

Research Paper

A gravity-independent powder-based additive manufacturing process tailored for space applications



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ABSTRACT

The future of space exploration missions will rely on technologies increasing their endurance and self-sufficiency, including manufacturing objects on-demand. We propose a process for handling and additively manufacturing powders that functions independently of the gravitational environment and with no restriction on feedstock powder flowability. Based on a specific sequence of boundary loads applied to the granular packing, powder is transported to the printing zone, homogenized and put under compression to increase the density of the final part. The powder deposition process is validated by simulations that show the homogeneity and density of deposition to be insensitive to gravity and cohesion forces within a discrete element method (DEM) model. We further provide an experimental proof of concept of the process by successfully 3D printing parts on-ground and on parabolic flight in weightlessness. Powders exhibiting high and low flowability are used as model feedstock material to demonstrate the versatility of the process, opening the way for additive manufacturing of recycled material.

As human reach into space expands, need arises for machines that work under extreme conditions – notably, in absence of gravity. Space exploration missions are severely constrained by payload capacity, and relying upon ground-support would largely increase the risk of failure of such mission [1]. As long endurance missions must be able to solve unexpected problems autonomously, a sustainable approach is the only valid alternative for human spaceflight to non-low Earth orbit: missions' self-reliability will be a key to their success [2].

A vision for space exploration is in-space manufacturing (ISM): fabrication, assembly and integration of small to large structures directly in space [1,3]. ISM has the potential to significantly enhance the self-sustainability of missions, as it could support space exploration missions by maintenance, repair and production of objects without depending on ground-support [4]. Having autonomous manufacturing

capabilities in space also opens the possibility to adapt the design of structural systems to their final function in zero-gravity environment, instead of over-engineering them to resist terrestrial gravity and launch. Approximately 30% of the structural mass of payload shipped to space today could be saved if the launch load constraints could be avoided [5], representing high economical and ecological gains.

Additive manufacturing (AM), also known as three dimensional (3D) printing, encompasses technologies that have two essential advantages for space applications: first, compared to subtractive technologies, they reduce the quantity of waste material produced [2]. Second, they open the possibility to access virtually any geometry, rendering obsolete the geometrical constraints of classical manufacturing techniques. The possibility to recycle former objects into new feedstock material would optimize payload all the more by up-cycling waste to minimize the

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necessary raw material mass.

Strictly speaking of manufacturing, AM already is a permanent tool in space: extrusion-based 3D printers have been on-board the International Space Station (ISS) since 2014 [6,7]. The so-called Additive Manufacturing Facility (AMF) has produced over 200 parts in orbit to this day, including spare parts and tools [7], highlighting AM as an essential tool for future space missions. However, extrusion-based technologies suffer inherent limitations. First, they are restricted to materials showing continuous viscosity decrease with increasing temperature, which makes such technique most adapted to thermoplastics [8]. Moreover, specially manufactured filament feedstock is necessary, which has to be carried along at the cost of large storage volume. Besides, filament-based technologies have limited resolution, restrained by the diameter of the deposited filament; parts produced are typically prone to delamination and highly anisotropic in their mechanical and physical properties [9].

As on Earth, different manufacturing technologies should be available for space in order to respond to the variety of needs. Among AM technologies available on-ground, powder bed fusion (PBF) technologies offer the highest resolution [10] and most versatile techniques [11–13]. The difficulty to handle powders in reduced gravity [14–17,18] has hitherto been an obstacle to further development of powder-based technologies for ISM. A recent breakthrough showed the possibility to adapt selective laser melting (SLM) of metal powders to weightlessness (μg). The method proposed by Zocca et al. [19–21] consists of stabilizing the powder bed by applying a pressure difference between the bottom and the top of the powder-bed using a suction pump. Tested in weightlessness between 2017 and 2019, it enabled the production of parts from ceramic and stainless steel powders [21] while depositing the powder in weightlessness during parabolic flight campaigns (PFCs). Despite the tremendous achievement of producing the first parts manufactured from powder deposited in weightlessness, this method suffers specific drawbacks, detailed by Zocca et al. [21]: as large closed surfaces would prevent the air flow from going through the parts and accessing the next deposited powder layer, closed horizontal surfaces cannot be printed. Using open structures connected by vertical walls, the thickness of those walls is limited to approx. 2 mm. Moreover, the required pump power increases with the powder bed height, necessitating a large quantity of hardware. Finally, powders which include many fines cannot be processed because the filling of interstitial volume becomes too high and annihilates the effect of the air flow. It is also noteworthy that as for all powder-based AM processes used on-ground, the powder deposition step is based on the high flowability of the powder feedstock [22–24]. This implies strict requirements on the manufacturing and storage of the powder, difficult to provide in remote, extreme environments. Furthermore, it complicates direct re-usage of material from previous batches and prohibits closed-loop recycling. Such inherent drawbacks question the superiority of additive technologies for ISM, as the limitation to neither reuse nor recycle powder amounts to the production of large quantities of waste material.

The contours of a technological gap appear: to be suitable for space applications, an AM technology would combine the assets of PBF with the possibility to use powders regardless of their flow-properties, and be robust against changes in g-level. While powder handling remains an important issue on-ground (1g) [25,26], and in absence of constitutive equations enabling large-scale predictions of granular flows in any environmental conditions [27], powder handling technologies for space applications face specific challenges. Primarily, to fulfill gravity-independence, the body force created on each particle by gravity cannot be used as transport mechanism, and normal pressure applied on top of the granular packing cannot be used to induce powder flow, since any such normal pressure is reoriented horizontally by the granular packing according to the Janssen effect [28]. Furthermore, versatility in raw material is required to ease powder storage and recycling: besides the higher stress required to overcome friction and mechanical locking between particles for low flowability powders, a jammed phase [29,30]

also appears at lower packing density for particles showing angular shape and rough surface state [31]. The appearance of a jammed region in a larger packing is a challenge in powder handling, and can draw complete industrial processes to a halt. Recent studies show that by changing the force balance acting on each particle, weightlessness also appears to decrease the *rearrangeability* of the particles' spatial configuration [32], ergo facilitating jamming. Actively avoiding the appearance of a jammed phase hence becomes yet another requirement to ensure reliable functioning of powder handling and 3D printing for space applications.

In the present work, we propose a method to 3D print powders, regardless of the rheological properties of the feedstock raw material, and independently of the gravitational environment. Our method rests on a mechanism for powder transport and homogenization, as well as solidification of the granular material, in a closed container. In the following, *solidification* describes the consolidation process by which the granular material is transformed into a solid, coherent object, by any appropriate physical process, chosen depending on the powder at hand. Robustness against gravity-variations is achieved by depositing powder solely using driving mechanisms shown to induce similar response regardless of the gravitational environment – namely, shear [33] and shaking [34] of the granular material. We focus on the aspects of flow properties posed by different powders and different gravitational environment, using two polystyrene (PS) demonstrator powders of different flowability, on-ground and in weightlessness. We demonstrate qualitatively through computer simulation, then quantitatively and directly through experiments performed on PFCs, that the method proposed is able to produce sintered parts of PS powder that are dense and homogeneous. A key result of our work is that the proposed process is capable of handling powders also of poor flowability, and that the microscopic properties of the finally sintered part are nearly independent of the gravitational environment under which they have been produced.

After describing the AM method in Section 1, discrete element method (DEM) simulation is used in Section 2 to model the powder handling process. An experimental implementation of the AM process follows in Section 3, providing a proof of concept on-ground and in weightlessness through PFCs. Parts manufactured from materials of variable flow-behaviors, under gravity conditions of 1g and μg , are analyzed in Section 4, enabling to assess the performances of the AM process. Section 5 provides concluding remarks and outlook.

1. Additive manufacturing method

AM generally amounts to multiple iterations of two main steps: material deposition, followed by material solidification. In the case of PBF, powder deposition consists in creating a thin and homogeneous layer of granular material, which will then be selectively solidified – the latter being achieved by melting, sintering, or the addition of an extrinsic phase. The present powder-based AM method aims to conduct the material deposition step without relying on the gravitational environment, nor imposing constraints on the raw material flow-properties.

1.1. Process confinement

The approach proposed here consists of confining the raw material in a closed container inside which the entire process takes place (whereas in conventional PBF, deposition and solidification happen on an open powder bed [10,21]). The deposition step amounts to controlling the powder flow inside the closed space to force material to the desired location. Inside the container is a platform or printing substrate on which the object will be 3D printed upside down; the desired location for each new layer to be deposited is the horizontal space under this printing platform. At the beginning of the manufacturing process, the platform is placed at the bottom of the container. It then moves up in discrete steps, each iteration allowing one new layer to be 3D printed underneath the platform and portion of object already printed. Fig. 1 illustrates this

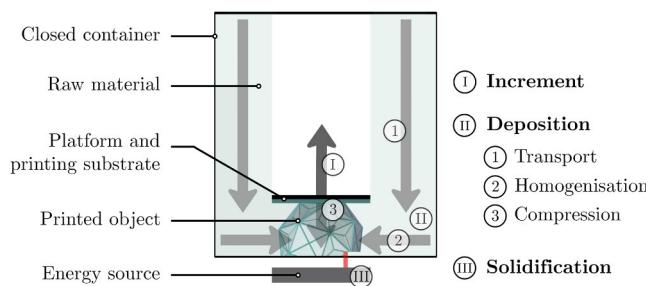


Fig. 1. Principle of the AM method. The feedstock material is confined in a closed container, inside which the entire process takes place. The stepwise process consists of: I. incremental platform rise, II. powder deposition and III. selective solidification of the newly deposited layer. The powder deposition step encompasses the following powder movements: 1. vertical downward transport towards the bottom of the container, 2. horizontal homogenization to create evenly distributed layers under the platform, and 3. compression of the newly deposited layer.

method.

Handling the confined raw material is achieved by moving the container itself to force the material to flow towards the desired location. The powder displacement can be divided into two types of movement: 1. the vertical, downward powder transport and 2. the horizontal, planar movement to create homogeneous layers under the platform. Once deposited, the powder can be selectively solidified. The solidification also takes place inside the container: an energy input is provided from outside to the material inside the container through the bottom wall of the container, transparent to the type of energy used to solidify the raw material.

1.2. Powder deposition

Motion is imposed on the powder exclusively by movements of the container itself, *i.e.* only through boundary forces. The container is cylindrical, axially symmetrical about the axis along which the platform rises. The process is schematized in Fig. 2 through the motion of each part.

Since direct compression of the powder might lead to a fully jammed phase, another transport mechanism must be sought. Shear stress applied to a granular packing creates a primary flow independent of the

gravitational field [33,35], and it can be applied to powders regardless of their flow-properties. Therefore, shear will be the preferred mechanism to trigger controlled granular motion. As shear can also lead to shear-jamming [36], a superposition of shear forces in different spatial directions is used to avoid the creation of stable force chains and thus to preempt jamming.

Also in an effort to avoid putting the granular packing under purely normal compression, the printing platform moving through the container is not a platform but the base of a cylinder; hence, no powder can remain compressed between the platform and the upper wall of the container as the platform moves upward to give space to the printed part. The rising cylinder on which the printing substrate is installed is labeled *inner cylinder*.

The rise of the inner cylinder at each new iteration increases the volume available for the powder under the printing platform, but also in the container in general. To maintain the total powder volume fraction constant throughout the process, the volume gain is compensated by lowering the part closing the container on its upper section. Labeled *closing disc*, this annular shaped part links the outer wall of the container to the inner cylinder; it descends on the feedstock area, to push downward the raw material stocked there. Again to avoid normal compression, the closing disc describes oscillatory rotation around the cylinder axis while descending. It rotates alternatively in each direction at a frequency of 1 Hz, and it is equipped with vanes penetrating the powder bed. The oscillatory motion forces the material to reorganize regularly, destroying and reforming the “fragile skeleton” [30] of force chains supporting the downward pressure. At each reorganization, the particles are pushed to a position of (temporary) stability lower along the z-axis than their previous one. It is ensured in this manner that the material is periodically pushed downwards and enters the next step of the powder handling system: the transport area.

Vertical transport of granular material in a closed container has been widely studied on-ground, for instance in the case of silo discharge, showing that normal pressure applied on top of a granular packing is reoriented horizontally [28]. The present application poses a supplementary requirement: the body force created on each particle by gravity can also not be used as transport mechanism, as it would render the powder handling method gravity-reliant. Therefore, a screw conveying system is used to transport the powder vertically: the rotating outer container (labeled *outer tube*) is equipped with helical blades that shear the material downward as they rotate. This mechanism enables the handling of a wide range of powders regardless of their physical or rheological properties, as will be demonstrated below.

During granular shear, force chains form oblique to the direction of shear [37]. Force chains are the lines of force through dense granular packings; as such, a fragile networks of force chains is desired in order to transmit force from the boundary to the bulk. If the force chains however percolate into a stable configuration, they might transfer the load directly from the screw conveyor to the inner cylinder, creating a jammed phase that is stable against further motion. To ensure that force chains forming are intermittently destroyed, a secondary force field is superposed by rotating the inner cylinder simultaneously with the screw conveyor. The inner cylinder is equipped at its bottom with blades to enhance powder-powder contact during the rotation. The mechanism used to defuse the force chains is illustrated by Fig. 3; superposition of perturbations in different directions have been used previously to tune jamming in dense shear thickening suspensions [38]. In the present case, the inner cylinder imposes a torque on the particles in contact with it as it rotates, which “elongates” the chain, thereby destabilizing it by rolling particles out of the main stress direction [30,39]. Superposing a secondary flow direction forestalls jamming by defusing the long force chains as they appear, constantly imposing plastic deformation to the packing. It is noteworthy, that if surface friction increases stability of the force chains, both mechanisms used in the superposition of directions of drive are enhanced by an increase in particle-surface friction, as friction also renders contacts between particles and container's surface more

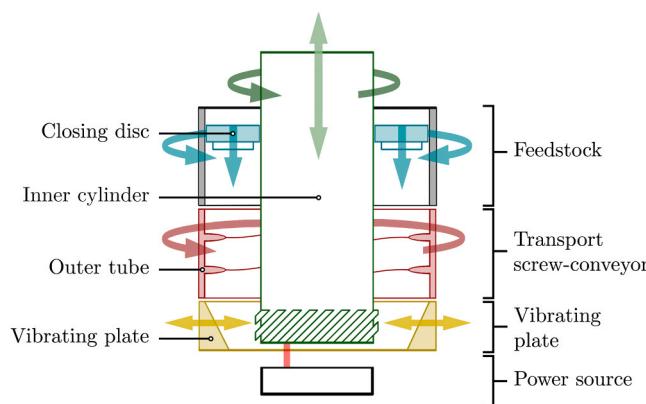


Fig. 2. Schematic representation of the different stages of the AM process. The powder container is divided in four parts, each moving independently: the closing disc descends while describing oscillatory rotation; the inner cylinder rises stepwise, rotates and descends to compress the newly deposited layer; the outer tube rotates to activate the screw conveying system; the vibrating plate produces horizontal shaking. The apparatus can be divided in four stages (from top to bottom): the powder feedstock, the material transport by screw conveyor and the homogenization by horizontal vibration and the selective solidification.

