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# A two-scale Weibull approach to the failure of porous ceramic structures made by robocasting: possibilities and limits

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## Abstract

This paper introduces our approach to modeling the mechanical behavior of cellular ceramics, through the example of calcium phosphate scaffolds made by robocasting for bone-tissue engineering. The Weibull theory is used to deal with the scaffolds' constitutive rods statistical failure, and the Sanchez-Palencia theory of periodic homogenization is used to link the rod- and scaffold-scales. Uniaxial compression of scaffolds and three-point bending of rods were performed to calibrate and validate the model. If calibration based on rod-scale data leads to over-conservative predictions of scaffold's properties (as rods' successive failures are not taken into account), we show that, for a given rod diameter, calibration based on scaffold-scale data leads to very satisfactory predictions for a wide range of rod spacing, i.e. of scaffold porosity, as well as for different loading conditions. This work establishes the proposed model as a reliable tool for understanding and optimizing cellular ceramics' mechanical properties.

### *Keywords:*

Cellular ceramics ; Fracture ; Mechanical properties ; Strength ;

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**1. Introduction**

Robocasting, also called direct-write assembly or solid freeform fabrication, is considered one of the top candidates for processing highly complex materials [Smay et al., 2002; Leong et al., 2003]. It consists of building three-dimensional structures, or scaffolds, layer-by-layer by extruding a continuous filament, or rod, through a tip guided by a computer-assisted positioning system [Saiz et al., 2007; Russias et al., 2007; Houmard et al., 2012b]. It allows very precise control of the scaffold’s macroscopic porosity, and allows the processing of a large range of materials, including bio-ceramics [Miranda et al., 2006] and bio-glass [Fu et al., 2011]. It is useful in many different areas, including battery electrodes [Liu et al., 2011], photonic crystals [Gratson et al., 2006], and tissue engineering [Woodard

1 et al., 2007; Lan Levengood et al., 2010].

2

3 Mechanical properties and reliability are key parameters in the appli-  
4 cation of any scaffold. For example, in bone-tissue engineering, stiffness  
5 plays a major role in cell proliferation toward bone growth [Woodard et al.,  
6 2007; Miranda et al., 2008b], while strength determines the capability to  
7 withstand *in vivo* loading [Woodard et al., 2007; Miranda et al., 2008b].  
8 Thus, it is necessary to quantitatively understand the scaffold’s mechanics  
9 (including elastic behavior, damage mechanisms, *etc.*), and to derive reli-  
10 able structure-properties relations. This effort requires the development of  
11 theoretical models and associated prediction tools along with experimental  
12 analysis. Such models, once carefully validated, will reduce the need for  
13 extremely time- and resource-consuming experiments, and will allow predic-  
14 tion of any scaffold’s behavior at no cost.

15 Several analysis have been proposed in the literature to improve the  
16 understanding of the mechanics of robocast scaffolds, but a general computa-  
17 tional framework with strong mechanical basis is still missing. For instance,  
18 the Finite Element (FE) method has been used to compute the elastic  
19 response of assemblies of rods and to elucidate the possible failure modes in  
20 compression and shear [Miranda et al., 2008a], but the analysis was based  
21 upon a critical stress criterion, which can be extremely mesh-dependent,  
22 and did not deal with the statistical aspect of failure. The classical Weibull  
23 framework has been used to deal with scaffold failure [Miranda et al., 2008b;  
24 Yang et al., 2010], but as the analysis was solely driven at the scaffold scale,  
25 it did not allow for an extrapolation of the role of the scaffold architecture  
26 outside the experimental domain. In another FE-based work, the effective  
27 properties of an element of volume representative of the scaffold’s architec-  
28 ture were computed in order to analyze the influence of bone growth within  
29 the scaffold on stiffness [McIntosh et al., 2009], but strength was not studied.

30

31 In this paper, we propose a two-scale model of the mechanical behavior of  
32 scaffolds made by robocasting, that is intended to overcome the limitations  
33 of existing approaches. The proposed framework involves representations at  
34 both the rod, or *micro*, scale (100  $\mu m$ ) and scaffold, or *macro*, scale (10 *mm*),  
35 allowing to derive mechanically sound structure-properties relations. It is  
36 based on the Sanchez-Palencia theory of periodic homogenization [Sanchez-  
37 Palencia, 1974] and the Weibull theory of failure probability [Weibull, 1939,  
38 1951]. Experimental rod and scaffold mechanical measurements are used in  
39 this paper for calibration and validation. We have focused on calcium phos-  
40 phate (HA & HA/TCP) bio-scaffolds, but most of the conclusions should be

1 transferable to other compositions, and more generally to any quasi-brittle  
2 porous materials.

## 3 **2. Materials & Methods**

### 4 *2.1. Uniaxial compression of scaffolds*

5 Cubic scaffolds were processed following the method detailed in [Franco  
6 et al., 2010; Houmard et al., 2012a]. The scaffold structure used in this work  
7 consists of stacked layers of parallel rods forming 90° angles (see Figure 1).  
8 Four sets of scaffolds were printed for this paper, three made out of pure  
9 hydroxylapatite (HA), and one made out of a 60% hydroxylapatite / 40%  
10 tricalcium phosphate (HA/TCP) composite:

