pressure until reaching 930 °C and 100 MPa, respectively. Four hours holding time was used, followed by a decrease in temperature and pressure to room conditions. Heating and cooling rate were 10 °C/min.

5.5. Numerical modelling of a simple-shaped demonstrator using DEM and FEM

A schematic of the DEM model at the beginning of the analysis is reported in Figure 5. The hopper and cavity were simulated as perfectly rigid surfaces. The diameter of the particles was implemented according to the particle size distribution shown in Figure 2. This can be noticed also in the magnification of the numerical model in Figure 5, where particles having different diameters can be observed. Considering a two-dimensional model and scaling up four times the particles dimension helped in containing significantly the number of particles used in the simulation. In fact, 14,076 particles were used. Number of particles considered in the simulation also assured to have the same amount of powder (weight) used in the experiment, which filled partially the hopper and the whole connection tube before filling. The value of the coefficients reported in bold in Table 2 were modified until the numerical results show

the ability of the model in simulating the behaviour of the titanium powder during filling. The values of the calibrated coefficients are reported in Table 3.

In the FEM, due to the axial symmetry, only a quarter of the cylindrical tool was simulated in order to reduce the computational cost of the analysis. The element's dimension was 1.5 mm and the number of elements involved in the simulation was 90,322. The boundary conditions applied are shown in Figure 7. In the case of simple-shaped demonstrator, the initial relative density was considered uniformly distributed over the powder domain and equal to the experimental tap density (59%). Temperature and pressure were applied to the canister in the simulation according to the experimental HIP cycle reported in Section 5.4.

5.6. Results

Numerical results of powder filling obtained using a DEM model were compared with experimental findings. The configuration of the powder during filling at three different times is compared in Figure 6 and Table 4. The numerical results agree well with the experiment in terms of the powder level during filling (h). The maximum error is about 5% over the steps observed, and it decreases during filling. The error on the angle α varies over the time

with a maximum value of 16.28%. In fact, the angle α was observed to change during the experiment. Images from the high-speed camera show that the powder accumulates at the centre and after propagates on the side toward the wall of the canister, causing first an increase of the angle α , and then a decrease. This repeats periodically during filling. Hence, since the free front of the powder changes periodically during filling the error of the numerical model could mainly be due to the out of phase movement of the free front of the powder in the analysis compared with the experiment. The out of phase is due to the scaling up of the particles and to the simplification of considering a two-dimensional model, which were carried out to reduce the computational time of the analysis.

Finally, the filling time in the model differs from that measured in the experiment. In particular, the powder took about 1 second in the model to fill the cylindrical canister, while it took 4.54 seconds in the experiment. This discrepancy is due to the simplification of considering a two-dimensional model and scaling up the particles to reduce the computational time. The ratio between the experimental and the numerical filling times defines the characteristic time of the model. This can be used to calculate the actual time from the simulation time for any other analysis carried out.

The numerical results of the HIPping process obtained by a FEM model were compared with experimental findings. The configurations of the tool at the end of the analysis and after HIPping (scanned component) are shown in Figure 7a and Figure 7b, respectively. The deviation of the tool shrinkage from the experimental result was measured by means of GOM Inspect 2017 software and shown in Figure 7c, where negative values represent a higher shrinkage in the prediction when compared with experiment. Prealignment option was used for the initial alignment, together with a global best-fit option in GOM Inspect 2017.

It is possible to notice a high deviation of the prediction of the numerical tool shrinkage at the welding points. In particular, it results equal to 1.60 mm for the upper welding zone and 1.20 mm for the lower welding zone. This is due to the additional stiffness provided by the welding compared with the model. Moving away from the welding zones the error reduces, with the model showing a better prediction. In particular, the error is generally below 1 mm and it becomes very small in correspondence of the lower surface of the cylinder (0.05 mm).

Finally, in general results show a good agreement between the model and the experiment in terms of tool shrinkage.

6. Effect of powder distribution on shrinkage in a complex-shaped demonstrator

Experiments carried out by Van Nguyen *et al.* [19–21] show that the powder inside the canister is generally distributed inhomogeneously leading to a relative density gradient prior HIPping, which usually contributes to a non-uniform tool shrinkage or high distortion of the final component. This is accentuated for a canister having a complex shape and small features, which unfortunately represents a common case in the industrial practice. For this reason the numerical models developed and validated for a simple-shaped demonstrator were used to study the effect of the relative density distribution after powder filling and preconsolidation on the tool shrinkage for a demonstrator having a complex geometry.