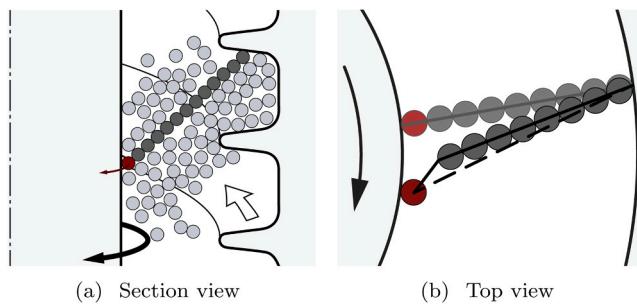


Fig. 3. Illustration of the superposition of force chains (in dark gray) imposed by the screw conveyor motion (white arrow), and motion created by rotation of the inner cylinder (black arrow), disrupting the end-components of those force chains (red particle).

stable.

Once the powder particles have been brought to the bottom of the container, the powder needs to be spread homogeneously on the entire bottom surface. To do so, the inner cylinder rises by more than one layer-height, leaving under the printing platform an empty volume greater than that of a powder layer. Then, homogenization is realized by applying horizontal shaking to the bottom of the container. Granular homogenization by planar shaking is a well-known mechanism on-ground [40,41]. In weightlessness, shaking a confined granular sample leads to the formation of a large cluster bouncing around the middle position of the axis along which shaking is applied. This has been shown through simulation [34,42,43] and experiments [44–46]. Using alternated shaking along x - and z -axis, powder is shifted towards the middle position. Powder coming from the sides is added as it reaches the lower part of the container. Shaking continues until powder completely fills the bottom layer. The duration needed to reach this state is monitored *in-situ* to ensure that the entire printing surface is filled with powder (see Sec. 1.3).

After having been transported down and homogeneously distributed horizontally, the newly deposited powder layer undergoes normal compression by the platform descending onto it. The powder layer is hence compressed between the bottom wall of the container and the previously solidified layer sitting on top of the platform, increasing the packing density up to close packing. The compression ratio, expressed as a function of the layer height, is a printing parameter.

Finally, the newly deposited powder layer can be selectively solidified through the bottom wall of the powder container, by the energy source placed outside. Since this wall must be transparent to the type of energy used to solidify the material, it is labeled *solidification window*.

1.3. *In-situ* monitoring

Quality and repeatability have been identified as the Achille's heel of AM [47]. The problem of defects appearing in printed parts constitutes a major obstacle for AM in industrial applications, as layer-wise material deposition increases the risk of defects appearance; yet it also enables a direct insight into the bulk of the object while it is manufactured. Using this specificity for *in-situ* monitoring would enable to spot defects and hinder their appearance [48,49].

The present process is designed to allow for closed-loop control by *in-situ* monitoring. Primarily, the torque needed to rotate the inner cylinder mono-directionally during material transport is recorded. The torque developed during oscillatory rotation of the closing disc as it descends is also recorded, providing a second source of information on the raw powder's rheological behavior. The adaptive control loop allows for the immediate reaction to changes in flowability upon changes in environmental conditions. It must be noted, that here flowability does not refer to an inherent property of the material, but to the flow exhibited by a powder in the given conditions and environment in which it is

processed, which includes – but is not limited to – the gravitational environment. Hence, the torque developed during powder transport is compared to a scale established *a priori*, giving the typical duration needed to deposit a material as a function of its flow response. This adaptive closed control loop allows for the optimization of powder deposition without being limited to situations formerly encountered.

In parallel, a quality assurance system is implemented to monitor the appearance of defects during material homogenization. The solidification window is transparent not only to the solidification energy but also to visible light; hence, live imaging captures the progression of the powder layer homogenization from below. This image-analysis procedure facilitates the detection and quantification of heterogeneities in the powder layer, continuing the material deposition procedure as long as the chosen metrics have not dropped under a threshold set to identify an acceptable degree of homogeneity.

Proposing a step toward autonomous manufacturing, concurrent use of the two “probe-and-adapt” systems mentioned above (*i.e.*, torque-based rheometrical feedback and *in-situ* monitoring) does not only provide traceability of defects, but their automated correction, ensuring constant and reliable manufacturing quality. The full printing procedure, including *in-situ* monitoring mechanisms, is schematized in Fig. 4.

This process was developed to function independently of feedstock flowability and variations in g-level. To observe the effect of varying those parameters on the powder deposition efficiency, for the sake of comparison, the process shall be tested in those different situations with the same (fixed) printing parameters. For this reason, the *in-situ* probing is not automated in the series of experiments reported here. Printing parameters include powder deposition time, rotation speed of the different parts, sintering time and compression ratio. In an effort to limit the μg time necessary in weightlessness, the experimental campaign is preceded by a preliminary simulation study. Besides the optimization of rotation speeds (not shown here), the simulation aims to minimize weightlessness μg -time needed for empirical parameter screening, by verifying if the same parameters can be used for manufacturing high and low flowability materials, on-ground and in weightlessness.

2. Simulation of powder flow

The following simulation study of material deposition is used to validate the printing parameters prior to experiment, for both 1g and μg . Lacking precedent on which to rely for comparison, and in an effort to narrow the possible sources of variations, the same printing parameters are used on all the situations presented. The simulation study guides the choice of these printing parameters, while minimizing the risk of failure, notably for weightlessness experiments, by testing the qualitative influence of key parameters, such as gravity and increase in powder cohesion.

2.1. *Simulation methodology*

DEM simulation [50] is used to validate qualitatively the powder deposition process. It is implemented in the open-source package LIGGGHTS [51] (version 3.8.0), a molecular dynamics (MD) variant suitable for granular materials.

The system modeled encompasses $N = 76,000$ deformable 3D polystyrene (PS) spherical particles of diameter $d = 2$ mm, surrounded by an aluminum (Al) container. Due to computational constraints, considering equivalent setup size, the particle size chosen in the simulation is significantly larger than in experiment ($d = 80\mu m$, see Sec. 3). In order to represent strongly cohesive powders, we include a simple model for attractive interactions among particles, and concentrate on a qualitative rather than quantitative comparison.

In the model, each point particle i is represented by a sphere, and overlaps with a particle j by a distance of δ_{ij} . The Hertz-Mindlin contact model [52–54] is used for the force calculations: each particle pair interacts through a non-linear spring-dashpot viscoelastic mechanical

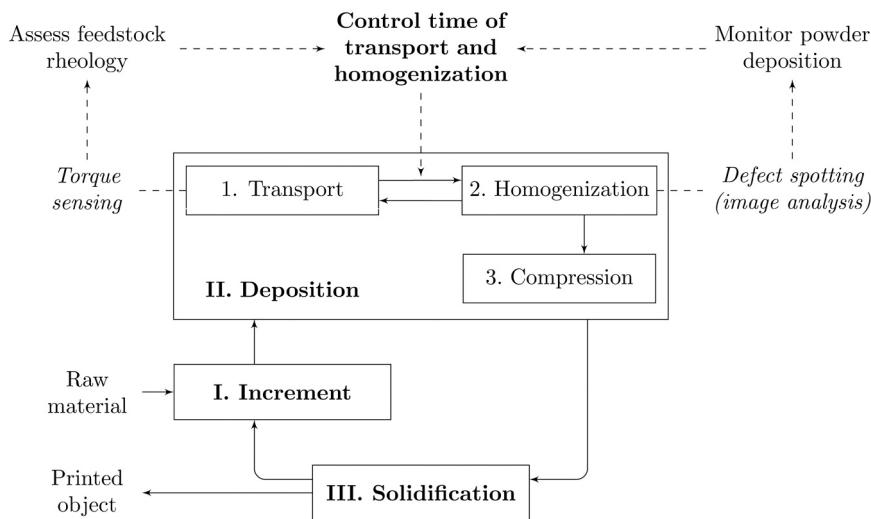


Fig. 4. Manufacturing procedure, including closed control loop used to optimize the duration of powder deposition and ensure defect-free powder layers.

response. The force \vec{F}_{ij} resulting from a collision is expressed as a function of the overlap δ_{ij} and relative velocity through its normal v_{nij} and tangential v_{tij} components:

$$\vec{F}_{ij} = (k_n \delta_{nij}^{3/2} - \gamma_n v_{nij} \delta_{nij}^{1/4}) \vec{n}_{ij} + (k_t \delta_{nij} \delta_{nij}^{1/2} - \gamma_t v_{tij} \delta_{nij}^{1/4}) \vec{t}_{ij} - \kappa A_{ij} \vec{n}_{ij}. \quad (1)$$

The first two terms of Eq. 1 are the normal and tangential components of the force governed by the stiffness parameters $k_{n,t}$ and viscoelastic damping parameters $\gamma_{n,t}$. They represent the mechanical properties of the material constituting the particles. While their numerical value can be linked to true material properties (elasticity modulus and Poisson ratio), they are bounded by numerical constraints. Notably, optimization of simulation time requires to fix a sufficiently large time step dt ; but deeming dt too large would result in overlooking certain collisions, thus invalidating the simulation. It is customary to consequently adapt the value of $k_{n,t}$ to remain at the lower end of the permissible spectrum, hence reducing computational effort while maintaining expected effects on the large scale. In the light of previous studies [55–57], we estimate that $k_{n,t} \gtrsim 10^5 \text{ N m}^{-3/2}$ is sufficient to obtain stiff particle behavior using a time step $dt = 5 \cdot 10^{-7} \text{ s}$. To reproduce inelastic particles, during a collision, most of the energy should be dissipated by viscous damping or through friction between the particles, which is obtained in the over-damped regime, once $\gamma_{n,t} \gg \sqrt{k_{n,t}} m$, which is by far the case with $\gamma_{n,t} \sim 10^8 \text{ kg m}^{-1/2} \text{ s}^{-1}$. The exact parameters used are given in Table 1. The tangential term is curbed to respect $|F_{tij}| \leq \mu |F_{nij}|$, where μ is the friction coefficient (here $\mu = 0.3$ for PS-PS contact), to account for frictional interactions between particles. Rolling friction is also implemented, and provides an additional torque to the particles [58].

Table 1

Simulation parameters $k_{n,t}$ and $\gamma_{n,t}$ for the two types of interactions present in our model: among polystyrene particles (PS-PS) and between polystyrene particles and aluminum container (PS-Al). Elastic coefficients $k_{n,t}$ are in $\text{N m}^{-3/2}$ and viscoelastic damping coefficients $\gamma_{n,t}$ in $\text{kg m}^{-1/4} \text{ s}^{-1}$.

Numerical parameters	PS-PS	PS-Al
k_n	Normal elastic coefficient, in $\text{N m}^{-3/2}$	$1.52 \cdot 10^6$
k_t	Tangential elastic coefficient, in $\text{N m}^{-3/2}$	$2.04 \cdot 10^5$
γ_n	Normal viscoelastic damping coefficient, in $\text{kg m}^{-1/4} \text{ s}^{-1}$	$4.56 \cdot 10^8$
γ_t	Tangential viscoelastic damping coefficient, in $\text{kg m}^{-1/4} \text{ s}^{-1}$	$4.10 \cdot 10^8$
		$8.01 \cdot 10^8$

The third term, $-\kappa A_{ij} \vec{n}_{ij}$, where A_{ij} is the disc-shaped contact area between spherical particles i and j , and κ the cohesion energy density, provides an extra cohesive component to the force calculation, in the form of a Simplified Johnson-Kendall-Roberts (SJKR) model (based on the corresponding model of solid adhesion [59]). It appends an additional attractive normal force contribution to the force calculation at each collision: as two particles enter into contact, this supplementary force tends to maintain the contact proportionally to the contact area, calculated from the overlap. The cohesion energy density κ represents all the cohesive forces between the particles, due to the reduction in surface free energy when particles are in contact. It encompasses many possible mechanisms responsible for cohesion in granular materials. We hence use κ as a proxy to tune the powder flowability in the simulation; values from $\kappa = 10^{-4} \text{ N m}^{-2}$ to 10^{-5} N m^{-2} are used to cover a wide range of interparticle-attraction strengths (see Fig. 8).

The boundary conditions are embodied by contact surfaces following the stepwise powder deposition process described in Sec. 1 (Fig. 2), with the dimensions of the apparatus used for experiment (see Fig. 9). The consecutive motions of each of those parts are listed in Table 2. The number of particles is calculated to fill the volume of the experimental container with a packing fraction $\varphi = 0.6$, slightly lower than random close packing (rcp) of frictionless monodispersed material [60]. To achieve a realistic insertion of the particles inside the complex geometry, this step (Move 0 in Table 2) is done by free fall of the particles inside the container (so-called sequential generation of rcp), followed by the descend of the closing disc, which seals the container.

Snapshots from the visualization of the simulation results are presented in Fig. 5, and a video is available as video 1 of the supplementary material [61].

2.2. Procedures for data analysis

The critical region regarding powder deposition quality is the center of the powder layer at the bottom of the processing container, which will be selectively solidified. In simulation, this region is a disc of radius $15d$ (where d is the diameter of one particle), and height $5d$ after the compression step. Analysis of the particles' distribution within this region is carried out by finding the local packing fraction φ associated to each particle from a Voronoi tessellation [62]: each particle is assigned a unique flat-faced polyhedron representing the region of space closer to the center-point of each particle than to the center of any other particle. The ratio of particle volume and polyhedron volume represents the local packing fraction [63]. The mean local packing fraction $\langle \varphi \rangle$ can hence be found for any region of space. Voronoi tessellation is performed by the

Table 2

Description of the consecutive motion of each element of the powder container modeled in the DEM simulation, corresponding to the 3D printing procedure described. Move 2.a and 2.b occur simultaneously. d is one particle diameter. Time steps are given in simulation units, $dt = 5 \cdot 10^{-7}$ s.

Time step (end of move)	Move	Part	Movement	Velocity
$1.0 \cdot 10^6$	Move 0	Closing disc	Linear movement in $-z$ -direction: close printing bed	Speed 0.23 ms^{-1}
$1.2 \cdot 10^6$			Particle settling (realized under $+1g$)	
$1.4 \cdot 10^6$	Move 1	Inner cylinder	Adapt g-level	
$1.6 \cdot 10^6$	Move 2.a	Inner cylinder	Linear movement in $+z$ -direction: incremental rise by $10d$	Speed 0.20 ms^{-1}
	Move 2.b	Closing disc	Clockwise rotation about z -axis	Period 0.025 s
$1.8 \cdot 10^6$	Move 3	Closing disc	Linear movement in $-z$ -direction	Speed 0.05 ms^{-1}
$2.0 \cdot 10^6$	Move 4	Closing disc	Counterclockwise rotation about z -axis	Period 0.05 s
$2.2 \cdot 10^6$	Move 5	Outer tube	Clockwise rotation about z -axis	Period 0.025 s
$2.6 \cdot 10^6$	Move 6	Vibrating disc	Shaking along x -direction	Period 0.05 s
$3.0 \cdot 10^6$	Move 7	Vibrating disc	Shaking along y -direction	Period 0.05 s
$3.2 \cdot 10^6$	Move 8	Inner cylinder	Linear movement in $-z$ -direction: compression by $5d$	Speed 0.1 ms^{-1}

Python package SciPy [64] and verified with the Voro++ open source software library [65]; ambiguous Voronoï cells (e.g. at the system's boundaries) are discarded. To visualize the simulation results, the bottom layer of the powder bed is divided into concentric rings, each equally spaced by $2d$. This division of the powder bed bottom layer is represented in Fig. 6(b), with the printing region represented in gray.