- 11 • In the first one (batch  $S_{V=45\%}^{\text{HA}}$ ), the rod spacing varied from 400  $\mu\text{m}$  to  
12 985  $\mu\text{m}$ , and the rod diameter was adjusted from 200  $\mu\text{m}$  to 610  $\mu\text{m}$ ,  
13 so that the porosity was kept constant at 45%.
- 14 • In the second and third ones (batches  $S_{d=250\mu\text{m}}^{\text{HA}}$  &  $S_{d=200\mu\text{m}}^{\text{HA}}$ ), the rod  
15 spacing varied from 350  $\mu\text{m}$  to 870  $\mu\text{m}$  (for batch  $S_{d=250\mu\text{m}}^{\text{HA}}$ ) or from  
16 300  $\mu\text{m}$  to 580  $\mu\text{m}$  (for batch  $S_{d=200\mu\text{m}}^{\text{HA}}$ ), and the rod diameter was  
17 kept constant at 250  $\mu\text{m}$  (for batch  $S_{d=250\mu\text{m}}^{\text{HA}}$ ) or 200  $\mu\text{m}$  (for batch  
18  $S_{d=200\mu\text{m}}^{\text{HA}}$ ), so that the porosity varied from 25% to 65% (for batch  
19  $S_{d=250\mu\text{m}}^{\text{HA}}$ ) or from 35% to 65% (for batch  $S_{d=200\mu\text{m}}^{\text{HA}}$ ).
- 20 • The last one (batch  $S_{d=250\mu\text{m}}^{\text{HA/TCP}}$ ) is very similar to batch  $S_{d=250\mu\text{m}}^{\text{HA}}$ , the  
21 only difference being that it is made out of HA/TCP instead of pure  
22 HA.

23 Previous dimensions refer to deposition conditions, sintering (1275°C for  
24 HA scaffolds; 1100°C for HA/TCP scaffolds; see [Houmard et al., 2012a] for  
25 further details) inducing a shrinkage of *ca.* 16% (for HA scaffolds) or *ca.* 8%  
26 (for HA/TCP scaffolds) [Houmard et al., 2012a].

27 For each set of microstructural parameters, between 3 and 8 scaffolds  
28 were printed and analyzed.

29 The scaffolds' mass and dimensions were measured after sintering, and  
30 their total porosity were deduced using the theoretical densities of HA  
31 ( $3.16 \text{ g/cm}^3$ ) and TCP ( $3.14 \text{ g/cm}^3$ ) [Houmard et al., 2012a]. And for  
32 each set of scaffolds with similar microstructure, the standard deviation for  
33 porosity is *ca.* 2%.

34 Uniaxial compression tests were performed on the processed scaffolds,  
35 using a servo-hydraulic testing machine (MTS810, MTS Systems, Eden

1 Prairie, MN, USA) with a cross-head speed of  $0.2 \text{ mm}/\text{min}$  (time-to-fracture  
 2 *ca.*  $30 \text{ s}$ ). These data have already been detailed in [Houmard et al., 2012a].  
 3 For batches  $S_{V=45\%}^{\text{HA}}$ ,  $S_{d=250\mu\text{m}}^{\text{HA}}$  &  $S_{d=200\mu\text{m}}^{\text{HA}}$ , only the vertical (out-of-plane)  
 4 direction was tested. For batch  $S_{d=250\mu\text{m}}^{\text{HA}/\text{TCP}}$ , both the vertical and horizontal  
 5 (in-plane) directions were tested.

6 The scaffolds' compressive strength were identified on the obtained force-  
 7 displacement curves, as the maximum reached stress.

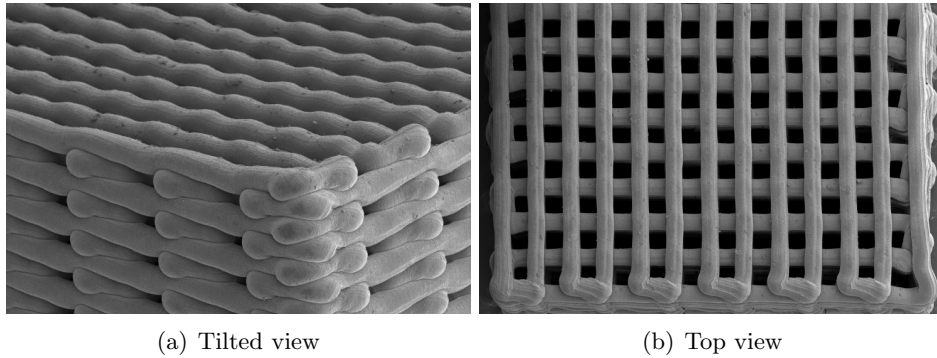


Figure 1: Representative SEM images of scaffolds made by robocasting. The scaffolds are approximately  $5 \text{ mm}$ -side cubes.

## 8 2.2. Three-point bending of single rods

9 Single rods, *i.e.* the elementary constituents of robocast scaffolds, were  
 10 processed by printing a single layer of lines. Because of strong adhesion with  
 11 the substrate, some lines broke into several pieces during sintering. However,  
 12 most of the pieces were long enough to be tested mechanically. Three sets of  
 13 lines were printed: batches  $R_{d=250\mu\text{m}}$ ,  $R_{d=510\mu\text{m}}$  &  $R_{d=840\mu\text{m}}$ , respectively  
 14 representing rods printed with a  $250 \mu\text{m}$ -,  $510 \mu\text{m}$ - &  $840 \mu\text{m}$ -diameter  
 15 tip, and respectively containing 16, 17 & 16 rods. After sintering (same  
 16 sintering conditions than for the scaffolds), their diameter was  $220 \pm 30 \mu\text{m}$ ,  
 17  $390 \pm 40 \mu\text{m}$  &  $670 \pm 60 \mu\text{m}$ , respectively. The rod diameters were chosen  
 18 so as to cover the largest range possible of available tips, thus allowing to  
 19 interpolate the rod data for any intermediate radius.