6.1. Experimental set-up

The powder filling and pre-consolidation were carried out applying a vibratory motion to the tool by means of a vibratory shaker AS 200-Control Retsch, assuring a fully filled canister.

About the HIPping, as for the simple-shaped demonstrator the can was filled with titanium powder, the air removed and the filling tube sealed by

hot crimping and the isostatic pressing was carried out in a HIP machine at the University of Birmingham. After HIPping the FARO Edge Scanner was used to scan the component. The CAD model obtained was then used for validating the FEM numerical model in terms of tool shrinkage.

6.2. Tooling design

The geometry of the complex-shaped demonstrator is shown in Figure 8. It is composed by a central core and an external ring, connected by a small passage at the bottom of the tool. Two filling holes were positioned on the top in order to reduce the probability of not filled zones and to obtain a relative density distribution as uniform as possible. The demonstrator presents dimensions of the cavity comparable with those of the simple-shaped demonstrator shown in Figure 5. This assures that the DEM model developed and validated for the simple-shaped demonstrator can be also used for the complex-shaped demonstrator providing reliable results.

6.3. Filling condition

The powder filling and pre-consolidation were carried out applying a vibratory motion to the tool by means of a vibratory shaker AS 200-Control Retsch, which applies to a canister a vertical throwing motion with angular

momentum. The vibratory shaker allows to control the amplitude and the time. The amplitude was set to 1 mm. In particular, the canister was filled until the top of the holes by free flowing using a funnel without extra taping or shaking. After, the filled canister was shaken with the vibratory shaker for 10 minutes and then re-filled again. This process was repeated 9 times, hence until the canister was fully filled.

6.4. HIP condition

The HIP cycle used was the same implemented for the simple-shaped demonstrator. In particular, it consisted of a simultaneous increase in temperature and pressure until reaching 930 °C and 100 MPa, respectively. Four hours holding time was used, followed by a decrease in temperature and pressure to room conditions. Heating and cooling rate were 10 °C/min.

6.5. Numerical modelling of a complex-shaped demonstrator using DEM and FEM

The DEM model calibrated using a simple-shaped demonstrator was used to simulate the powder filling and pre-consolidation for the canister shown in Figure 8. The shaker AS 200-Control Retsch applies a vertical throwing motion with angular momentum. Since the model is two-dimensional, only

the shaking along the vertical direction was simulated. In order to contain the computational cost, the model simulated simultaneous filling and shaking of the canister for 21 seconds that, considering the characteristic time of the numerical model calculated in Section 5.6, are equivalent to 1 min and 35 sec in the reality. The number of particles involved in the model was 38,416. As for the simple-shaped tooling the canister was simulated using rigid surfaces.

The configuration of the numerical model at the beginning of the analysis for simulating the HIP process is shown in Figure 8c. Only half of the geometry was simulated in order to reduce the computational cost. A big discrepancy between experimental and numerical results for the tool shrinkage in a simple-shaped demonstrator was due to the absence of the welding zones into the model. For this reason, the welding zones were also taken into account in the simulation according to the geometry of the demonstrator used for the experimental work, as it is possible to notice from the comparison of Figure 8c and Figure 8d. The model is composed by 323,081 elements, with size of 1.5 mm.

Two different FEM models were developed. The first implemented a uniform relative density distribution prior HIPping equal to the experimental tap density (*Model 1*), while the second implemented a non-uniform rel-

ative density distribution obtained from simulation of powder filling and pre-consolidation using DEM model (*Model 2*). In the latter, a USDFLD subroutine was used in order to assign a different relative density to the elements in the FEM model depending by their location, according to the DEM model results. Since the DEM model for the filling and pre-consolidation is two-dimensional, but the model for simulating the HIP process is three-dimensional, only the left half of the DEM model was used to calculate the relative density distribution. This distribution was then applied with cylindrical symmetry to the three-dimensional HIP model. The methodology for calculating the relative density after simulation of powder filling and pre-consolidation is described in Section 6.6.