2.3. Simulation results: influence of gravity

The influence of the gravitational environment is studied by adjusting the gravitational constant $g = 9.81 \text{ m s}^{-2}$, by multiplying it by $+1$, 0 and -1 (labels used in the subsequent text and in Fig. 6 are respectively $+1g$, $0g$ and $-1g$). Under $0g$, no mass-dependent external force field is applied: particle flow is induced solely by boundary motion and forces transmitted through surrounding particles. The $+1g$ environment promotes the fall of particles toward the bottom of the process container, whereas the $-1g$ condition tends to pull the particles towards the container's top, working against the desired flow direction.

The evolution of $\langle\varphi\rangle$ at the end of each step of the powder deposition process, averaged over the printing region in Fig. 6(a), shows that the gravitational vector strongly modifies the granular density after the rise of the inner cylinder (move 1), before the container's motion begins to shift material downwards: under $+1g$, the particles' weight creates a collective motion towards the bottom, and then horizontally redistributes the particles as they slide on each other, piling until they reach a slope corresponding to the angle of repose. The empty space under the printing platform is filled at $\langle\varphi\rangle \approx 0.08$ solely under the effect of gravity at the boundary of the printing region, which can be observed in Fig. 7(a): at $t = 1.4 \cdot 10^6$ time steps, the outer part of the bottom layer, at a distance $l > 15d$, is already filled at $\langle\varphi\rangle \approx 0.41$, and particles have reached the outer diameter of the printing region, with $\langle\varphi\rangle(10d \leq l \leq 15d) \approx 0.23$, while this region is completely empty of particles under $0g$ – see Fig. 7(b). In contrast, under $0g$ and $-1g$, the

particles are not pushed towards this empty space, respectively due to a lack of mass-dependent force or a force towards the top of the container. With move 2 begins the powder transport phase, which triggers collective granular downward motion, regardless of the gravity-level. $\langle\varphi\rangle$ undergoes a steep increase, but the normalized difference in packing fractions between $+1g$ and $0g$ – represented in the inset of Fig. 6(a) – remains at 40%. Throughout this transport phase (moves 2–5), material pushed downwards slowly invades the printing region, already creating a relatively homogeneous layer under $+1g$, and remaining at the outskirts in $0g$, see Figs. 7(a) and 7(b). In Fig. 6(a), the large standard deviation in $\langle\varphi\rangle$ results from the variability throughout the printing region: as material is slowly pushed downwards, the center remains at low or null packing fraction – see Fig. 7(b). The normalized difference between $+1g$ and $0g$ has dropped to 30% by move 4. During move 5, the screw conveyor transports a large quantity of material downwards, which is forced towards the container's bottom: some is pushed towards the center, reducing the difference in $\langle\varphi\rangle$ between $+1g$ and $0g$ to approximately 5%. The homogenization phase follows (moves 6 and 7): the granular density becomes homogeneous throughout the printing substrate ($l \leq 15d$ in Figs. 7(a) and 7(b)), regardless of the gravitational environment (see also Fig. 6), culminating for all g-levels at $\langle\varphi\rangle \approx 0.55$. Finally, compression of the newly deposited layer (move 8) compacts the powder and erases the remaining difference between g-levels, with $\frac{|\langle\varphi_{1g}\rangle - \langle\varphi_{0g}\rangle|}{\langle\varphi_{1g}\rangle} \approx 0.15\%$. Zooming-in on the state of the bottom layer at the end of the powder deposition, $\langle\varphi\rangle$ after move 8 is shown in Fig. 6(b) throughout the bottom layer. The packing fraction within the printing region (for $l \leq 15d$) is very high, with $\langle\varphi\rangle \approx 0.87$. The average packing fraction achieved is significantly higher than the expected close packing of monodispersed spheres, indicating that particles overlap due to compression. It is noteworthy that the most central region, being composed of the smallest volume, is more prone to statistical variability, which explains its stronger variation in $\langle\varphi\rangle$ as a function of the g-level.

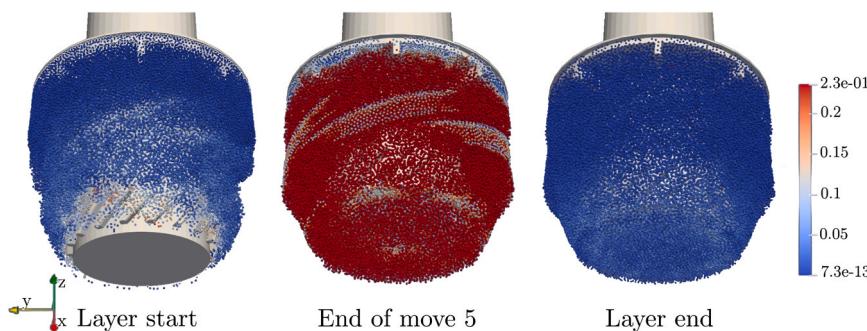


Fig. 5. Snapshots of the simulation results: on the left, at the beginning of the process, before the inner cylinder rise; in the middle at the end of move 5, corresponding to the end of powder transport; on the right at the end of the deposition of one layer. The particles' color indicates their velocity magnitude, made explicit by the scale bar on the right (in ms^{-1}).

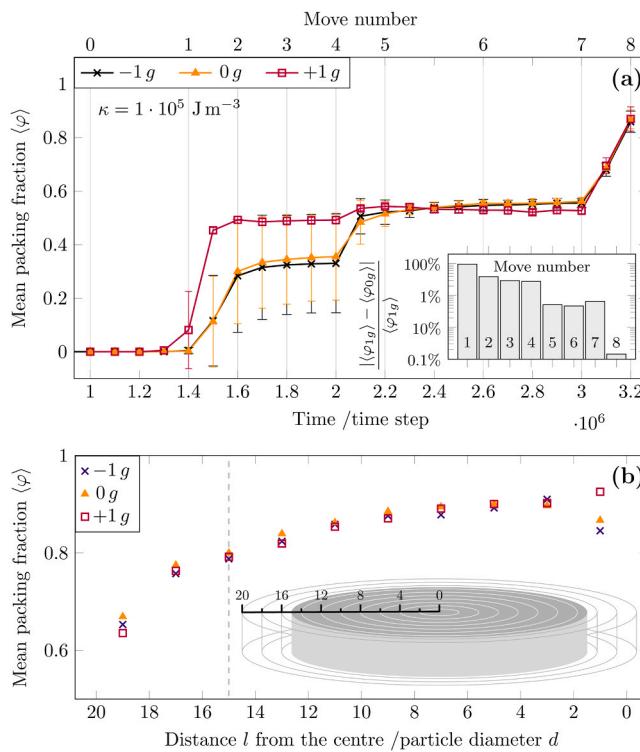


Fig. 6. (a) Mean packing fraction $\langle \varphi \rangle$ on the printing region after each move of the powder deposition process (moves 0–8), for gravitational acceleration $+1g$, $0g$ and $-1g$ ($g = 9.81 \text{ m s}^{-2}$). The local packing fraction φ is obtained for each particle by Voronoi tessellation; error bars represent the standard deviation of the φ distribution over the entire printing region. The inset shows the normalized difference between $+1g$ and $0g$ at the end of each move, expressed in percentage. (b) $\langle \varphi \rangle$ per ring of the bottom layer at the end of move 8 (end of layer deposition), for $+1g$, $0g$ and $-1g$. The division of the powder bed bottom layer in concentric rings, equally spaced by two particle diameters d , is represented as an inset. The printing region is only the middle cylinder, represented in gray (and marked by a vertical dashed line).

Besides, the closeness between the $0g$ and $-1g$ results is explained by the fact that we look solely at the bottom layer center, enforcing the importance of the horizontal motion (layer homogenization).

To summarize, the quality of the final powder deposition shows no dependence on the gravitational environment, although the simulations show, for the parameters chosen, that the powder transport does depend on the g-level. This suggests that the process is robust against changes in gravitational environment.

2.4. Simulation results: influence of interparticle cohesion

The other relevant parameter for the AM process is dependence on the flow-properties of the powder feedstock. The corresponding simulation variable is the cohesion energy κ , which adds to the contact force an attractive term along the axis defined by the aligned particles centers, proportional to κ times the area of contact A_{ij} (see Eq. 1). Numerical values of κ are varied from 10^{-4} J m^{-3} (for a very low cohesion, hence highly flowable powder) to 10^5 J m^{-3} (for a highly cohesive powder). $\langle \varphi \rangle$ throughout the powder deposition is presented in Fig. 8.

For all $\kappa \leq 10^4 \text{ J m}^{-3}$, our model shows very similar results: such high flowability powder flows under the printing platform quickly, as it is already put into motion by the concurrent inner cylinder rotation and closing disc descend (move 2). Non-cohesive powders are very sensitive to collisions and tend to move freely within the container. The remaining steps of the process have little effect on the powder repartition, with the exception of move 5, which slightly increases powder density in the printing region by bringing some more material

downward, and the compression step (move 8) which significantly increases $\langle \varphi \rangle$ from 0.52 to 0.85. Locally measured $\langle \varphi \rangle$ shown in Fig. 7(c) reveal the same trend, as the transition between the initial state with the absence of powder at $l \leq 15d$ and the post-transport stage where $\langle \varphi \rangle \approx 0.5$ at $l \leq 15d$ happens in less than $0.2 \cdot 10^6$ time steps.

The highest interparticular cohesion available in our model is $\kappa = 10^5 \text{ J m}^{-3}$, which in the simulation represents a low flowability powder. Such highly cohesive particles tend to remain together, as outward forces resulting from collisions are minimized. This is particularly visible on moves 2–5 in Fig. 8, where the difference between highest and lowest κ reaches its maximum ($\approx 35\%$). The efficiency of the powder downwards screw conveying (move 4) in microgravity is remarkable, closing the gap to bring all powders to $\langle \varphi \rangle \approx 0.5$. In Fig. 7(b), the powder is pushed slowly downwards but does not invade the full printing region before being homogenized by shaking (moves 6–7). This homogenization step brings the entire printing layer to $\langle \varphi \rangle \approx 0.56$, slightly higher than for high flowability powders. Again, the final layer compression completes the deposition process by fixing the particles in a state of high density, independently of their cohesive interactions: it erases all differences and brings the final $\langle \varphi \rangle$ to ≈ 0.9 for all materials.

To conclude this simulation study, using the same 3D printing parameter set, all the simulation results have shown that whereas increased powder cohesion, as well as reversed gravity or absence thereof, modifies the powder flow behavior at each step of the powder transport taken separately, the general powder deposition functions independently of the raw material's flowability (cf. Fig. 8) and of the gravitational level (cf. Fig. 6). On average among all experiments discussed, it results in a final layer packed at $\langle \varphi \rangle \approx 0.87$, with a standard deviation among all experiments of 0.01 only, erasing both internal and external variability factors. Universally fixed printing parameters can hence be used in our weightlessness experimental campaign, enabling acute assessment of the effect of feedstock flowability decrease and g-level change.

3. Experimental proof of concept

Having confirmed by simulation the working principle of the proposed AM process, the latter is implemented in two 3D printers and tested on-ground and in weightlessness. Parabolic flight campaigns (PFCs) supported by the German Aerospace Center (DLR) and European Space Agency (ESA) are used to conduct weightlessness (μg) experiments: the 34th DLR PFC in September 2019, used for testing the hardware in weightlessness conditions, and the 72nd ESA PFC in November 2019 (in the context of the ESA Education FlyYourThesis! GrainPower project), during which five samples were successfully manufactured fully in weightlessness, providing a proof of concept for the AM process. PFCs allow, by flying an airplane describing parabolic trajectories, a period of free-fall of about 22 s, which allows to perform experiments in weightlessness. This weightlessness period is surrounded by hypergravity phases as the plane rises and swoops. This maneuver is typically repeated thirty-one times per flight day, a campaign consisting of three to four flight days. The final experimental rack used to produce the weightlessness samples contains two 3D printers, used to each produce one sample per flight-day.

3.1. Experimental set-up

A 3D printer built to implement the AM process described and its digital counterpart are shown in Fig. 9. The powder container is composed of two coaxial cylinders: (A) the inner cylinder and (C) the outer tube, respectively of diameter 65 mm and 120 mm. From above, the container is enclosed by (B) the closing disc, filling the space between the two cylinders and moving down to control the powder bed volume. On the bottom, it is closed by (D) the vibrating disc, which contains the solidification window, through which the raw material will be sintered by (E) the energy source, placed underneath the powder

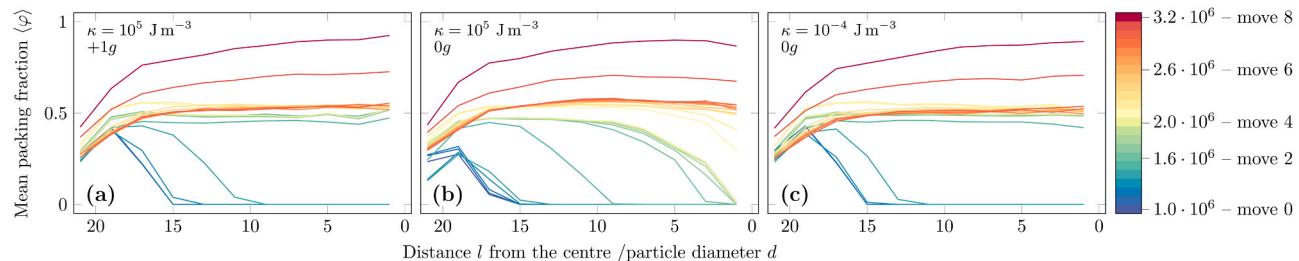


Fig. 7. Mean packing fraction $\langle \varphi \rangle$ per concentric ring (equally spaced by $2d$) as a function of the distance from the bottom layer center (at abscissa 0) to its outer bound (at 20), expressed in particle diameter d . The color code corresponds to the time t given in simulation time steps.

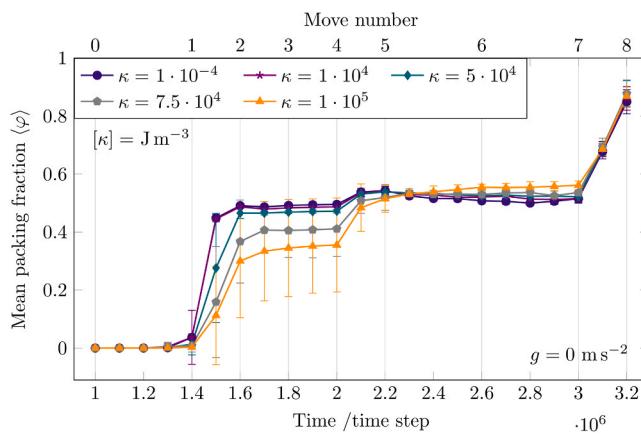


Fig. 8. Mean packing fraction $\langle \varphi \rangle$ on the printing region after each move of the powder deposition process (moves 0–8), for values of the cohesion parameter κ ranging from 10^{-4} J m^{-3} to 10^5 J m^{-3} , the extreme cases available in the present simulation.