20 Three-point bending tests with a horizontal microindenter (Micro Mate-  
 21 rials) were performed on the rods to measure their flexural strength. Rods  
 22 of  $220 \pm 30 \mu\text{m}$ -,  $390 \pm 40 \mu\text{m}$ - &  $670 \pm 60 \mu\text{m}$ -diameter were mounted on  
 23 an aluminum holder atop  $0.56 \text{ mm}$ ,  $1.13 \text{ mm}$ , &  $1.76 \text{ mm}$  trenches, respec-  
 24 tively. They were bent by indenting at a constant load rate of  $200 \text{ mN}/\text{s}$

1 on their suspended center on the trench (time-to-fracture *ca.* 10 s). We  
 2 used either a diamond cono-spherical probe with a 25  $\mu\text{m}$  tip radius, or a  
 3 diamond spherical probe of 350  $\mu\text{m}$  tip radius (depending on rod diameter).  
 4 The rods' Weibull coefficients were identified on the obtained force-  
 5 displacement curves. For three-point bending, in the classical Euler frame-  
 6 work, the failure probability according to Weibull theory writes:

$$P^F = 1 - \exp\left(-\frac{V}{V_0} H(m) \left(\frac{\bar{\sigma}}{\sigma_0}\right)^m\right) \quad (1)$$

with 
$$\begin{cases} H(m) = \frac{\Gamma(\frac{m+1}{2})}{2\sqrt{\pi}(m+2)\Gamma(\frac{m+2}{2})} \\ \bar{\sigma} = Fl/\pi r^3 \end{cases}$$

7 where  $V$  is the rod's volume and  $V_0$  a reference volume (in this paper we take  
 8  $V_0 = 1 \text{ mm}^3$ ),  $H(m)$  a stress heterogeneity factor involving the classical  $\Gamma$   
 9 function [Hild and Marquis, 1992],  $\bar{\sigma}$  the Largest Positive Stress (LPS) in  
 10 the rod, and  $\sigma_0$  &  $m$  the two Weibull coefficients that have to be identified.  
 11 This equation can be rewritten in the classical Weibull form:

$$\ln\left(\ln\left(\frac{1}{1-P^F}\right)\right) = m \ln(\bar{\sigma}) + \ln\left(\frac{V}{V_0}\right) + \ln(H(m)) - m \ln(\sigma_0) \quad (2)$$

12 Thus, for each rod (index  $i$ ), one has:

$$\begin{cases} \bar{\sigma}_i^F = F_i^F l_i / \pi r_i^3 \\ V_i = \pi l_i r_i^2 \\ P_i^F = r / (N + 1) \end{cases} \quad (3)$$

13 where  $N$  is the number of rods in the set, and  $r$  the rank of the rod  $i$  (the  
 14 rod with the smallest LPS has  $r = 1$ ; the one with the largest LPS has  
 15  $r = N$ ). And for each set of rods, the Weibull coefficients were identified  
 16 by computing the least-square plane that best fits the  $(\bar{\sigma}_i^F, V_i, P_i^F)$  data in  
 17 the  $\left(\ln(\bar{\sigma}^F), \ln\left(\frac{V}{V_0}\right), \ln\left(\ln\left(\frac{1}{1-P^F}\right)\right)\right)$  space. Indeed, although the usual  
 18 procedure computes the least-square line in the  $\left(\ln(\bar{\sigma}^F), \ln\left(\ln\left(\frac{1}{1-P^F}\right)\right)\right)$   
 19 space, here the rods' diameter varies significantly, even in the same batch,  
 20 and this must be taken into account in the identification process.

### 1 **3. Modeling & Methods**

#### 2 *3.1. Micro-Macro bridge*

3 In this work, we use the Sanchez-Palencia theory of homogenization  
 4 [Sanchez-Palencia, 1974], also called periodic or first-order asymptotic ho-  
 5 mogenization, to link the elastic behavior of the scaffolds and the one of  
 6 their constitutive rods. Basically, this consists of (i) successively applying  
 7 six macroscopic deformations  $\underline{\underline{E}}^i$  (three extensions, three shears) on top of  
 8 free periodic deformations to a cell  $\omega$  representative of the microstructure  
 9 (also called Representative Volume Element, RVE), (ii) computing the as-  
 10 sociated microscopic stress fields  $\underline{\underline{\sigma}}^i$ , and (iii) integrating them to obtain the  
 11 corresponding macroscopic stress:

$$\underline{\underline{\Sigma}}^i = \frac{1}{|\omega|} \int_{\omega} \underline{\underline{\sigma}}^i d\omega \quad (4)$$

12 Finally, the homogenized stiffness tensor writes:

$$\widehat{H}_{ij} = \widehat{\Sigma}_j^i \quad (5)$$

13 (Engineering notations, denoted by  $\widehat{\cdot}$ , are used for simplicity.)

14 The underlying computations are performed using the finite element  
 15 method (FEM). To take into account the scaffolds' complex microstructure  
 16 (see Figure 1), level sets [Sethian, 1999] are used to represent the interfaces,  
 17 and a selective integration method is used to handle variations of material  
 18 properties [Moës et al., 2003; Genet, 2010]. Figure 2 presents a possible  
 19 RVE. Each rod (index  $i$ ) is located by (i) a point  $\underline{X}_i$  of its middle line, (ii)  
 20 the direction  $\underline{N}_i$  of its middle line, and (iii) its diameter  $d_i$ . The associated  
 21 level set function must (i) vanish at the rod's border, (ii) be positive inside  
 22 the rod, and (iii) be negative outside. We take the following function:

$$\phi_i(\underline{x}) = 1 - \frac{\|\underline{y} - {}^t\underline{N}_i \underline{y} \underline{N}_i\|}{d_i/2} \quad (6)$$

$$\text{with } \underline{y} = \underline{x} - \underline{X}_i$$

23 Then a global level set function is built. It must be the Boolean sum of the  
 24 level sets associated with all the rods in the RVE:

$$\phi(\underline{x}) = \max_i (\phi_i(\underline{x})) \quad (7)$$

25 Note that contrary to eXtended-FEM [Moës et al., 2003] and other methods  
 26 with enhanced-kinematics elements [Jirasek, 2000; Benkemoun et al., 2010],