6.6. Relative density distribution from DEM model

The particles' distribution after filling and pre-consolidation obtained from the DEM model, shown in Figure 9, was then used to calculate the variation of the relative density across the powder domain. To this end, the domain of interest (left half of the two-dimensional model) was divided in 136 cells C_i , as shown in Figure 9. For each of them the relative density was calculated by means of the image analysis using Matlab software. In particular, it was calculated as the ratio between the area occupied by

the particles in the considered cell and the area of the cell. The image of each cell containing particles was exported from Abaqus so that the particles had white colour and the empty space a black colour. Then the command "bwarea" was used in Matlab to calculate the number of on pixels, i.e. the pixels coloured in white. Knowing the pixel dimension, the area coloured in white was calculated. This area corresponds to the area occupied by the particles. Since the area of the cell is known, the relative density in the two-dimensional space was calculated. Since the relative densities were calculated using a two-dimensional model, a method to calculate the relative density distribution in the three-dimensional space is proposed in this section. In fact, moving from a two-dimensional cell to a three-dimensional cell, the way the particles can be packed/arranged changes affecting the relative density value in that cell.

Given a single particle representing the powder in the result of the DEM model (Scenario I), it is possible to consider a square cell containing this particle and having an edge equal to the diameter of the particle, i.e. equal to $2R$ (Figure 10a). The relative density (D) in the cell can be calculated as following:

$$D_{2D} = \frac{A_{circle}}{A_{cell}} = \frac{\pi R^2}{4R^2} = \frac{\pi}{4} \quad (5)$$

where R is the radius of the particle, and A_{circle} and A_{cell} represent the area of the particle and the area of the cell, respectively. In the three-dimensional space the particle is spherical with radius R and the cell is a cube with edge equal to $2R$ (Figure 10b). The relative density in the three-dimensional case can be calculated as following:

$$D_{3D} = \frac{V_{sphere}}{V_{cell}} = \frac{\frac{4}{3}\pi R^3}{8R^3} = \frac{\pi}{6} \quad (6)$$

where V_{sphere} and V_{cell} represent the volume of the particle and the volume of the cell, respectively. It is now possible to calculate the ratio between the relative density in a three-dimensional space and in a two-dimensional space:

$$\frac{D_{3D}}{D_{2D}} = \frac{\frac{\pi}{6}}{\frac{\pi}{4}} = \frac{2}{3} \quad (7)$$

$$D_{3D} = \frac{2}{3}D_{2D} \quad (8)$$

Finally, using Equation (8) it is possible to calculate the relative density in a three-dimensional cell knowing the relative density in a two-dimensional cell when the cell contains only one particle.

Usually there is more than one particle in a two-dimensional cell, especially when particles with different radius are implemented in the simulation (Scenario II). This case is shown in Figure 10c. Using the image analysis technique in Matlab, the relative density in the two-dimensional cell can be calculated. It can be expressed as it follows:

$$D_{2D} = \frac{\sum_{i=1}^n A_i}{4R^2} \quad (9)$$

where n is the number of particles appearing in the cell, and A_i is the area of the portion of the i -th particle that is contained into the cell. It is possible to consider a configuration equivalent to that reported in Figure 10c, where only one particle is positioned into the cell. The area of this particle is equal to the sum of the areas A_i , and its radius R_{eq} is an equivalent radius calculated as following:

$$R_{eq} = \sqrt{\frac{\sum_{i=1}^n A_i}{\pi}} \quad (10)$$

This configuration is shown in Figure 10d, and the relative density into the cell can be expressed as it follows:

$$D_{2D_{eq}} = \frac{\pi R_{eq}^2}{4R^2} \quad (11)$$

where *eq* stands for equivalent. At this point it is possible to proceed as for the previous case where only one particle was placed in the cell. Hence, it is possible to consider a spherical particle with radius R_{eq} in the three-dimensional space placed in a cubic cell with edge equal to $2R$ (Figure 10e). The relative density in the three-dimensional case can be then calculated as following:

$$D_{3D_{eq}} = \frac{\frac{4}{3}\pi R_{eq}^3}{8R^3} = \frac{\pi R_{eq}^3}{6 R^3} \quad (12)$$

It is now possible to calculate the ratio between the relative density in a three-dimensional space and in a two-dimensional space:

$$\frac{D_{3D_{eq}}}{D_{2D_{eq}}} = \frac{\pi R_{eq}^3}{6 R^3} \frac{4R^2}{\pi R_{eq}^2} = \frac{2 R_{eq}}{3 R} \quad (13)$$

$$D_{3D_{eq}} = \frac{2 R_{eq}}{3 R} D_{2D_{eq}} \quad (14)$$

Finally, using the Equation (14) it is possible to calculate the relative density in a three-dimensional cell knowing the relative density in a two-dimensional cell from image analysis technique, when the cell contains more than one particle.