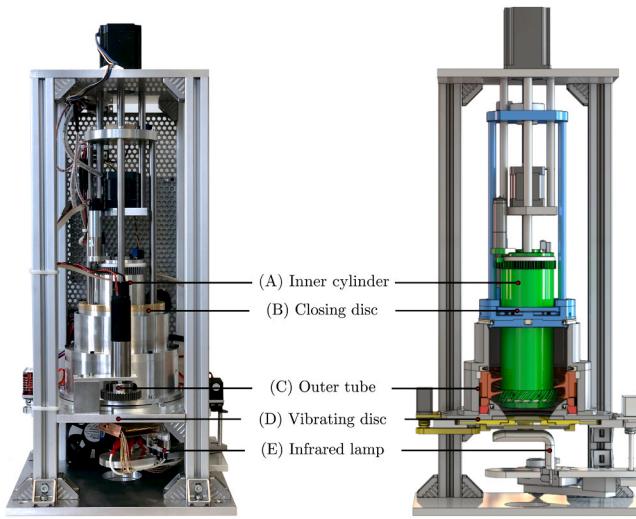


Fig. 9. (Left) photography and (right) Computer Assisted Design (CAD) of the 3D printer. The main components of the printer are labeled. On the CAD, the fixed structure is represented in gray while the moving parts are colored, each color representing a movement block.

container. A thorough technical description is available elsewhere [66].

The type of energy source and the solidification window's material determine the maximum temperature allowed, hence the adequate raw materials. In the demonstration experiment presented here, polymer powders are used; an infrared (IR) lamp serves as energy source (Quattro IR emitter from Heraeus, Germany), and the solidification window is a

doubled 5 mm thick fused silica plate (proQuartz, Germany). The general structure is formed by $30 \times 30 \text{ mm}^2$ aluminum profiles on which the individual parts are mounted. The printing volume available is a cylinder of diameter 65 mm and height 50 mm. The motion of each part exactly follows the description given in Fig. 2 – specific motion parameters are given in Table 3.

The 3D printing procedure is as follows: first, the inner cylinder rises vertically to make space for the new layer. At the end of the powder deposition phase, it moves vertically downward to compress the powder underneath. To maintain a constant volume inside the powder container, the closing disc translates down to counteract the motion of the inner cylinder; simultaneously, it rotates in alternating direction to probe the packing and keep it from jamming. Powder downward transport is carried out by the screw conveyor placed inside the outer tube, rotating to push the powder towards the bottom of the container. The shell of the inner cylinder (excluding the printing substrate) also rotates (independently of its translation motion) to probe the powder flow from within the container, providing a rheological characterization of the feedstock powder.

As mentioned previously, in this first study the *in-situ* probing control loop is not automatized to allow better overview of the effects of our variable parameters (gravity and powder flowability) on the powder deposition.

3.2. Material

To demonstrate feasibility of AM from raw materials of high to mediocre flowability, two exemplary demonstrator powders are used, which share all physical characteristics except for their surface roughness. This enables us to test solely the effect of a decreased flowability on the powder deposition and sintering process. The model substances are crafted as follows. A monodisperse spherical polystyrene (PS) powder of main diameter 80 μm is used (where size and dispersity are given by the manufacturer). Produced by the company Microbeads under the name Dynoseeds, the powder as-received from the manufacturer is dry-coated with a sub-micron angular PS dust, as shown in Fig. 10(a) and 10(b). This powder is labeled *Rough Surface (RS)* in the subsequent text. The

Table 3
3D printing parameters, l being the layer height.

Part of printer	Motion	Operating parameters
(A) Inner cylinder	Translation	z steps: $+1000 \mu\text{m}$, $-500 \mu\text{m}$ ($l = 500 \mu\text{m}$, compression ratio 1/2)
	Rotation	Rotation speed 0.102 ms^{-1}
(B) Closing disc	Translation	z step: $-200 \mu\text{m}$
	Rotation	Rotation speed 0.126 ms^{-1}
(C) Outer tube	Rotation	Rotation speed 0.226 ms^{-1}
(D) Vibrating disc	Shaking	Amplitude 2 mm
		Frequency 5 Hz
(E) Infrared lamp		Lamp power 500 W
		Sintering time 20 s

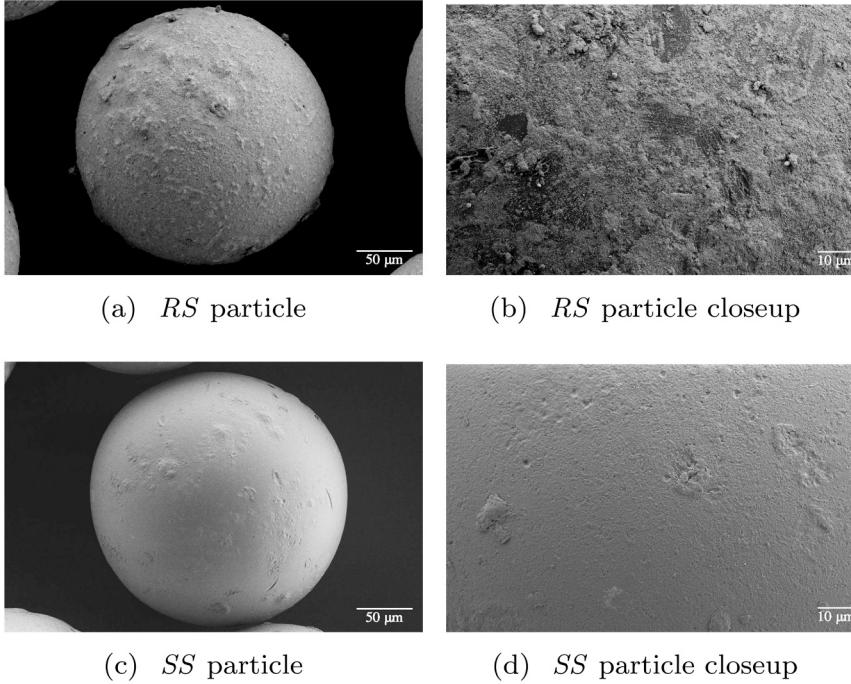


Fig. 10. Scanning Electron Microscopy (SEM) images of the polystyrene (PS) powder used as raw material (the microscopies are done for particles of diameter 250 μm), (a) for powder with rough surface state (RS), at the scale of a particle and (b) at the scale of its surface; (c) for powder with smooth surface state (SS), at the scale of a particle and (d) at the scale of its surface. The increased surface roughness in the sub-micrometer range is clearly visible in (a) and (b), while (c) and (d) exhibit a much smoother surface. SEM imaging is done at 1 keV.

surface coating is removed by wet-sieving the powder batch and subjecting it to ultrasound at a frequency of 20 kHz for a duration of 8 hours per batch. The resulting particle surface state is shown in Fig. 10(c) and 10(d): the asperities have been removed, leaving exposed the smooth surface of the spherical particles. This powder is named *Smooth Surface* (SS) in the subsequent text.

The influence of the surface coating on the flowability of the powder is not *a priori* evident, as the addition of smaller guest particles on large host particles can either enhance or hinder their flowability. A small quantity of hard guest particles, of diameter much smaller than the host particles [67], can enhance flow by reducing the curvature radius at the contact point, hence the Van-der-Waals attractive interactions [68]. However, it was shown empirically that if the surface area coverage (SAC) of the host particles is greater than approximately 20%, the inverse effect can be observed, *viz.* a reduction of the flow properties as compared to the pure host particle [68,69]. We thus expect our RS particles to constitute a powder with reduced flowability compared to the SS particles; in the following, we will also confirm this experimentally by flow energy tests.

The resistance to wear of the rough coating is tested to ensure its persistence throughout the experiment. To validate the principle of our experimental study, it is essential that the model powders retain their flow-properties throughout the entire experiment, independently of the

load to which they will be submitted during powder deposition. To test this, we have subjected the particles to shear in a Couette-Taylor shear cell [70], continuously for 20 hours at increasing shear rate, $\dot{\gamma} = 10^{-2} \text{ s}^{-1}$ to 10^3 s^{-1} , including air-fluidization before and after at a flow rate of 5 L min⁻¹; SEM microscopies taken before and after are shown in Fig. 11. The surface is visibly unchanged by the long duration shear test: the rough coating is still distributed on the entire particle surface, showing that it remains despite frictional contacts.

How to best characterize the rheology of powders for AM is an open question [22]. The term *powder flowability* is widely used and intuitively understood; however, it lacks a clear definition, as it does not rely on a normalized measurement method, nor on international system (SI) units. A powder is defined as *flowable* if it tends to plastically deform (*i.e.* flow akin to a liquid) under a certain stimulus – which may simply be its own weight. In contrast, a *non-flowable* powder resists flowing and tends to maintain its shape akin to a solid. If it is forced into flowing by an external load, it will do so in large chunks of material themselves preserving their shape, in an erratic manner and showing a higher tendency to block the flow by forming stable aggregates that can withstand a finite amount of stress before yielding. In other words, contacts between particles tend to be more enduring [68]. The terms *high flowability* and *low flowability* are used throughout the present work following this phenomenological definition.

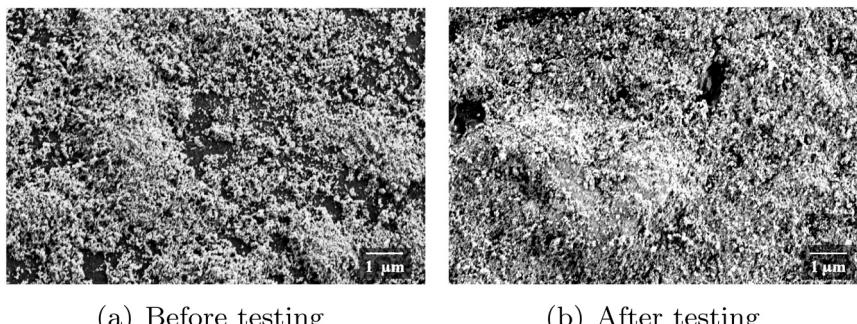


Fig. 11. SEM images of the *Rough Surface* (RS) polystyrene powder used as raw material (a) before and (b) after a twenty hours long shear test (see text for details). The sub-micrometer surface roughness remains present in the same quantity and homogeneity on both images. Imaging is done at 1 keV.

In absence of a universal definition, the procedure that we used to characterize flowability of the SS and RS powders is the so-called *flow energy* measurement available on the Freeman Technology 4 Powder Rheometer (FT4) [71,72]. It consists of extracting from a powder bed of height h an helix of angle α and radius r , at different speeds v , recording the torque M and normal force F to calculate the *flow energy* E . Following Wenguang et al. [73,74], a dimensionless *flow energy* E^* is introduced by normalizing the *flow energy* by the potential energy of the sample:

$$E^* = \frac{E}{m_s h g} = \frac{1}{m_s h g} \int_0^h \left(\frac{M(h')}{r t \tan \alpha} + F(h') \right) dh', \quad (2)$$

where m_s is the total mass of the sample and g the gravitational acceleration. Qualitatively, an increase in *flow energy* E^* corresponds to a decrease in powder flowability. Fig. 12 shows the specific *flow energy* E^* , measured at penetration rotation speeds between 10 and 100 mm s⁻¹ for powders at high packing fraction $\varphi = 0.6$. Different rotation speeds are used to ensure that the results are robust; note that a speed of 100 mm s⁻¹ is comparable to the rotation speed used in our printers. In an effort to contextualize our demonstrator powders, the reader is provided with material for comparison: E^* is measured for RS powders of smaller and larger particle diameter (respectively $d = 40 \mu\text{m}$ and $d = 250 \mu\text{m}$), and for a Ti-64 metal alloy powder, typically used in AM. The latter metal powder is used to compare the flow properties of the two polymer demonstrator powders to that of a commercial AM material, although the AM process presented is not in its current form adapted to 3D print such material.

It has been shown that particles of smaller diameter tend to exhibit higher cohesion, due to the predominance of van der Waals interactions [68,76–78]. Hence the corresponding powders undergo a flowability decrease. This decrease is indeed captured by the specific *flow energy* test shown in Fig. 12(a): as the diameter d doubles (40–80 μm), the *flow energy* needed to make the powder flow decreases (by 5.7% on average), showing a *better* flowability of the powder comprised of larger particles. The particle diameter is then increased to 250 μm , inducing a further decrease of E^* (by 9.6% in average), again showing that larger particles amount to a powder of *higher* flowability. The specific *flow energy* increases with decreasing particle size; this trend is preserved over all the rotation speeds measured.

In Fig. 12(b), E^* is presented for the SS and RS 80 μm diameter powders used in our experiment. In the dense packing of rough particles, surface friction is activated as asperities on the particles' surfaces interlock: the *flow energy* is 15% higher for the RS powder. The effect of surface roughness is clearly visible: increased friction begets higher stress necessary for particles to slide along each other, thus higher stress to trigger flow; the RS powder exhibits lower flowability than the SS powder. This effect is at least comparable to the one induced by a change of the particles' diameter by more than a factor 6.

Finally, a comparison is provided in Fig. 12(c) to a commercial 3D printing metal powder: the polydisperse Ti-64 alloy powder [75], with particle diameter in the range 10 μm to 40 μm . Its flowability is slightly higher than the RS powder but much lower than the SS powder, placing our two demonstrator powders as boundaries framing a typical material used in powder-based AM, in terms of flow-behavior. Note that the RS powder of similarly small particle size would be significantly less flowable than the Ti-64 powder at all rotation speeds.

3.3. Manufacturing procedure & sample characterization

3D printing procedure

For the sake of comparison, all samples are obtained using the same printing parameters. The material deposition lasts 20 s. The layer height is 500 μm (corresponding to $\sim 6d$) and the compression rate is 50% – i.e. the printing platform rises by 1000 μm before each deposition step, then descends by 500 μm to compress the newly deposited layer. Following the compression stage, which immobilizes the newly deposited layer, sintering takes place. For the μg -samples manufactured in weightlessness, sintering starts at the next parabola, to allow the entire manufacturing procedure to be carried out in weightlessness. To provide as much information as possible on the powder deposition, no further compression is applied during solidification, and sintering is preferred over melting, as it maintains possible heterogeneities of the deposited powder layer. The sintering parameters were chosen such that at each new layer, a depth of 1000 μm be sintered (i.e. twice the layer height), to ensure full cohesion between the subsequent layers.

3.3.1. Printing substrate

All samples were 3D printed on a pre-manufactured printing substrate. Examples of printing substrates are visible in Fig. 13(a) (circled in yellow and labeled “full printing area”) and Fig. 14. Substrates were manufactured by oven-sintering the same PS powder as used in the experiments on an aluminum holder for approximately 1 hour at 200 °C.