1 we do not introduce any discontinuity in the displacement or strain fields  
 2 within the finite elements cut by the level set. Thus, the interfaces are  
 3 somehow smoothed within the finite elements they cut, introducing another  
 4 source of approximation within the FE solution. However, this method is  
 5 particularly simple to implement, and is accurate enough for our needs [Moës  
 6 et al., 2003; Genet, 2010]. (In all computations performed for this paper,  
 7 we made sure the initial mesh was fine enough so the induced error on the  
 8 homogenized stiffness was less than 1%. Typically, this entailed taking at  
 9 least 10 elements per rod diameter.)

10 To perform FE computations, materials properties, *i.e.* Young modulus  
 11 & Poisson ratio, must be prescribed for all integration points of the FE mesh,  
 12 even those outside the rods. Also, they must be non-null in order to avoid  
 13 having a singular stiffness matrix. We consider the following heterogeneous  
 14 isotropic material properties:

$$\begin{cases} E(\underline{x}) = E^{\text{rod}}H(\phi(\underline{x})) + \frac{E^{\text{rod}}}{M}(1 - H(\phi(\underline{x}))) \\ \nu(\underline{x}) = \nu^{\text{rod}}H(\phi(\underline{x})) \end{cases} \quad (8)$$

15 where  $E^{\text{rod}}$  &  $\nu^{\text{rod}}$  are the rod's Young modulus & Poisson ratio,  $H$  the  
 16 Heaviside function, and  $M$  a large number. (Typically, we took  $10^6$ .) The  
 17 stress fields are then post-treated using the following material properties:

$$\begin{cases} E(\underline{x}) = E^{\text{rod}}H(\phi(\underline{x})) \\ \nu(\underline{x}) = \nu^{\text{rod}}H(\phi(\underline{x})) \end{cases} \quad (9)$$

18 On a more technical basis, all meshes were generated using GMSH  
 19 [Geuzaine and Remacle, 2009], and FE computations were done within the  
 20 LMT++ library [Leclerc, 2010], which makes use of the CHOLMOD linear  
 21 solver [Chen et al., 2008].

### 22 3.2. Macroscopic failure probability

23 After building the previous computational bridge between microscopic  
 24 and macroscopic strain and stress fields, we use the Weibull theory of failure  
 25 probability [Weibull, 1939, 1951] to predict the probability of failure of a  
 26 given scaffold under a given load.

27 Basically, if the Weibull theory was first introduced based on phenomeno-  
 28 logical considerations [Weibull, 1939, 1951], it was later shown to rely on  
 29 more fundamental basis: it is based on a Poisson process of the distribution  
 30 of activated defects, the process intensity being a function of volume (with

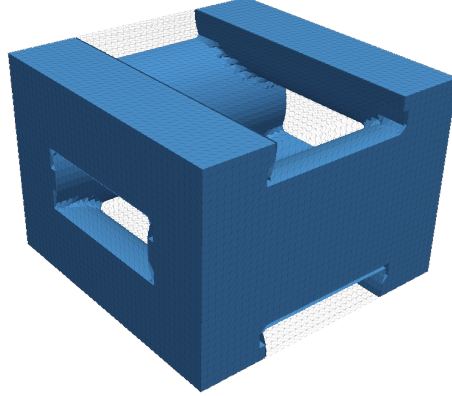


Figure 2: Computational periodic Representative Volume Element (RVE) of the robocast scaffolds used to link microscopic (*i.e.* rod) and macroscopic (*i.e.* scaffold) elastic properties through Sanchez-Palencia homogenization [Sanchez-Palencia, 1974]. The zero-level-set of the function representative of each single rod is visible, as are all elements fully inside each rod. The small distance between the elements and the surface is induced by use of a mesh that does not conform to the actual geometry.

1 linear dependence) and stress (with power dependence), and the weakest  
 2 link principle [Freudenthal, 1968; Hild and Marquis, 1992]. In the case of  
 3 a body of volume  $V$  submitted to an homogeneous uniaxial stress  $\sigma$ , the  
 4 theory leads to the following widespread failure probability law:

$$P^F = 1 - \exp\left(-\frac{V}{V_0} \left(\frac{\sigma}{\sigma_0}\right)^m\right) \quad (10)$$

5 where  $\sigma_0$  and  $m$  are the classical Weibull coefficients, and  $V_0$  a reference  
 6 volume. (Here we took  $V_0 = 1 \text{ mm}^3$ .)

7 Several extensions have been proposed to this law in order to take into  
 8 account multiaxial stress fields [Evans, 1978; Lamon, 1988]. In this paper,  
 9 we use a classical approach, based on the idea that only positive defor-  
 10 mations will turn existing defects into propagating cracks, which has been  
 11 experimentally evidenced for robocast scaffolds [Miranda et al., 2007], and  
 12 used in several models *e.g.* for concrete [Desmorat, 2006] and ceramic ma-  
 13 trix composites [Genet et al., 2012b]. Thus, we will consider the following  
 14 probability law:

$$P^F = 1 - \exp\left(-\frac{1}{V_0} \int_V \left(\frac{\|\langle \underline{\epsilon} \rangle_+\|}{\epsilon_0}\right)^m dV\right) \quad (11)$$

15 where  $\langle \underline{\epsilon} \rangle_+$  is the positive part of the deformation tensor, built by removing

1 all non-positive eigenvalues from  $\underline{\underline{\epsilon}}$ .