In the practice, the dimension of the two-dimensional cell is much bigger than the biggest particle in the model (Scenario III), which leads to the configuration shown in Figure 10f. Using the image analysis technique it possible to obtain the relative density in the two-dimensional cell:

$$D_{2D} = \frac{\sum_{i=1}^n A_i}{L^2} \quad (15)$$

where L represents the edge of the square cell. Similarly to the previous case, it is possible to consider the equivalent configuration shown in Figure 10g. The relative density can be written then as it follows:

$$D_{2D_{eq}} = \frac{\pi R_{eq}^2}{L^2} \quad (16)$$

Considering a three-dimensional cell having an equivalent particle of radius R_{eq} placed at its centre (Figure 10h), the relative density results to be:

$$D_{3D_{eq}} = \frac{\frac{4}{3}\pi R_{eq}^3}{L^3} \quad (17)$$

The ratio between the relative density in a three-dimensional space and in a two-dimensional space is:

$$\frac{D_{3D_{eq}}}{D_{2D_{eq}}} = \frac{\frac{4}{3}\pi R_{eq}^3}{L^3} \frac{L^2}{\pi R_{eq}^2} = \frac{4}{3} \frac{R_{eq}}{L} \quad (18)$$

$$D_{3D_{eq}} = \frac{4}{3} \frac{R_{eq}}{L} D_{2D_{eq}} \quad (19)$$

Equation (19) can be used to calculate the relative density in a three dimensional cell when the two-dimensional cell has a square geometry and it is much bigger than the biggest particle in the model. However, when the image of the canister containing the particles obtained from the DEM model is divided into cells, the geometry of the cells is usually a rectangle. Hence, the Equation (19) needs to be generalised for a rectangular cell (Scenario IV). This condition is shown in Figure 10i. The relative density in the two-dimensional rectangular cell can be obtained using the image analysis technique, and it can be expressed as following:

$$D_{2D} = \frac{\sum_{i=1}^n A_i}{L_1 L_2} \quad (20)$$

It is possible to consider an equivalent configuration in the two-dimensional space where the equivalent particle is positioned at the centre of a square cell (Figure 10j). The equivalent radius of the particle can be calculated as expressed by Equation (10), while the equivalent edge of the square cell is calculated as following:

$$L_{eq} = \sqrt{L_1 L_2} \quad (21)$$

The relative density for the equivalent configuration is:

$$D_{2D_{eq}} = \frac{\pi R_{eq}^2}{L_{eq}^2} \quad (22)$$

As for the previous cases, the relative density for a cubic cell, and its link with the two-dimensional relative density can be found as it follows:

$$D_{3D_{eq}} = \frac{\frac{4}{3}\pi R_{eq}^3}{L_{eq}^3} \quad (23)$$

$$\frac{D_{3D_{eq}}}{D_{2D_{eq}}} = \frac{\frac{4}{3}\pi R_{eq}^3}{L_{eq}^3} \frac{L_{eq}^2}{\pi R_{eq}^2} = \frac{4}{3} \frac{R_{eq}}{L_{eq}} \quad (24)$$

$$D_{3D_{eq}} = \frac{4}{3} \frac{R_{eq}}{L_{eq}} D_{2D_{eq}} \quad (25)$$

Equation (25) reduces to Equation (19) in the case that the initial geometry of the cell in the two-dimensional space is a square. The Equation (25) has been implemented in the Matlab software, and used in this paper for calculating the relative density in the three-dimensional space from results obtained by means of a two-dimensional DEM model.

6.7. Results

The configuration of the particles during the powder filling and preconsolidation is shown Figure 11. It is possible to observe the particles entering from the two filling tubes and filling the cavity. The vertical motion applied by the shaker is visible on the left side of the cavity, where the particles are thrown up at the time-step considered. The configuration of the particles at the end of the analysis is shown in Figure 9. It is possible to observe that the model predicts a fully filled canister in agreement with the experiment.