3.3.2. Sample preparation

Samples are prepared to faithfully reflect the powder deposition at its most challenging position: in the center of the printing volume. The energy source for sintering (IR-lamp) provides heat in a rather evenly distributed manner over an entire area, enabling solidification of the entire sample-section within less than 20 s. However, due to design limitations the IR-lamp provides slightly stronger heating on two regions of the printing bed of approx. 10 mm by 30 mm, represented in blue in Fig. 13(a)–(b). The lamp is placed accordingly to ensure that the center of the printing bed be under one of the areas of preferred heating, as shown in Fig. 13(a). Each 3D printed sample is hence cut to extract a square sample of approx. 10 by 10 mm, cut out of the center of the

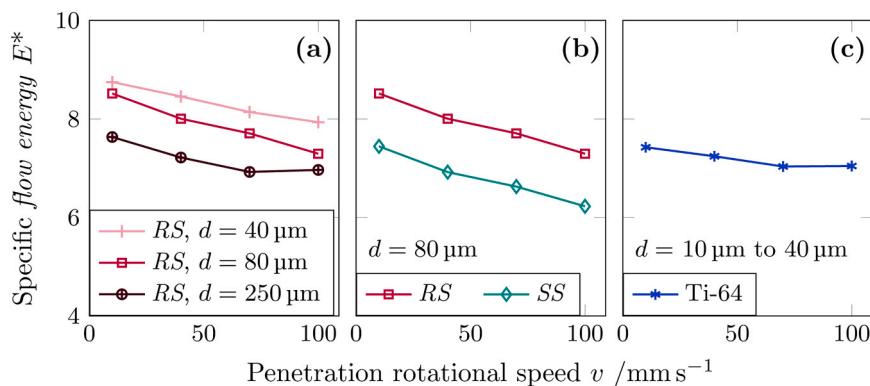
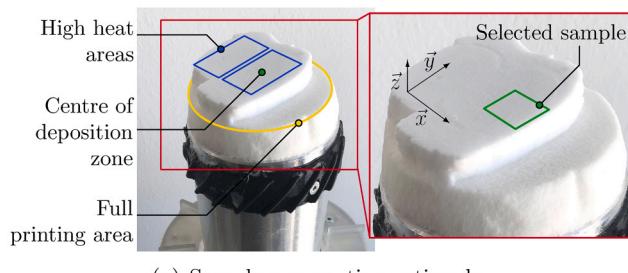


Fig. 12. Specific *flow energy* E^* as a function of the helix speed v for samples (a) of Rough Surface (RS) polystyrene powder of diameter 40 μm , 80 μm and 250 μm ; (b) of 80 μm -diameter (SS) and RS powders (model materials used for additive manufacturing experiment); (c) of polydisperse Ti-64 powder with size in the range 10 μm to 40 μm , a typical material for metallic powder-based AM [75].



(a) Sample preparation rationale

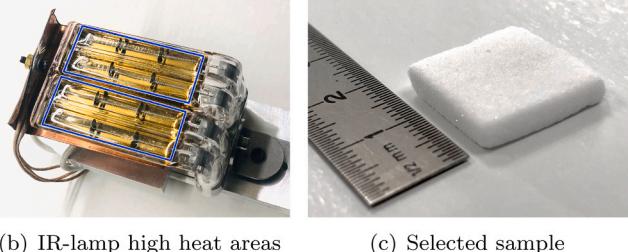


Fig. 13. Sample preparation rationale. (a) Superposition of high heat zones with the center of the printing area, including delimitation of the selected sample. The sample represented here is manufactured on-ground from *Rough Surface* powder. (b) IR-lamp including schematic representation of high heat zones (in blue on the picture), corresponding to the zones marked in blue on the full sample. (c) Resulting sample after cutting (the sample represented here, labeled sample L3, 3D printed in weightlessness from *Rough Surface* powder).

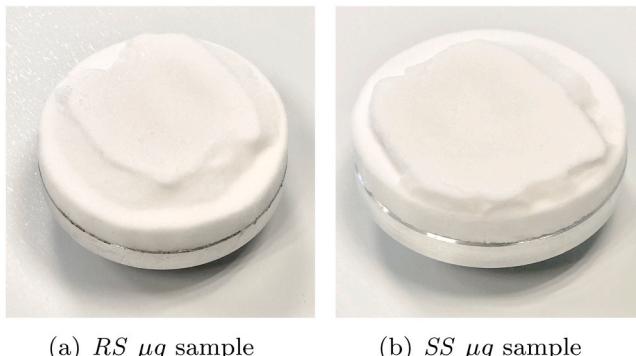


Fig. 14. Samples 3D printed in weightlessness (μg), from (a) RS and (b) SS powders, as extracted from the printing bed.

printing bed – see Fig. 13(c).

3.3.3. Reference “known good” and “known bad” samples

One sample was sintered on-ground under the best possible conditions to serve as a reference “known good” sample for comparison with 3D printed ones. It is sintered from the highly flowable SS powder in an oven at 200 °C for one hour, under a weight of 3 kg to ensure continuous pressure *during* sintering. This aims to increase material density, as the continuous pressure enhances degassing and porosity size reduction, akin to hot isostatic pressing [79]. The resulting “known good” exemplary sample is shown in Fig. 17(a).

Another sample, 3D printed in weightlessness under wrongly tailored solidification parameters, serves as a “known bad” sample (see Fig. 17(d)). In this sample, the solidification energy was too high, so that overheating resulted in partial melting instead of sintering into homogeneous layers. As the empty space between particles becomes trapped in molten material, the volume loss is not counteracted by reduction of the printing bed volume. Therefore, the supplementary volume transforms into large porosities scattered along the sample. This sample is

used here to validate the characterization procedure by showing the results obtained for a “worst case scenario”.

3.3.4. XCT specifications & data analysis

In-bulk characterization of the samples is done by X-ray computed tomography (XCT). The machine and scanning parameters used are presented in Table 4.

The size-homogeneity of the porosities in the samples is assessed by two automatized image analysis procedures implemented in the Python PoreSpy library [80]. First, images are made binary by the automated procedure available in ImageJ [81] (*intermodes* method, automated thresholding). Then, an average density is calculated per “slice” of depth 8 μm along the y-axis. The pore size distribution is found by determining for each pore the maximal radius of a sphere that fits inside. This method is adapted for samples showing relatively spherical porosities, homogeneous in shape, which is the case for most of our 3D printed samples. To capture the length of pores with “un-spherical” shapes and identify a possible anisotropy in pore shape, the “chord length” method is employed. It consists of drawing chords that span across each pore in a given direction; the appearance frequency of each chord length is extracted along x- and z-directions and compared, to detect large defects, in the deposition direction (xy-plane) or regarding interlayer adhesion (z-direction).

4. Results & discussion

The primary result to report is the successful manufacturing of samples 3D printed from the SS and RS powders, both on-ground and in weightlessness (see Fig. 14). The resulting parts, constituted of up to 15 layers deposited successively in weightlessness and up to 20 on-ground, maintain their shape, show homogeneous external appearance and smooth surface, without obvious defects, holes, nor heterogeneous powder repartition. Samples were put through further analysis to verify if this macroscopic assessment could be extended to the microscopic scale.

As a side remark, the images captured from under the printing area (below the solidification window) show that at each rise of the printing substrate, the entire consolidated layer (including its sintered and non-sintered regions) rises simultaneously, leaving the bottom space free for the next layer. For printing complex shapes, it is important that the homogeneity of the formerly deposited layers remains intact, as it allows to distribute compressive stresses throughout the former layers (where “former layers” comprises the already printed sample *and* the raw material situated in the non-solidified spaces). In our test case, where the 3D printed samples are of rectangular cross section and placed in the middle of a circular printing substrate, the rise of the entire consolidated powder layer is shown in Fig. 15: a difference image obtained from *in-situ* imaging just after (Fig. 15(b)) and just before (Fig. 15(a)) the rise of the inner cylinder, shows that the entire printing substrate can be identified (Fig. 15(c); yellow dashed circle), but the previously solidified rectangular shape cannot be distinguished (dashed blue line). The full video is available in supplementary material (video 2) [61]. The fact that the layer remains consistent during and after the substrate’s rise is attributed to the compression step, which consolidates the layer before solidification by sintering. This conclusion emphasizes the importance of the compression step.

4.1. Density of 3D printed samples

The average packing fraction of each sample is compared for all samples in Fig. 16 by calculating the percent area of material *versus* voids per slice, for each sample. Since the slices have a finite thickness that is much smaller than the typical particle size, the average over the different slices thus represents the volume fraction of material in the sample. This allows for verification of the quantity of the material that was effectively deposited. Typical XCT slices used for in-bulk

Table 4

Scanning parameters for X-ray computed tomography of sintered PS.

Computed tomography system	Source voltage	Output current	Projection per scan	Measurements per projection	Exposure time	Voxel size
CT-ALPHA (ProCon X-ray, Germany)	80 kV	70 μ A	1600	10	1000 ms	8 μ m

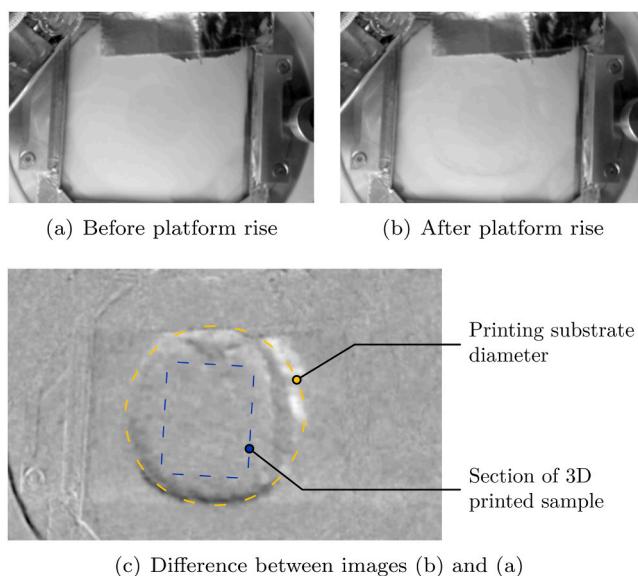


Fig. 15. Snapshots of *in-situ* monitoring by image capture from below the solidification window. The extract corresponds to a sample 3D printed from SS base granular material, in weightlessness (third day of PFC), for the 4th layer deposited at time: (a) just before the rise of the platform following the partial sintering of layer 3 and (b) after platform rise. Panel (c) shows the difference between (b) and (a); the dashed yellow circle indicates the approximate boundary of the printing substrate, and the dashed blue rectangle indicates the region corresponding to the previously solidified part of the layer (*i.e.* the zone where the lamp heating is strongest).

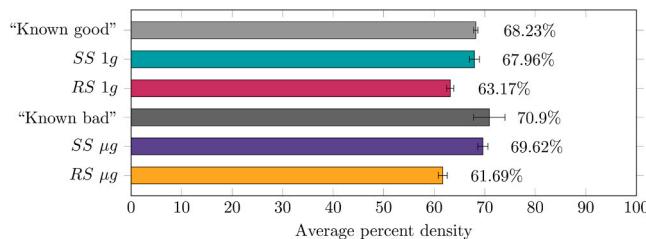


Fig. 16. Percent area of material *versus* voids per slice for each sample, representing the average density of the samples. Error bars represent the standard deviation per xz-slice percent area for all slices, in each sample.

characterization are shown in Fig. 17. Visual inspection of the XCT slices shows a rather homogeneous distribution of the porosities throughout the sample, for all but the "known bad" sample. The spatial homogeneity is also confirmed by the fact that the distribution of average densities calculated per slice is very narrow (low standard deviation indicated in Fig. 16 and discussed below). Bare-eye observation of the XCT images also shows that all porosities display a relatively spherical shape, with neither a preferred direction nor obvious signs of delamination between layers, for all samples but the "known bad" one. Notably, the 500 μ m-high layers cannot be distinguished with bare eye, although the samples each comprise multiple layers.

As the samples are sintered and not melted, the average density of a good quality 3D printed part is expected to be slightly higher than random close packing (rcp) for a monodispersed spheres packing, *i.e.* $\approx 64\%$

[60,82]. The average densities of the 3D printed samples shown in Fig. 16 are higher than rcp, showing that the powder is effectively deposited in the center of the printing bed. The "known good" sample sintered under compression and the SS samples 3D printed under 1g and μ g reach $\approx 70\%$ density, with standard deviations less than 1%. The RS samples show a density $\approx 5\%$ lower, regardless of g-level and of the fact that the same printing parameters are deployed for both types of feedstock material. Although higher porosity is reflected in the lower average density of the RS samples, very low standard deviations of 0.7% and 0.9% (respectively for 1g and μ g) show that mediocre flowability powder can be used as AM base-material without triggering major defects in the printed parts. Comparatively, the "known bad" sample shows a surprisingly high average density of $\approx 70\%$, but also high variability, with a standard deviation of approx. 3% (more than three times higher than for all other samples), as from one "slice" to the next the large porosities observed overtake most of the sample or are reduced to a minimal volume. This shows that regardless of the sample quality (unquestionably bad because of heterogeneous, large porosities), powder is indeed transported to the printing region: although the solidification energy was overestimated, which produced an unevenly melted sample, the powder deposition procedure functioned and brought the necessary quantity of material to the printing bed.

The average packing fractions depend on the base material, but are independent of gravity. This result is reminiscent of the printing-zone densities obtained through simulation (Figs. 6–8): the observed changes are minimal between 1g and μ g, but the RS powder is more difficult to deposit. Simulation predicted that the homogenization step would erase material-dependence, but experiments show that RS powder remains at lower density after homogenization and compression. This discrepancy might be due to simulated particles being smooth in essence, leading to lower reliability of our model for particles with roughened surface.

4.2. Influence of feedstock flowability on pore size

Fig. 18.

The pore size distribution is shown in Fig. 18. The XCT imaging resolution sets the minimum pore size detectable at 15 μ m. The comparison between the SS samples in Fig. 18(a) shows that all have most pores in the range of 15 μ m to 25 μ m. The sample sintered under weight ("known good" sample) has the largest amount of small pores (diameter 15 μ m), which shows, as expected, the best powder repartition and compaction. The 3D printed samples also show a peak at small pores; however for both material-qualities, a second peak appears at 30 μ m, increasing the mean pore size. Most importantly, large pores of 35 μ m to 60 μ m have a very low probability in the "known good" sample, and low for the SS 3D printed samples. Despite small differences, the three samples have mostly similar pore size distributions, and consistently show virtually no large pores of size $\geq 55 \mu$ m.