2

3 It is important to note that the integral of Equation (11) is theoretically  
4 defined over all the rods of the scaffold, *i.e.* on the whole microscopic domain,  
5 and is then non affordable *a priori*. However, thanks to the computational  
6 bridge built in Section 3.1, the computation of this integral can actually be  
7 split into two computations: one on the scaffold scale and one on the rod  
8 scale but on a single periodic cell. This splitting is illustrated in Figure 3.  
9 On the macroscopic scale, one has:

$$P^F = 1 - \exp\left(-\frac{1}{V_0} \int_{\Omega} I(\underline{\underline{E}}(\underline{\underline{X}})) d\Omega\right) \quad (12)$$

10 While for each macroscopic point,  $I$  is computed on the microscopic scale:

$$I(\underline{\underline{E}}) = \frac{1}{|\omega|} \int_{\omega} \left( \frac{\|\langle \underline{\underline{\epsilon}}^{\underline{\underline{E}}}(\underline{\underline{x}}) \rangle_+\|}{\epsilon_0} \right)^m d\omega \quad (13)$$

11 Note that  $I$  is a non-linear function of  $\underline{\underline{E}}$ , and cannot be computed for a  
12 reduced set of basic deformations and then computed by linear combina-  
13 tions. As a consequence, it must actually be computed for every single  
14 macroscopic deformation.

15

16 In summary, the following steps are used to compute the failure proba-  
17 bility of a given scaffold under a given load (see Figure 3):

- 18 • Thanks to the homogenized properties obtained in Section 3.1, the  
19 problem of the loaded scaffold is solved, and the macroscopic strain  
20 and stress fields are computed.
- 21 • For every required macroscopic point (typically, integration points),  
22 the microscopic strain and stress fields are computed, and the integral  
23 of Equation (13) is computed.
- 24 • Finally, the integral of Equation (12) is computed.

25 Thus, this strategy accounts for the influence of both structural parame-  
26 ters such as global volume and geometry (through the computation on the  
27 macroscopic scale, step 1) and microstructural parameters such a rod di-  
28 ameter and rod spacing (through the computation on the microscopic scale,  
29 step 2).

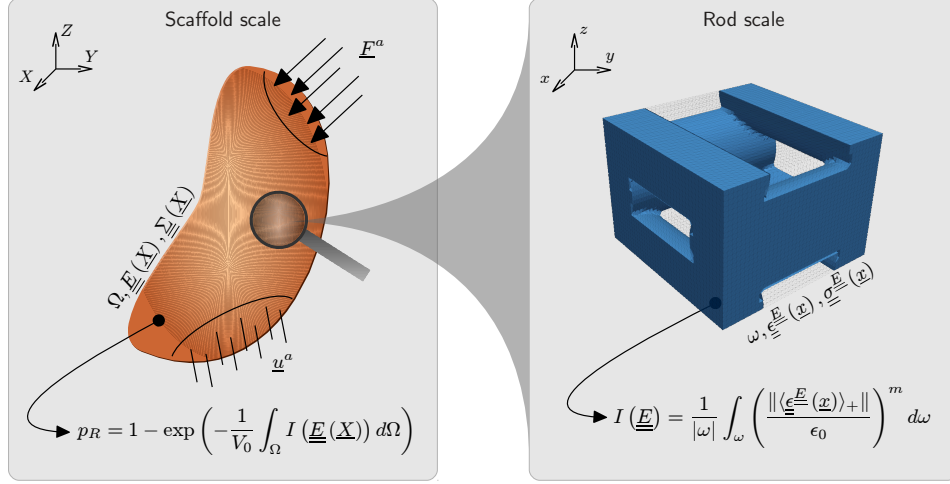


Figure 3: The proposed two-scale approach to predict the failure probability of a given scaffold under a given load. On the macroscopic scale (left),  $\Omega$  denotes the volume of the scaffolds, and  $\underline{E}$  &  $\underline{\Sigma}$  the macroscopic strain and stress fields. On the microscopic scale (right),  $\omega$  denotes the volume of the periodic representative cell,  $\underline{\epsilon}^{\underline{E}}$  &  $\underline{\sigma}^{\underline{E}}$  the microscopic strain and stress field associated with the macroscopic strain  $\underline{E}$ , and  $\epsilon_0$  &  $m$  are the two Weibull coefficients.

### 1 3.3. Strength-Probability relations

2 For many materials, and especially ceramics, strength is not an actual  
3 intrinsic material parameters, as it is strongly probabilistic and most of all  
4 geometry- and load-dependent. Thus, the only intrinsic magnitudes are the  
5 parameters of the law giving strength distribution as a function of geometry  
6 and load, *i.e.* the Weibull-like law of Equation (12). However, another key  
7 magnitude is the strength associated with a given probability of failure of  
8 a given structure under a given load [Davidge et al., 1973; Gauthier and  
9 Lamon, 2009; Ladevèze and Genet, 2010], as it allows engineers to select  
10 materials and structures, and is the core of any certification process [Davidge  
11 et al., 1973]. It can be derived from the strength distribution as follows. For  
12 simplicity, consider that the macroscopic load is proportional to a scalar  
13 magnitude denoted  $\sigma$  (extension to more complex cases is straightforward):

$$14 \quad \underline{F}^a(\underline{X}) = \sigma \bar{\underline{F}}^a(\underline{X}) \quad (14)$$

15 Thus, thanks to the linearity of the macroscopic problem, one has:

$$\underline{\underline{\Sigma}}(\underline{X}) = \sigma \bar{\underline{\underline{\Sigma}}}(\underline{X}), \quad \underline{\underline{E}}(\underline{X}) = \sigma \bar{\underline{\underline{E}}}(\underline{X}) \quad (15)$$