The relative density obtained from the DEM model was calculated as discussed in Section 6.6 and using Equation (25). It is shown in Figure 12 in terms of initial condition applied to the powder in the FEM model for simulating the HIPping. The relative density varies between 56.77% and 62.78%. The average value calculated over the powder domain is 60%, which is about the experimental tap density (59.1%). This result shows that the developed equation is able to predict with good accuracy the relative density in a 3D space from a relative density calculated from a two-dimensional DEM model. Hence, the developed equation is able to intrinsically account for the different way in which the particles are packed when moving from 2D to 3D. A low magnitude was observed near the top of the tool (zones 1 and 2) and in presence of corners (zone 3). A higher value was found in the central part of the canister (zone 4) when compared with the external part of the tool (zone 6). This could be explained considering the position of the filling tube. In fact, the zone 4 is easily filled by the powder since the filling tube is located on top of it. Conversely, in order to reach the zone 6 the powder has to flow through the zone 5 and then ride up until to fill the external ring. Hence, since the powder encounters more difficulty in filling the zone 6, the relative density results lower than in zone 4. It is important to highlight

that the DEM model simulated 1 min and 35 sec of simultaneous filling and shaking in order to reduce the computational cost of the analysis. It could maybe possible to increase the relative density in zone 6 by simulating a longer shaking time.

The numerical results of the HIPping process obtained by both FEM models (*Model 1* and *Model 2*) were compared with experimental findings, i.e. with the CAD model obtained scanning the HIPped component (Figure 13a). The configuration of the tool at the end of the analysis for the *Model 2*, and after HIP in the experiment, are shown in Figure 13b and Figure 13c, respectively. The GOM Inspect 2017 software was used to measure the deviation of the tool shrinkage obtained by the numerical models from the experiment. The results obtained are shown in Figure 14 for both models. Negative values represent a higher shrinkage in the prediction when compared with experiment. Six points were chosen over the tool section in order to better compare the numerical and experimental results. The comparison at each point is shown in Table 5. It is possible to notice that the *Model 2* is able to provide a more accurate prediction of the tool shrinkage. In particular, the model accuracy improves of 18.54% at point 2 and of 19.58% at point 4 when compared to Model 1. The highest difference between numerical models was

found at point 3, where the Model 2 improves the shrinkage prediction of about 90%.

Both models generally predict a higher tool shrinkage when compared with experimental result, with *the Model 2* being closer to the experiment. However, a longer analysis of the filling and pre-consolidation process using the DEM model could help in increasing the relative density in the external ring of the tool, as previously observed. This will help in a further improvement of the *Model 2*, reducing the tool shrinkage in that area.

7. Conclusions

Two numerical models were developed to simulate the powder filling (DEM) and the HIP process (FEM) using a simple cylindrical geometry. In-house experiments were carried out for their validation. The results for the filling agree well with experimental observations in terms of the powder level during filling, with a maximum error of about 5% over the steps observed. The geometry of the free front of the powder is also similar to that observed in the pictures from experiments. The FEM model based on the Abouaf and co-workers model is able to predict the final shrinkage of the tool with an error generally lower than 1 mm. Higher deviation from the

experimental results were found at the welding points. A uniform relative density distribution was implemented in the simulation.

The validated DEM and FEM models were then used for studying the effect of the relative density distribution on the tool shrinkage for a more complex shape of the demonstrator. For this, the simulation of the filling and pre-consolidation processes (DEM) was carried out in a two-dimensional model. Afterwards, by means of the image analysis technique the distribution of the relative density over the powder domain in the three-dimensional space was calculated using a new method suggested by the authors. The distribution was then applied to the elements of the FEM model at the beginning of the analysis of the HIP process. The results obtained in terms of the tool shrinkage were compared with that of the FEM model implementing a relative density equal to the experimental tap density and uniformly distributed over the powder domain (classic approach), and with the in-house experimental results. The numerical results showed a significant improvement of the tool shrinkage prediction when using a non-homogeneous relative density distribution, which can reach values up to 90%, and therefore highlighting the importance of taking into account the relative density distribution over the powder domain to improve the capacity of the model in predicting the final

shrinkage of the tool. Results also showed the capacity of the discrete element method to simulate the powder filling and pre-consolidation, providing furthermore reliable values for the relative density distribution.