The RS samples in Fig. 18(b) show an obvious difference in pores size for the "known bad" sample: its pore diameter distribution has a much longer tail than all other samples, with a low probability density of small pores under 50 μ m and some very large pores up to 700 μ m. Apart from this outlier experiment, our RS results (Fig. 18(b)) in comparison to the SS results (Fig. 18(a)) confirm that a clear difference between base materials can be made: even if the distribution is relatively close, with two peaks at pore diameters 15 μ m and 30 μ m, for the smooth-surface samples the smaller pore size is almost twice more likely to appear than the smaller one, while for the RS, the two pore diameters are almost equally likely to appear. Fig. 18 confirms that the samples printed on

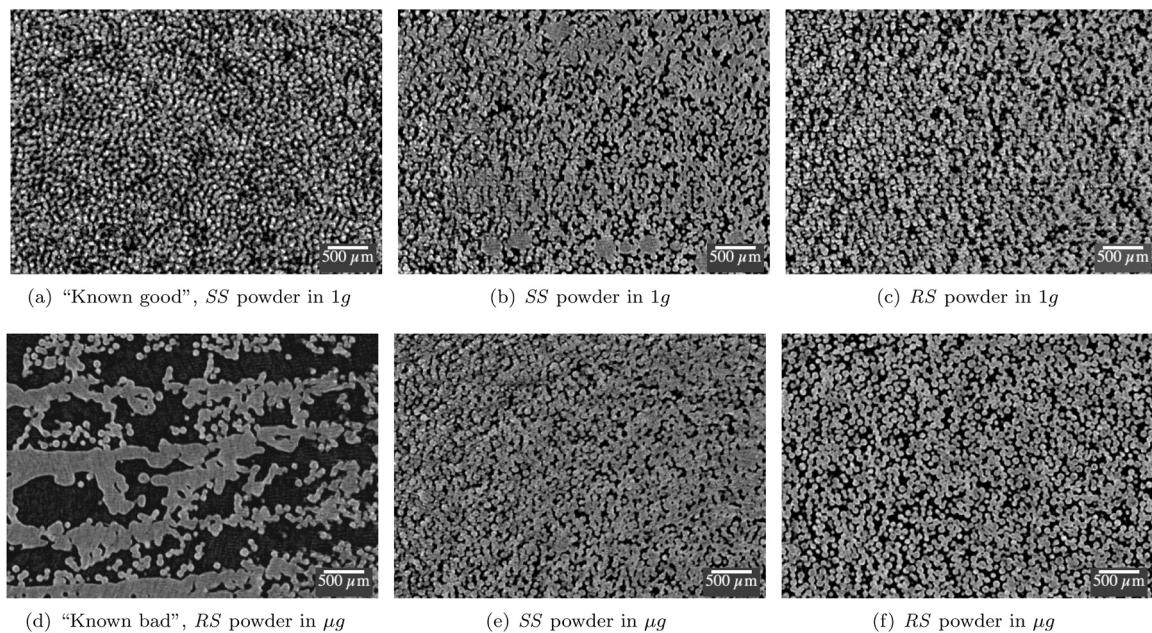


Fig. 17. Extracts from XCT slices of samples 3D printed in all situations studied. xz -plane is shown, x to the right, z to the top, with z being the height (direction orthogonal to the layers). (a) is the “known good” sample: SS powder sintered under weight on-ground. (b) is the SS powder and (c) the RS powder, both 3D printed in 1g. (d) is the sample labeled “known bad” (partially molten RS powder in weightlessness, with no compression during melting). (e) and (f) are respectively SS and RS powders 3D printed in weightlessness.

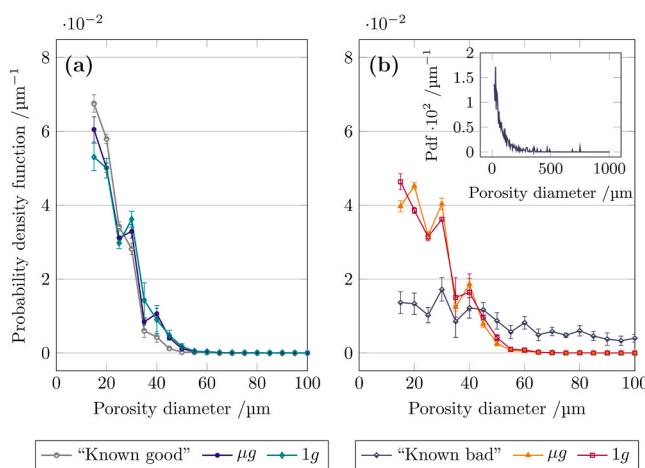


Fig. 18. Probability density function (pdf) per circle diameter fitted in each pore for (a) the “known good” sample, and the SS samples 3D printed under μg and 1 g; (b) the “known bad” sample, and RS samples 3D printed under μg and 1 g. Points represent the average over each full sample, and error bars the standard deviation in one sample. The inset in (b) shows the “known bad” sample on appropriate scale.

ground and in weightlessness from their respective base-materials show very similar pore size distributions: within experimental error, the gravitational environment in which the samples have been manufactured does not play a role in the quality of 3D printed samples.

The pore size distribution corresponding to the RS powder has a larger width than that corresponding to the SS powder (Fig. 18). Hence, the RS sample has a higher fraction of larger pores than the SS sample, showing that its lower average density (Fig. 16) is the result of generally larger pores, similarly well scattered throughout the samples (from visual inspection of the XCT slices). Such larger porosities are linked to the decreased packing efficiency of materials exhibiting increased surface roughness: as the stress needed for particles to slide on each other is increased by surface roughness [83], under the same external stress

input, the packing reorganizes into a less dense configuration [31]. The maximum packing density for each specific type of powder is effectively reached in our experiments, in absence of other means implemented to increase material density (e.g. compression during sintering).

4.3. Gravity-dependent anisotropy

Chord length analysis is used to capture anisotropy in the pores’ shape and test whether the manufacturing process begets a preferred direction; it is presented in Fig. 19 for the x - and z -directions. To allow precise comparison of the different samples, each data set is fitted with a log-normal distribution. This distribution is plausible because the chord lengths are not independent: each pore that contributes to chord length L , also contributes to all smaller chord lengths, so that the addition of a long chord rescales the entire distribution at smaller chord lengths. Assuming that pore sizes are independent from each other and randomly distributed, the central limit theorem hence suggests a log-normal distribution (since the multiplicative increments become independent random additive increments of the logarithm). Previous work involving the distribution of geometrical shapes enclosed within randomly distributed voids also found a log-normal distribution [84]. The fitting parameters, μ_x, z and $\sigma_{x, z}$, corresponding to the mean and standard deviation of the logarithm of the data, are presented for each sample in x - and z -directions in Table 5. The mean chord length $\langle l_{x, z} \rangle = \exp(\mu_{x, z} + \sigma_{x, z}^2/2)$ is also given for each fit to ease interpretation.

For the “known good” sample in Fig. 19(a), without surprise the mean chord lengths are the lowest of all experiments. They are also very close in size along x - and z -directions, with $\langle l_z \rangle$ larger than $\langle l_x \rangle$ by only 4 μm . The fit for this dataset (x -dir.) is shown on all panels of Fig. 19 (solid black line) to provide a comparative baseline.

Comparison of the 3D printed samples to this baseline confirms that all samples have slightly larger pores than our reference sample. The comparative sample is sintered continuously under weight, applying a constant pressure to allow porosities to close during sintering, while the 3D printed samples are all compressed to a fixed height rather than under a constant pressure: the compression is not maintained constant during sintering. This is necessary for assessing the powder repartition,

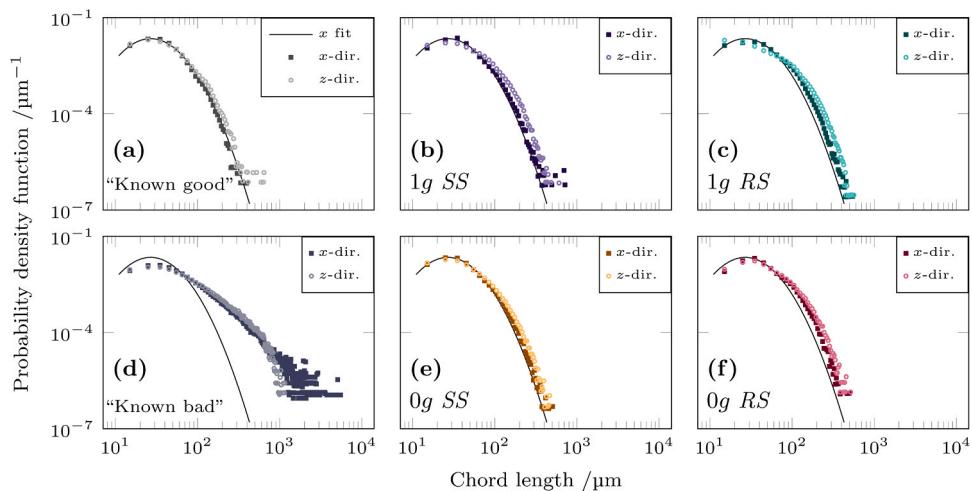


Fig. 19. Relative frequency of chord spanning porosities per chord length, for all samples studied, with chords extension done along x - and z -directions for each sample. The “known good” sample data along the x -axis is fitted to a log-normal distribution (solid black line), reproduced on all graphs for comparison.

Table 5

Fitting parameters used to fit the chord length probability density obtained for each sample in x - and y -directions to the log-normal distribution, $\mu_{x, z}$ and $\sigma_{x, z}$, and mean chord length $\langle l_{x, z} \rangle$, given in μm . The ratio of standard deviations, $\frac{\sigma_z}{\sigma_x}$, is also given for reference.

Sample reference	μ_x	σ_x	$\langle l_x \rangle$	μ_z	σ_z	$\langle l_z \rangle$	$\frac{\sigma_z}{\sigma_x}$
“Known good”	3.6	0.57	44	3.7	0.63	48	1.105
Smooth surface, 1g	3.7	0.57	48	3.8	0.65	57	1.140
Rough surface, 1g	3.9	0.54	56	3.9	0.67	60	1.241
“Known bad”	3.9	1.1	92	4.1	1.1	110	1
Smooth surface, μg	3.7	0.58	47	3.7	0.64	51	1.103
Rough surface, μg	3.8	0.59	56	3.9	0.67	60	1.136

but a constant compression pressure should be maintained during the solidification step in further manufacturing campaigns to decrease the size of porosities, thereby increasing the prints’ quality.

The “known bad” sample’s chord length presented in Fig. 19(d) is notable. The distribution shows a long tail at large chord lengths $l_{x,y} \geq 10^3 \mu\text{m}$ in x -direction, which does not appear along z -direction, indicating the long horizontal porosities due to delamination between layers, clearly visible in Fig. 17(d). This shows an exemplary anisotropic sample with preferential direction of pore growth along the x -direction, populated by many elongated porosities of length $l_x \geq 10^3 \mu\text{m}$.

The μg SS sample has the highest isotropy of all 3D printed samples. The μg RS sample also exhibits high isotropy, despite slightly larger porosities. Generally, the raw material’s flowability (SS or RS) does not beget anisotropy in the printed samples.

Comparing the 3D printed samples by base-material depending on the g -level during manufacturing, a slight elongation of the pores in the z -direction is remarked for the 1g samples. Precisely, σ_z/σ_x is always larger for the 1g samples than for the corresponding μg samples (see Table 5), showing that 1g samples have a stronger anisotropy than μg samples. To understand the origin if this divergence, we look at the homogenization step of the 3D printing process.

In microgravity, the homogenization step (horizontal shaking of the powder at the bottom of the apparatus), results in a wave of powder forming “clusters” or zones of heterogeneous density: bubble-like circular structures progressing towards the center of the printing area. The homogenization step of the process consists of merging those “powder clusters” into a homogeneous powder layer. This can be seen in video 2 of the supplementary material [61]. On-ground, the same wave-like progression is observed, although the circular structures within the powder are not observed.

In absence of the preferred acceleration direction due to gravitational acceleration, the “clusters” that form are composed of particles in configurations that also do not have a preferred direction, which results in an isotropic packing. Those clusters then merge into a powder layer; as this still happens in absence of gravity, the particles are not reordered in a denser packing due to their respective weight, but retain the isotropic configuration they had in the clusters. In turn, this results in porosities with isotropic shapes for the microgravity-3D printed samples, while the ground-3D printed samples have reorganized under their own weight into a less isotropic packing.

Microscopies of cuts of RS samples that were 3D printed under 1g and μg respectively, are presented in Figs. 20(a) and 20(b). Although partial crystallization is visible on both samples, on the sample that was 3D printed under 1g, large crystallized areas are visible. For both samples, the crystalline ordering is observed in the xy -plane, in which the horizontal shaking takes place.

Under 1g particles tend to crystallize layer-wise in the xy -plane. Such self-ordering has been shown previously [85]; it results in a superposition of high-density, crystallized grain-like regions, surrounded by lower density boundaries (as visible in Fig. 20(a)). As this phenomenon occurs along the shaking direction in the xy -plane, it creates voids elongated in the z -direction, and explains the mild anisotropy observed in samples manufactured under 1g.

The minute elongation of porosities along the z -axis in the ground-manufactured samples is generally compliant with the presence of a preferred direction under gravity, and partial crystallization is observed in the horizontal shaking plane, in particular in the 1g samples (Fig. 20). The pores’ chord length distributions are very similar to the comparative “known good” samples: all 3D printed samples show high isotropy (Fig. 19 and Table 5). The decrease in density of the RS samples is linked to an increase in pore size, but not accompanied by the appearance of defects in the deposition. The quantity of material brought to the printing area, as well as the homogenization time (representing the amount of material brought down and then to the printing-bed center) are hence deemed sufficient.

5. Conclusion and outlook

To be used for in-space applications, AM methods will need to evolve into more versatile technologies with increased reliability [5,10]. We proposed an AM process that enables part production from powder independently of the gravitational environment and of material flowability. This process emancipates from the current limitations on granular feedstock, as it does not rely on highly flowable powder for material

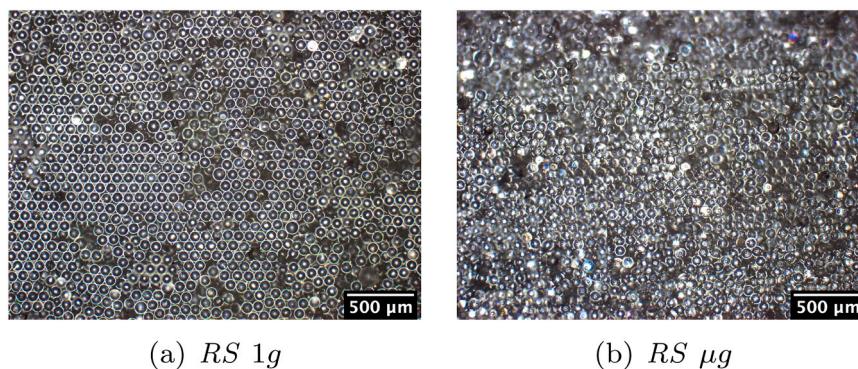


Fig. 20. Bright field optical microscopies of sections cut of the 3D printed samples along the xy-direction, for samples manufactured from RS powder, (a) under 1g and (b) in μ g.

deposition. Besides, it places no further geometrical constraints compared to ground-based PBF, while remaining superior to extrusion-based processes by allowing a wider range of materials. Tested through DEM simulation and on parabolic flights, a proof of concept was provided on-ground ($1g$) and in weightlessness (μg), using as feedstock material a $80\text{ }\mu\text{m}$ -diameter PS powder modified to obtain a good flowability powder (*Smooth Surface, SS*) and a low flowability powder (*Rough Surface, RS*). The *RS* powder was shown to have lower flowability than the typical 3D printing metal powder Ti-64. Analysis of samples that were 3D printed under gravity and in absence thereof shows that the powder deposition is realized equally efficiently under both g -levels. High reproducibility is found between samples manufactured from the same base-material in different gravitational environments, with a homogeneous pore size distribution and isotropy of the samples. The ground-printed samples show slightly higher anisotropy, which we attribute to a layer-wise crystallization in our highly monodisperse powders. A difference between samples realized with each base materials persists, which is attributed to the lower ability to pack densely for *RS* powder. Delamination is observed in none of the 3D printed samples.