1 To express  $I$  as a simple function of  $\sigma$ , it is important to note the following  
 2 property:

$$I(\sigma \underline{\underline{E}}) = \sigma^m I(\underline{\underline{E}}) \quad \forall \sigma > 0 \quad (16)$$

3 Thus, coupling Equations (12), (15) & (16), one obtains the structure  
 4 strength  $\sigma^F$  associated with a given probability of failure  $P^F$ :

$$\sigma^F = \left( \frac{V_0}{\int_{\Omega} I(\underline{\underline{E}}(\underline{\underline{X}})) d\Omega} \ln \left( \frac{1}{1 - P^F} \right) \right)^{1/m} \quad (17)$$

## 5 4. Results & Discussion

### 6 4.1. Experimental results

#### 7 4.1.1. Uniaxial compression of scaffolds

8 Two representative force-displacement curves are presented in Figure 4.  
 9 They both present a first stage with successive failures of non-critical rods  
 10 (that do not cause the failure of the scaffold as a whole), and then, after the  
 11 failure of the critical rod, a second stage of relatively sharp failure of the  
 12 whole scaffold. This indicates that the scaffold's failure in compression is  
 13 not brittle, but quasi-brittle: it requires multiple lines breaking before final  
 14 failure.

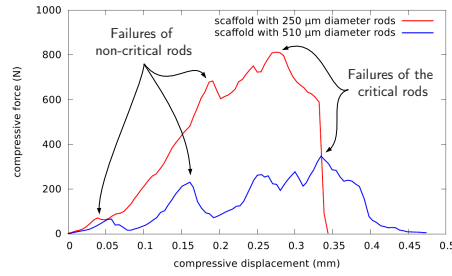


Figure 4: Representative force-displacement curves of the uniaxial (vertical) compression tests performed on robocast scaffolds. Shown are two examples of scaffolds printed respectively with a 250  $\mu m$ -diameter tip and a 490  $\mu m$ -spacing between the lines (both before sintering), and with a 510  $\mu m$ -diameter tip and a 830  $\mu m$ -spacing between the lines (both before sintering).

15 Figure 5 presents the compressive strength for each set of scaffolds.  
 16 Strength varies significantly from one morphology to another even when

- 1 changing both rods spacing and diameter so as to keep the porosity con-
- 2 stant (see Figure 5(a)). The porosity-dependence of strength for a given rod
- 3 diameter (250  $\mu\text{m}$  before sintering) is presented in Figure 5(b).

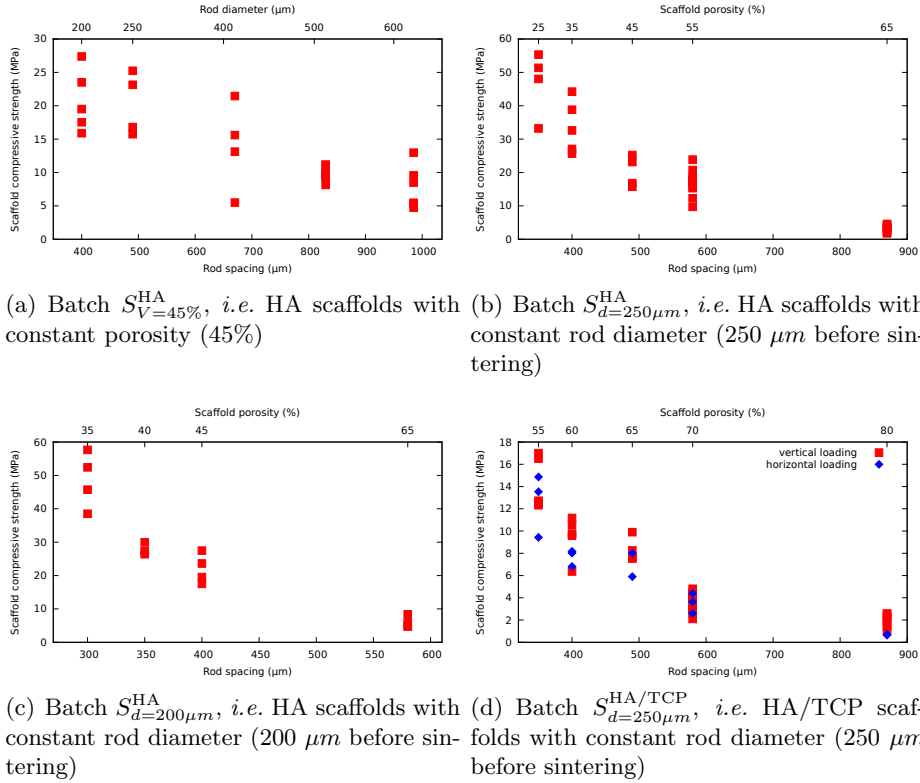


Figure 5: Compressive strength of scaffolds with multiple rods diameter and center-to-center spacing (every point corresponds to an individual test). One can see in (a) that even for scaffolds with constant porosity, strength depends on the rods diameter and spacing; and in (b), (c) & (d) the porosity-dependence of strength for multiple materials, rod diameters and loading directions.

#### 4.1.2. Three-point bending of single rods

The force-displacement curves (not represented) present a linear regime, followed by a sharp failure, indicating that the rod’s failure in bending is fully brittle.

Table 1 presents the average strength measured for each batch of rods, as well as the standard deviation of each distribution. The printing process uses inks with relatively high water and organic components, resulting in

1 a significant amount of microporosity within the rods [Franco et al., 2010;  
 2 Houmard et al., 2012a]. These micropores are defects from the mechanical  
 3 point of view, which explains the variability in the data. It is important  
 4 to note that for some biological applications, microporosity is required so a  
 5 balance must be found between biological and mechanical properties. The  
 6 strong volume-dependence and high scattering in the data illustrate the need  
 7 for a robust statistical analysis based on the Weibull theory.