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Table 1: Chemical composition of Ti-6Al-4V gas atomized TLS Technik powder

Al [%]	V [%]	Fe [%]	C [%]	H ppm	N [%]	O [%]	Ti [%]
6.31	3.92	0.19	0.012	34.8	0.013	0.10	Bal.

Table 2: Forces acting on a particle during a collision

Force	Formula	Coefficient
Repulsion	$F_r = \mathbf{k}\delta$	$\mathbf{k}$ =contact stiffness coefficient
Friction (c-p)	$F_f = \boldsymbol{\mu}F_r$	$\boldsymbol{\mu}$ =friction coefficient
Friction (p-p)	$\mu = \boldsymbol{\mu}_k + (\boldsymbol{\mu}_s - \boldsymbol{\mu}_k)e^{-\mathbf{d}_c\dot{\gamma}_{eq}}$	$\boldsymbol{\mu}_k$ =kinetic friction coefficient
		$\boldsymbol{\mu}_s$ =static friction coefficient
		$\mathbf{d}_c$ =decay coefficient
Dissipation	$F_d = \boldsymbol{\mu}_0\sqrt{4m\mathbf{k}V_r}$	$\boldsymbol{\mu}_0$ =damping coefficient
		$\mathbf{d}_c$ =decay coefficient

Table 3: Calibrated coefficients for DEM model

Force	Coefficient value
Repulsion	$k=0.05$ N/mm
Friction (c-p)	$\mu=0.3$
Friction (p-p)	$\mu_k=0.1$
	$\mu_s=0.5$
	$d_e=0.02$
Dissipation	$\mu_0=0.3$ (c-p)
	$\mu_0=0.4$ (p-p)

Table 4: Comparison of the configuration of the powder during filling between experimental and numerical results

Filling time	Experiment			Model			Error (%)		
	h [mm]	α_L [deg]	α_R [deg]	h [mm]	α_L [deg]	α_R [deg]	h	α_L	α_R
30%	10.33	33.29	29.06	9.8	32.55	28.5	5.13	2.22	1.92
54%	17.55	34.65	31.26	17	37.1	36.35	3.13	7.07	16.28
100%	Filled	31.46	29.5	Filled	33.14	33.5	0	5.34	13.56

Table 5: Deviation of the numerical results from experiment (mm)

Point	<i>Model 1</i>	<i>Model 2</i>	Improvement using <i>Model 2</i> [%]
1	0.93	0.91	2.15
2	-1.51	-1.23	18.54
3	-0.75	-0.08	89.33
4	-0.97	-0.78	19.85
5	-1.03	-0.93	9.71
6	-0.39	-0.39	0

Figure 1: SEM image of the titanium Ti-6Al-4V gas atomized TLS Technik powder

Figure 2: Particle size distribution for titanium Ti-6Al-4V gas atomized TLS Technik powder

Figure 3: Schematic of a collision between two particles in a DEM model

Figure 4: Schematic of the experimental set-up for powder filling of a simple cavity, showing powder configuration and cylindrical cavity before filling

Figure 5: Schematic of the DEM model for simulating the powder filling in a simple-shaped demonstrator with dimensions in mm

Figure 6: Experimental (left) and DEM (right) results for powder filling configuration at (a) 30%, (b) 54% and (c) 100% of the filling time

Figure 7: (a) FEM for HIPping of a simple-shaped demonstrator, (b) 3D scanned geometry of HIPped part and (c) comparison between the shrinkage obtained by FEM and the 3D scanning

Figure 8: (a) CAD drawing, (b) cross-sectional view, (c) FE model and (d) as-filled tooling for the complex-shaped demonstrator

Figure 9: Creation of 136 cells C_i over the powder domain obtained by the DEM simulation

Figure 10: Scenarios for the calculus of the relative density in a 3D cell from its value in a 2D cell provided by DEM simulation

Figure 11: DEM simulation of powder filling of a complex-shaped demonstrator

Figure 12: Relative density distribution obtained from result of DEM model

Figure 13: (a) 3D scanning of the HIPped complex-shaped demonstrator, (b) configuration of *Model 2* at the end of the FE analysis and (c) cross-sectional view of the actual HIPped demonstrator

Figure 14: Deviation of the tool shrinkage obtained by (a) *Model 1* and (b) *Model 2* from the experiment