All the samples analyzed in-bulk (*SS* and *RS*, under 1 g and μ g) were manufactured using the same printing parameters. The mild differences obtained in material deposition, show that the use of material with different flowability is reflected in the process; yet all powders could be deposited and 3D printed. This suggests that the *RS* powder could be more densely packed by using better suited printing parameters. In particular, while the deposition process might be ineffective in increasing packing density if that is a material-dependent variable [31], this difference could be amended *during* consolidation. Ideally, the compression ratio could be increased as the consolidation is taking place, by maintaining a constant vertical pressure, triggering degassing and thereby increasing part's density, akin to hot isostatic pressing. This simple amelioration would allow for the drastic reduction in the samples' porosity, and in turn enable the control of printed parts density (including density gradients throughout the additively manufactured part).

The possibility to compress the material layer after deposition also has a specific drawback: it implies that the material of the solidification window and the printing material are in contact before and most importantly during consolidation. For the polymer powders used as model substances in the work presented here, the quartz glass plate was not reactive at the sintering temperature of polystyrene (~ 200 °C). However, to 3D print metal or ceramic powders would require significant modifications of the system: if consolidation happens at higher temperature, material exchange between the molten powder and the glass plate could result in the newly solidified layer sticking irreversibly to the solidification window. The production of metal and ceramic parts would hence require not only to adapt the energy source to provide accordingly higher energy, but also a new study of affinity between the base-material and the window's material. The material of the solidification window

would have to be chosen accordingly, to minimize material exchange and avoid the powder layer remaining stuck to the window. For example, the stability of a sapphire glass plate should be investigated when exposed repeatedly to molten metal alloys. Ensuring that no heat-induced chemical reaction happens will be the challenge to adapt this AM process to metal and ceramic powders.

Besides controlling parts' density by compressing the newly deposited powder layer, defect appearance is mitigated by constant *in-situ* monitoring during powder deposition. The next step will be to automatize the control loops (torque sensing during powder transport and image analysis during layer homogenization). On the one hand, homogenization time could be optimized through the image analysis procedure proposed, to minimize fabrication time. On the other hand, assessment of print quality and live-correction during manufacturing will allow AM to access a wider range of applications by increasing stability in prints quality, including application for space and ISM.

Using those strategies to ensure high printing quality without requirement on the rheology of the feedstock material will facilitate the use of recycled materials for AM. First tests on-ground have shown that powder produced by closed-loop recycling (*i.e.* by grinding former 3D printed parts) can be directly used as raw material in the process presented here [66]. The possibility to not only reuse material from previous batches, but also recycle former objects into new feedstock, would drastically reduce costs associated with AM, on Earth as well as in space.

Focusing on the powder handling aspect, the powder deposition process was developed to allow for the use of powders regardless of their physical and rheological properties, meaning that it functions for *any* base-material (polymers, metals, ceramics...). The powder handling method could hence be adapted for granular transport even beyond 3D printing in reduced gravity environments, regardless of the material's flowability. Notably, on sand-covered planetary surface (*e.g.* the Moon, Mars or certain asteroids), powder handling technologies will be necessary to process regolith, the main *in-situ* resource and a powder of notoriously poor flowability [86].

As technological progress and space explorations will go hand-in-hand in the coming years, the authors hope that the AM process presented will be part of a movement to spur the development of in-space manufacturing in general, ultimately enabling long-term human presence in space.

CRediT authorship contribution statement

Olfa D'Angelo: Conceptualization, Investigation, Methodology, Data curation, Formal analysis, Funding acquisition and Project administration, Writing – original draft. **Felix Kuthe:** Investigation, Methodology, Data curation. **Szu-Jia Liu:** Investigation, Data curation. **Raphael Wiedey:** Resources. **Joe M. Bennett:** Resources. **Martina Meisnar:** Resources. **Andrew Barnes:** Resources. **W. Till Kranz:** Supervision, Writing – review & editing. **Thomas Voigtmann:**

Methodology, Data curation, Validation, Supervision, Writing – review & editing. **Andreas Meyer:** Funding acquisition, Project administration, Resources, Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.addma.2021.102349](https://doi.org/10.1016/j.addma.2021.102349).

REFERENCES

- [1] R.S. Skomorohov, C. Welch, A.M. Hein, In-orbit spacecraft manufacturing: Near-term business cases, Tech. rep., International Space University / Initiative for Interstellar Studies, 2016.
- [2] A. Owens, O.D. Weck, C. Stromgren, K.E. Goodliff, W. Cirillo, Supportability challenges, metrics, and key decisions for human spaceflight, Proc. AIAA SPACE Astronaut. Forum Expo., Orlando, FL, USA (2017), <https://doi.org/10.2514/6.2017-5124>.
- [3] National Research Council of the National Academies, *3D Printing in Space*, The National Academies Press, Washington, DC, 2014.
- [4] T. Prater, C. Hill, F. Ledbetter, M. Fiske, J. Edmunsson, M. Meyyappan, P. Hall, C. Roberts, L.D. Huebner, N. Werkheiser, NASA's in-space manufacturing project: Update on manufacturing technologies and materials to enable more sustainable and safer exploration (Washington DC, USA), Proc. 70th IAC (2019) (Washington DC, USA).
- [5] A.E. Trujillo, M.T. Moraguez, S.I. Wald, A.C. Owens, O.L. de Weck, Feasibility analysis of commercial in-space manufacturing applications (Orlando, FL, USA), Proc. AIAA Space.: Space. Astronaut. Forum Expo. (2017), <https://doi.org/10.2514/6.2017-5360>.
- [6] T. Prater, Q. Bean, R. Beshears, T. Rolin, N. Werkheiser, E. Ordonez, R. Ryan, F. Ledbetter III, Summary report on phase I and phase II results from the 3D printing in zero-g technology demonstration mission, volume I, Tech. rep., Marshall Space Flight Center, Huntsville, Alabama, 2016.
- [7] T. Prater, N. Werkheiser, F. Ledbetter, D. Timucin, K. Wheeler, M. Snyder, 3D printing in zero g technology demonstration mission: complete experimental results and summary of related material modeling efforts, Int. J. Adv. Manuf. Technol. 101 (2018) 391–417, <https://doi.org/10.1007/s00170-018-2827-7>.
- [8] M.A. Gibson, N.M. Mykulowycz, J. Shim, R. Fontana, P. Schmitt, A. Roberts, J. Ketkaew, L. Shao, W. Chen, P. Bordeenthikasem, J.S. Myerberg, R. Fulop, M. D. Verminski, E.M. Sachs, Y.-M. Chiang, C.A. Schuh, A. JohnHart, J. Schroers, 3D printing metals like thermoplastics: fused filament fabrication of metallic glasses, Mater. Today 21 (2018) 697–702.
- [9] G.D. Goh, Y.L. Yap, H.K.J. Tan, S.L. Sing, G.L. Goh, W.Y. Yeong, Process-structure-properties in polymer additive manufacturing via material extrusion: a review, Crit. Rev. Solid State Mater. Sci. 45 (2) (2020) 113–133, <https://doi.org/10.1080/10408436.2018.1549977>.
- [10] T. DebRoy, H.L. Wei, J.S. Zuback, T. Mukherjee, J.W. Elmer, J.O. Milewski, A. M. Beese, A. Wilson-Heid, A. De, W. Zhang, Additive manufacturing of metallic components - Process, structure and properties, Prog. Mater. Sci. 92 (2018) 112–224, <https://doi.org/10.1016/j.pmatsci.2017.10.001>.
- [11] M. Schmid, K. Wegener, Additive manufacturing: Polymers applicable for laser sintering (LS), in: International Conference on Manufacturing Engineering and Materials (ICMEM), vol. 149, Nový Smokovec, Slovakia, 2016, 457–464.<https://doi.org/10.1016/j.proeng.2016.06.692>.
- [12] C.Y. Yap, C.K. Chua, Z.L. Dong, Z.H. Liu, D.Q. Zhang, L.E. Loh, S.L. Sing, Review of selective laser melting: materials and applications, Appl. Phys. Rev. 2 (2015), 041101, <https://doi.org/10.1063/1.4935926>.
- [13] S.L. Sing, W.Y. Yeong, F.E. Wiria, B.Y. Tay, Z. Zhao, L. Zhao, Z. Tian, S. Yang, Direct selective laser sintering and melting of ceramics: a review, Rapid Prototyp. J. 23 (3) (2017) 611–623, <https://doi.org/10.1108/RPJ-11-2015-0178>.
- [14] J. Blum, G. Wurm, S. Kempf, T. Poppe, H. Klahr, T. Koza, M. Rott, T. Henning, J. Dorschner, R. Schräpler, H.U. Keller, W.J. Markiewicz, I. Mann, B.A. Gustafson, F. Giovane, D. Neuhäus, H. Fechtig, E. Grün, B. Feuerbacher, H. Kochan, L. Ratke, A. ElGoresy, G. Morfill, S.J. Weidenschilling, G. Schwem, K. Metzler, W.H. Ip, Growth and form of planetary sediments: results from a microgravity aggregation experiment, Phys. Rev. Lett. 85 (12) (2000) 2426–2429, <https://doi.org/10.1103/PhysRevLett.85.2426>.
- [15] R. Weidling, C. Güttler, J. Blum, Free collisions in a microgravity many-particle experiment. I. Dust aggregate sticking at low velocities, Icarus 218 (1) (2012) 688–700, <https://doi.org/10.1016/j.icarus.2011.10.002>.
- [16] S.G. Love, D.R. Petit, S.R. Messenger, Particle aggregation in microgravity: Informal experiments on the international space station, Meteorit. Planet. Sci. 49 (2014) 732–739, <https://doi.org/10.1111/maps.12286>.
- [17] R.A. Wilkinson, R.P. Behringer, J.T. Jenkins, M.Y. Louge, Granular materials and the risks they pose for success on the moon and mars, AIP Conf. Proc. 746 (2005) 1216–1223, <https://doi.org/10.1063/1.1867248>.
- [18] O. D'Angelo, T. Voigtmann, Rheology of dense granular flows in microgravity: A Review, unpublished manuscript (2021).
- [19] J. Günster, A. Zocca, C.M. Gomes, T. Muehler, Method for Stabilizing a Powder Bed by means of Vacuum for Additive Manufacturing, United States Patent 9533452 B2, 2017.
- [20] A. Zocca, C.M. Gomes, T. Mühler, J. Günster, Powder-bed stabilization for powder-based additive manufacturing, Adv. Mech. Eng. 2014 (2014), 491581, <https://doi.org/10.1155/2014/491581>.
- [21] A. Zocca, J. Lüchtenborg, T. Mühler, J. Wilbig, G. Mohr, T. Villatte, F. Léonard, G. Nolze, M. Sparenberg, J. Melcher, K. Hilgenberg, J. Günster, Enabling the 3D printing of metal components in μ -gravity, Adv. Mater. Technol. 4 (2019), 1900506, <https://doi.org/10.1002/admt.201900506>.
- [22] S. Vock, B. Klöden, A. Kirchner, T. Weißgärtner, B. Kieback, Powders for powder bed fusion: a review, Prog. Addit. Manuf. 4 (4) (2019) 383–397, <https://doi.org/10.1007/s40964-019-00078-6>.
- [23] V. Seyda, D. Herzog, C. Emmelmann, Relationship between powder characteristics and part properties in laser beam melting of ti-6al-4v, and implications on quality, J. Laser Appl. 29 (2) (2017), 022311, <https://doi.org/10.2351/1.4983240>.
- [24] P. Kiani, U. ScipioniBertoli, A.D. Dupuy, K. Ma, J.M. Schoenung, A statistical analysis of powder flowability in metal additive manufacturing, Adv. Eng. Mater. 22 (10) (2020), 2000022, <https://doi.org/10.1002/adem.202000022>.
- [25] H.M. Jaeger, S.R. Nagel, R.P. Behringer, Granular solids, liquids, and gases, Rev. Mod. Phys. 68 (4) (1996) 1259–1273, 0034-6861/96/68.4/1259.15.
- [26] Y. Forterre, O. Pouliquen, Flows of dense granular media, Annu. Rev. Fluid Mech. 40 (2008) 1–24, <https://doi.org/10.1146/annurev.fluid.40.111406.102142>.
- [27] I.S. Aranson, L.S. Tsimring, Patterns and collective behavior in granular media: theoretical concepts, Rev. Mod. Phys. 78 (2006) 641–692, <https://doi.org/10.1103/RevModPhys.78.641>.
- [28] H.A. Janssen, Versuche über Getreide druck in Silozellen [On grain pressure in silos], Z. Des. Ver. Dtsch. Ing. 39 (35) (1895) 1045–1049.
- [29] C.S. O'Hern, L.E. Silbert, A.J. Liu, S.R. Nagel, Jamming at zero temperature and zero applied stress: the epitome of disorder, Phys. Rev. E 68 (2003), 011306, <https://doi.org/10.1103/PhysRevE.68.011306>.
- [30] M.E. Cates, J.P. Wittmer, J.P. Bouchaud, P. Claudin, Jamming and stress propagation in particulate matter, Phys. A 263 (1999) 354–361, [https://doi.org/10.1016/s0378-4371\(98\)00491-9](https://doi.org/10.1016/s0378-4371(98)00491-9).
- [31] R. Mari, R. Seto, J.F. Morris, M.M. Denn, Shear thickening, frictionless and frictional rheologies in non-Brownian suspensions, J. Rheol. 58 (2014) 1693, <https://doi.org/10.1122/1.4890747>.
- [32] O. D'Angelo, A. Horb, A. Cowley, M. Sperl, W.T. Kranz, Granular piston-probing in microgravity: powder compression, from densification to jamming, unpublished manuscript, 2021.
- [33] N. Murdoch, B. Rozitis, S.F. Green, T.L. De Lophem, P. Michel, W. Losert, Granular shear flow in varying gravitational environments, Granul. Matter 15 (2) (2013) 129–137, <https://doi.org/10.1007/s10035-013-0395-y>.
- [34] E. Opsomer, F. Ludewig, N. Vandewalle, Dynamical clustering in driven granular gas, EPL 99 (2012) 40001, <https://doi.org/10.1209/0295-5075/99/40001>.
- [35] N. Murdoch, B. Rozitis, K. Nordstrom, S.F. Green, P. Michel, T.L. De Lophem, W. Losert, Granular convection in microgravity, Phys. Rev. Lett. 110 (2013), 018307, <https://doi.org/10.1103/PhysRevLett.110.018307>.
- [36] R. Seto, A. Singh, B. Chakraborty, M.M. Denn, J.F. Morris, Shear jamming and fragility in dense suspensions, Granul. Matter 21 (3) (2019) 82, <https://doi.org/10.1007/s10035-019-0931-5>.