Batch	Average Strength (MPa)	Standard deviation (MPa)
$R_{d=250\mu m}$	190	109
$R_{d=510\mu m}$	119	34
$R_{d=840\mu m}$	110	36

Table 1: Average strength and standard deviation for each set of rods. On top of the orders of magnitude, one can see here the important volume-dependence and scattering of the rods' strength, establishing the need for a Weibull analysis.

8 Figure 6 presents the computed values of Weibull coefficients for each  
 9 set of rods. It was checked that these values are statistically converged.  
 10 Both  $\sigma_0$  and  $m$  are found to depend on rod diameter, the dependence being  
 11 linear. As a consequence, the rods' Weibull coefficients are not intrinsic,  
 12 *i.e.* the distribution of defects depends on rod diameter. This is probably  
 13 induced by the problems associated with drying and the burn out of the  
 14 ink's organic components in larger rods, as well as the variations in local  
 15 sintering conditions for rods with different diameters (even though the global  
 16 sintering conditions are similar for all single rods), which has been shown  
 17 to modify the average shape of pores for hydroxylapatite [Prokopicv and  
 18 Sevostianov, 2006]. Moreover, solvent elimination/drying could be different  
 19 for different rod diameters, resulting in different pores populations.

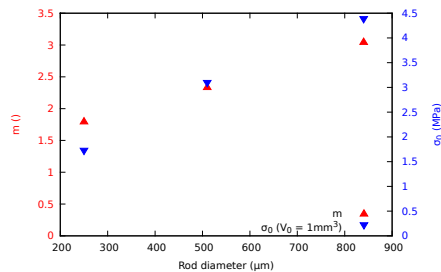


Figure 6: Weibull coefficients of single rods as a function of their diameter. One can see the linear dependence of the coefficients to the rod diameter.

1 *4.1.3. Summary & Discussion*

2 If the porosity-dependence of the scaffold’s compressive strength illus-  
 3 trated on Figures 5(b), 5(c) & 5(d) is a rather expected tendency, the  
 4 architecture-dependence illustrated on Figure 5(a) needs to be further dis-  
 5 cussed. Indeed, two mechanisms can be responsible for this: (i) the varia-  
 6 tions in rods’ failure properties with regard to their diameter; and (ii) the  
 7 variations in the sequence of rods failure from the onset of damage to final  
 8 failure, with regard to rods diameter. Thus, it is necessary to investigate all  
 9 potential scenarios to establish the importance of each in the failure of robo-  
 10 cast scaffolds. Such an investigation has been carried out based upon the  
 11 computational modeling described in Section 3, and its outcome is presented  
 12 below.

13 *4.2. Strength predictions*

14 Here we apply the strength modeling framework described in Section  
 15 3.3 to the experimental conditions of Sections 2.1, *i.e.* cubic scaffolds un-  
 16 der uniaxial compression (the compressive stress is denoted  $\sigma$ ). Thus, the  
 17 mechanical problem can be solved analytically, and leads to simple homo-  
 18 geneous macroscopic fields:

$$\underline{\underline{\Sigma}} = \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & -\sigma \end{pmatrix}, \quad \underline{\underline{E}} = \begin{pmatrix} \frac{\tilde{\nu}_{13}\sigma}{\tilde{E}_1} & 0 & 0 \\ 0 & \frac{\tilde{\nu}_{12}\sigma}{\tilde{E}_1} & 0 \\ 0 & 0 & \frac{-\sigma}{\tilde{E}_3} \end{pmatrix} \quad (18)$$

19 where the  $\tilde{\nu}$  variables are the homogenized Young moduli & Poisson ratios of  
 20 the scaffolds. Having an homogeneous solution of the macroscopic problem  
 21 greatly simplifies the computation of Equations (12) & (13).

22 For the rods elastic properties, we used the ones measured through mi-  
 23 croindentation in [Miranda et al., 2008b]. They are presented in Table 2.  
 24 Elastic properties of HA/TCP rods were interpolated between pure HA and  
 25 pure TCP rods properties through the rule of mixture.

	HA	TCP
Young modulus (GPa)	82	36
Poisson ratio ( )	0.28	0.28

Table 2: Elastic properties of HA & TCP rods, as reported in [Miranda et al., 2008b].



1 The model predictions for the scaffolds' strength relies on two scalar pa-  
2 rameters: the rods' Weibull coefficients  $\epsilon_0$  &  $m$ , which characterize a pop-  
3 ulation of defects (see Section 3.2). The experimental data used to identify  
4 these parameters determine the considered population of defects, and then  
5 the outcome of the model. Two sets of data are considered here: (i) the one  
6 on the rod scale (see Section 4.1.2), thus following a *bottom-up* approach,  
7 and (ii) the one on the scaffold scale (see Section 4.1.1), thus following a  
8 *top-down* approach.

#### 9 4.2.1. Predictions based on rod data

10 When considering data on the rod scale (see Section 4.1.2, especially Fig-  
11 ure 6, with  $\epsilon_0 = \sigma_0/E^{\text{rod}}$ ) to predict the failure of a whole scaffold through  
12 the two-scale Weibull model proposed here, one actually considers that the  
13 failure of a single rod in the scaffold leads to the failure of the whole scaffold.  
14 It is an extremely conservative hypothesis, but it is used in several models  
15 in the literature, especially models for brittle materials with industrial ap-  
16 plications [Cluzel et al., 2009; Genet et al., 2012a].