- [37] K. Mair, J.F. Hazzard, Nature of stress accommodation in sheared granular material: insights from 3D numerical modeling, *Earth Planet. Sci. Lett.* 259 (3–4) (2007) 469–485, <https://doi.org/10.1016/j.epsl.2007.05.006>.
- [38] R. Niu, M. Ramaswamy, C. Ness, A. Shetty, I. Cohen, Tunable solidification of cornstarch under impact: How to make someone walking on cornstarch sink, *Sci. Adv.* 6 (2020) 19, <https://doi.org/10.1126/sciadv.aay6661>.
- [39] S.F. Edwards, R.B. Oakeshott, Theory of powders, *Physica A* 157 (3) (1989) 1080–1090, [https://doi.org/10.1016/0378-4378\(89\)90034-4](https://doi.org/10.1016/0378-4378(89)90034-4).
- [40] S. Nadler, O. Bonnefoy, J.M. Chaix, G. Thomas, J.L. Gelet, Parametric study of horizontally vibrated grain packings, *Eur. Phys. J. E* 34 (2011) 66, <https://doi.org/10.1140/epje/i2011-11066-y>.
- [41] S.M. Khefif, A. Valance, F. Ould-Kaddour, Spreading of a granular droplet under horizontal vibrations, *Phys. Rev. E* 97 (2018), 062903, <https://doi.org/10.1103/PhysRevE.97.062903>.
- [42] E. Opsomer, F. Ludewig, N. Vandewalle, Phase transitions in vibrated granular systems in microgravity, *Phys. Rev. E* 84 (2011), 051306, <https://doi.org/10.1103/PhysRevE.84.051306>.
- [43] M. Noirhomme, E. Opsomer, N. Vandewalle, F. Ludewig, Granular transport in driven granular gas, *Eur. Phys. J. E* 38 (2015) 9, <https://doi.org/10.1140/epje/i2015-15009-4>.
- [44] A. Kudrolli, M. Wolpert, J.P. Gollub, Cluster formation due to collisions in granular material, *Phys. Rev. Lett.* 78 (1997) 1383–1386, <https://doi.org/10.1103/PhysRevLett.78.1383>.
- [45] E. Falcon, R. Wunenburger, P. Evesque, S. Fauve, C. Chabot, Y. Garrabos, D. Beysens, Cluster formation in a granular medium fluidized by vibrations in low gravity, *Phys. Rev. Lett.* 83 (2) (1999) 440–443, <https://doi.org/10.1103/PhysRevLett.83.440>.
- [46] E. Opsomer, M. Noirhomme, N. Vandewalle, E. Falcon, S. Merminod, Segregation and pattern formation in dilute granular media under microgravity conditions, *NPJ Microgravity* 3 (2017) 1, <https://doi.org/10.1038/s41526-016-0009-1>.
- [47] G. Tapia, A. Elwany, A review on process monitoring and control in metal-based additive manufacturing, *J. Manuf. Sci. Eng.* 136 (2014), 060801, <https://doi.org/10.1115/1.4028540>.
- [48] M. Grasso, B.M. Colosimo, Process defects and in situ monitoring methods in metal powder bed fusion: a review, *Meas. Sci. Technol.* 28 (4) (2017), 044005, <https://doi.org/10.1088/1361-6501/a5c4f>.
- [49] S.K. Everton, M. Hirsch, P.I. Stavroulakis, R.K. Leach, A.T. Clare, Review of *in-situ* process monitoring and *in-situ* metrology for metal additive manufacturing, *Mater. Des.* 95 (2016) 431–445, <https://doi.org/10.1016/j.matdes.2016.01.099>.
- [50] S. Luding, Molecular dynamics simulations of granular materials, in: H. Hinrichsen, D.E. Wolf (Eds.), *The Physics of Granular Media*, John Wiley & Sons, Ltd, 2005, pp. 297–324, <https://doi.org/10.1002/352760362X.ch13>.
- [51] C. Kloss, C. Goniva, A. Hager, S. Amberger, S. Pirker, Models, algorithms and validation for opensource dem and cfd-dem, *Prog. Comput. Fluid Dyn.* 12 (2012) 140–152, <https://doi.org/10.1504/PCFD.2012.047457>.
- [52] H. Hertz, Über die Berührung fester elastischer Körper, *J. Reine Angew. Math.* 92 (1882) 156–171.
- [53] R. Mindlin, Compliance of elastic bodies in contact, *J. Appl. Mech.* 16 (1949) 259–268.
- [54] R. Mindlin, H. Deresiewicz, Elastic spheres in contact under varying oblique forces, *J. Appl. Mech.* 20 (1953) 327–344.
- [55] S. Lommen, D. Schott, G. Lodewijks, DEM speedup: stiffness effects on behavior of bulk material, *Particuology* 12 (2014) 107–112, <https://doi.org/10.1016/j.partic.2013.03.006>.
- [56] D. Vescovi, S. Luding, Merging fluid and solid granular behavior, *Soft Matter* 12 (2016) 8616–8628, <https://doi.org/10.1039/C6SM01444E>.
- [57] K.F. Malone, B.H. Xu, Determination of contact parameters for discrete element method simulations of granular systems, *Particuology* 6 (2008) 521–528, <https://doi.org/10.1016/j.partic.2008.07.012>.
- [58] J. Ai, J.-F. Chen, J.M. Rotter, J.Y. Ooiv, Assessment of rolling resistance models in discrete element simulations, *Powder Technol.* 206 (2011) 269–282, <https://doi.org/10.1016/j.powtec.2010.09.030>.
- [59] K.L. Johnson, K. Kendall, A.D. Roberts, D. Tabor, Surface energy and the contact of elastic solids, *Proc. R. Soc. Lond. A* 324 (1971) 301–313, <https://doi.org/10.1098/rspa.1971.0141>.
- [60] C.S. O'Hern, S.A. Langer, A.J. Liu, S.R. Nagel, Random packings of frictionless particles, *Phys. Rev. Lett.* 88 (2002), 075507, <https://doi.org/10.1103/PhysRevLett.88.075507>.
- [61] Supplementary Material, available online at tbd.
- [62] G. Voronoi, Nouvelles applications des paramètres continus à la théorie des formes quadratiques. deuxième mémoire. recherches sur les paralléloédres primitifs, *J. Angew. Math.* 1908 (134) (1908) 198–287, <https://doi.org/10.1515/crll.1908.134.198>.
- [63] F.M. Schaller, S.C. Kapfer, J.E. Hilton, P.W. Cleary, K. Mecke, C.D. Michele, T. Schilling, M. Saadatfar, M. Schröter, G.W. Delaney, G.E. Schröder-Turk, Non-universal voronoi cell shapes in amorphous ellipsoid packs, *EPL (Europhys. Lett.)* 111 (2015) 24002, <https://doi.org/10.1209/0295-5075/111/24002>.
- [64] P. Virtanen, R. Gommers, T.E. Oliphant, M. Haberland, T. Reddy, D. Cournapeau, E. Burovski, P. Peterson, W. Weckesser, J. Bright, S.J. van der Walt, M. Brett, J. Wilson, K.J. Millman, N. Mayorov, A.R.J. Nelson, E. Jones, R. Kern, E. Larson, C. Carey, İ. Polat, Y. Feng, E.W. Moore, J. VanderPlas, D. Laxalde, J. Perktold, R. Cimrman, I. Henriksen, E.A. Quintero, C.R. Harris, A.M. Archibald, A.H. Ribeiro, F. Pedregosa, P. van Mulbregt, A. Vijaykumar, A.P. Bardelli, A. Rothberg, A. Hüboll, A. Kloeckner, A. Scopatz, A. Lee, A. Rokem, C.N. Woods, C. Fulton, C. Masson, C. Häggström, C. Fitzgerald, D.A. Nicholson, D.R. Hagen, D. V. Pasechnik, E. Olivetti, E. Martin, E. Wieser, F. Silva, F. Lenders, F. Wilhelm, G. Young, G.A. Price, G.-L. Ingold, G.E. Allen, G.R. Lee, H. Audren, I. Probst, J. P. Dietrich, J. Silterra, J.T. Webber, J. Slavice, J. Nothman, J. Buchner, J. Kulick, J. Schönberger, J. de Miranda Cardoso, J. Reimer, J. Harrington, J.L.C. Rodríguez, J. Nunez-Iglesias, J. Kuczynski, K. Tritz, M. Thoma, M. Newville, M. Kümmeler, M. Bolingbroke, M. Tartre, M. Pak, N.J. Smith, N. Nowaczyk, N. Shebanov, O. Pavlyk, P.A. Brodtkorb, P. Lee, R.T. McGibbon, R. Feldbauer, S. Lewis, S. Tygier, S. Sievert, S. Vigna, S. Peterson, S. More, T. Pudlik, T. Oshima, T.J. Pingel, T. Robitaille, T. Spura, T.R. Jones, T. Cera, T. Leslie, T. Zito, T. Krauss, U. Upadhyay, Y.O. Halchenko, Y. Vázquez-Baeza, SciPy 1.0 Contributors, SciPy 1.0: fundamental algorithms for scientific computing in python, *Nat. Methods* 17 (3) (2020) 261–272, <https://doi.org/10.1038/s41592-019-0686-2>.
- [65] C.H. Rycroft, Voro: A three-dimensional voronoi cell library in c, *Chaos: an Interdisciplinary Journal of Nonlinear Science* 19 (2009), 041111, <https://doi.org/10.1063/1.3215722>.
- [66] O. Lopez-D'Angelo, Powder-based additive manufacturing for space - From granular rheology in varying gravitational environment to the development of a gravity-independent powder handling method, Ph.D. thesis, RWTH Aachen University, Aachen, Germany, 2021.
- [67] J. Yang, A. Sliva, A. Banerjee, R.N. Dave, R. Pfeffer, Dry particle coating for improving the flowability of cohesive powders, *Powder Technol.* 158 (2005) 21–33, <https://doi.org/10.1016/j.powtec.2005.04.032>.
- [68] A. Castellanos, The relationship between attractive interparticle forces and bulk behaviour in dry and uncharged fine powders, *Adv. Phys.* 54 (2005) 263–376, <https://doi.org/10.1080/17461390500402657>.
- [69] F. Fulchini, U. Zafar, C. Hare, M. Ghadiri, H. Tantawy, H. Ahmadian, M. Poletto, Relationship between surface area coverage of flow-aids and flowability of cohesive particles, *Powder Technol.* 322 (2017) 417–427, <https://doi.org/10.1016/j.powtec.2017.09.013>.
- [70] R.G. Larson, *The Structure and Rheology of Complex Fluids*, Oxford University Press, Oxford, 1998.
- [71] M. Leturia, B. Mohammed, S. Lagarde, I. Ronga, K. Saleh, Characterization of flow properties of cohesive powders: a comparative study of traditional and new testing methods, *Powder Technol.* 253 (2014) 406–423, <https://doi.org/10.1016/j.powtec.2013.11.045>.
- [72] H.-J. Yang, F.-Q. Wei, K.-H. Hu, G.-D. Zhou, J. Lyu, Comparison of rheometric devices for measuring the rheological parameters of debris flow slurry, *J. Mt. Sci.* 12 (5) (2015) 1125–1134, <https://doi.org/10.1007/s11629-015-3543-5>.
- [73] W. Nan, V. Vivacqua, M. Ghadiri, Y. Wang, Numerical analysis of air effect on the powder flow dynamics in the FT4 powder rheometer, *EPJ Web Conf.* 140 (2017) 03036, <https://doi.org/10.1051/epjconf/201714003036>.
- [74] W. Nan, M. Ghadiri, Y. Wang, Analysis of powder rheometry of FT4: effect of particle shape, *Chem. Eng. Sci.* 173 (2017) 374–383, <https://doi.org/10.1016/j.ces.2017.08.004>.
- [75] S. Liu, Y.C. Shin, Additive manufacturing of ti6al4v alloy: a review, *Mater. Des.* 164 (2019), 107552, <https://doi.org/10.1016/j.matdes.2018.107552>.
- [76] I. Zimmermann, M. Eber, K. Meyer, Nanomaterials as flow regulators in dry powders, *Z. Phys. Chem.* 218 (2004) 51–102, <https://doi.org/10.1524/zpch.218.1.51.25388>.
- [77] J.N. Israelachvili, *Van der Waals Forces between Particles and Surfaces*, 3rd Edition, 13, Elsevier, 2011, pp. 253–289.
- [78] L. Heitmeier, T. Voigtmann, O. D'Angelo, Rheological response of granular materials under varying gravitational conditions: a comparative study of discrete element method simulation and experiment, unpublished manuscript, 2021.
- [79] H.V. Atkinson, S. Davies, *Fundamental aspects of hot isostatic pressing: an overview*, *Metall. Mater. Trans. A* 31 (2000) 2981–3000.
- [80] J.T. Gostick, Z.A. Khan, T.G. Tranter, M.D. Kok, M. Agnau, M. Sadeghi, R. Jervis, PoreSpy: a Python toolkit for quantitative analysis of porous media images, *J. Open Source Softw.* 4 (37) (2019) 1296, <https://doi.org/10.21105/joss.01296>.
- [81] C.A. Schneider, W.S. Rasband, K.W. Eliceiri, NIH image to imagej: 25 years of image analysis, *Nat. Methods* 9 (2012) 671–675, <https://doi.org/10.1038/nmeth.2089>.
- [82] J.D. Bernal, J. Mason, Packing of spheres: co-ordination of randomly packed spheres, *Nature* 188 (1960) 910–911, <https://doi.org/10.1038/188910a0>.
- [83] E. DeGiul, J.N. McElwaine, M. Wyart, Phase diagram for inertial granular flows, *Phys. Rev. E* 94 (2016), 012904, <https://doi.org/10.1103/PhysRevE.94.012904>.
- [84] E. Marakis, M.C. Velsink, L.J. Corbijn van Willenswaard, R. Uppu, P.W.H. Pinkse, Uniform line fillings, *Phys. Rev. E* 99 (2019), 043309, <https://doi.org/10.1103/PhysRevE.99.043309>.
- [85] O. Pouliquen, M. Nicolas, P.D. Weidman, Crystallization of non-brownian spheres under horizontal shaking, *Phys. Rev. Lett.* 79 (1997) 3640–3643, <https://doi.org/10.1103/PhysRevLett.79.3640>.
- [86] O.R. Walton, C.P.D. Moor, K.S. Gill, Effects of gravity on cohesive behavior of fine powders: implications for processing lunar regolith, *Granul. Matter* 9 (2007) 353–363, <https://doi.org/10.1007/s10035-006-0029-8>.