17 However, in our case, such an hypothesis is far too conservative, and scaf-  
18 folds out-of-plane strength predictions (not shown) are much lower ( $< 10\%$ )  
19 than experimental values. Furthermore, the model does not reproduce the  
20 morphological dependence of the strength, either for scaffolds with constant  
21 porosity or for scaffolds with constant rod diameter. This establishes that  
22 even if variations in rods' Weibull moduli with regard to rod diameter have  
23 a slight role in scaffold strength, it is not the main mechanism in scaffold  
24 failure. As a consequence, the successive breaking of rods must be the  
25 key mechanism to the scaffold's final failure, and must be rod diameter-  
26 dependent: for scaffolds with small rods diameter, the stress level can be  
27 drastically increased even after several single rods break; this is not the case  
28 for scaffolds with large rods diameter.

#### 29 4.2.2. Predictions based on scaffold data

30 When considering the data on the scaffold scale (see Section 4.1.1) to  
31 predict the failure of scaffolds through the two-scale Weibull model proposed  
32 here, one does not consider anymore the largest microdefects present within  
33 the scaffolds (which are responsible for the failure of the first single rods),  
34 but the critical ones, *i.e.* the ones responsible for the failure of the whole  
35 scaffold (see Figure 4). In other words, the model tracks the failure of the  
36 critical rod, *i.e.* the one that finally causes the whole scaffold to fail. And  
37 as the successive failures of rods toward the scaffolds' final failure is a rod

1 diameter-dependent phenomenon, a set of Weibull coefficients characterizing  
 2 the scaffolds critical defects must be identified for each rod diameter.

3 *HA scaffolds.* For HA scaffolds, the batch  $S_{V=45\%}^{\text{HA}}$  was used as it contains  
 4 scaffolds with same porosity but multiple rod diameter. Figure 7 & Table 3  
 5 present the outcome of this identification process, made through the best-fit  
 6 method. For the sake of simplicity, we have kept the dispersion coefficient  $m$   
 7 equal for all scaffolds, while changing only the mean coefficient  $\sigma_0$ . The fact  
 8 that  $\sigma_0$  decreases very significantly with rod diameter is related to the fact  
 9 that breaking a single rod of small diameter is less critical than breaking a  
 10 single rod of large diameter, even in scaffolds with the same overall porosity.

11 Figure 8 presents the scaffolds' out-of-plane strength predicted by the  
 12 proposed model based upon this identification, compared with the exper-  
 13 imentally measured ones (see Section 4.1.1): one can clearly see that it is  
 14 suitable to predict the strength of scaffolds with a large range of rods spacing,  
 15 *i.e.* of scaffold porosity, outside the identification domain. In other words,  
 16 based on the data of a single set of scaffolds with one line diameter and one  
 17 macroporosity, the proposed two-scale Weibull model can predict the be-  
 18 havior of scaffolds with a wide range of macroporosities. As a consequence,  
 19 using numerical optimization [Allaire et al., 2002], it could be used to find  
 20 optimal scaffold design (with optimal shape and optimal graded porosity)  
 21 with regard to strength for a given application (*i.e.* given fixations, given  
 22 load). The only restriction regarding strength optimization is to keep the  
 23 rod diameter constant, which is imposed by the process anyway.

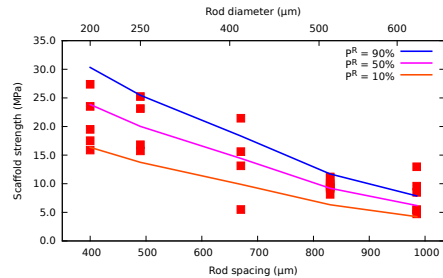
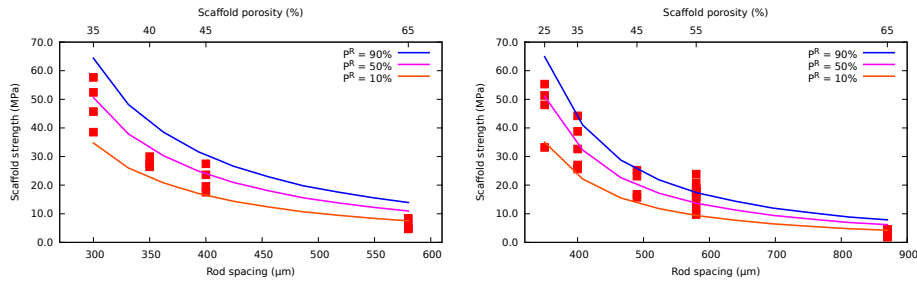


Figure 7: Identification of the HA rods' Weibull coefficients characterizing the population of HA scaffold critical defects based on scaffold scale data. Batch  $S_{V=45\%}^{\text{HA}}$  is used so as to identify a set of Weibull coefficients for each rod diameter.

24 *HA/TCP scaffolds.* For HA/TCP scaffolds, scaffolds with only one rod di-  
 25 ameter ( $250 \mu\text{m}$ ) were printed. One particular printing configuration (rod  
 26 spacing  $590 \mu\text{m}$ , porosity 70 %) and loading condition (vertical compression)

$d$ ( $\mu m$ )	200	250	410	510	610
$m$ ( )	5	5	5	5	5
$\sigma_0$ (MPa)	180	140	70	45	30

Table 3: HA rods' Weibull coefficients characterizing the population of critical defects in HA scaffolds, *i.e.* the ones responsible for the scaffolds' final failure. The large diameter-dependence of these coefficients, despite the fact that rods' Weibull coefficients characterizing their largest defects (*i.e.* the one responsible for the failure of the first rod) are only slightly diameter-dependent, is induced by the rod diameter-dependence of the successive failures of rods in the scaffold. This phenomenon is not explicitly modeled in the proposed approach, but implicitly through this diameter-dependence of the Weibull coefficients.



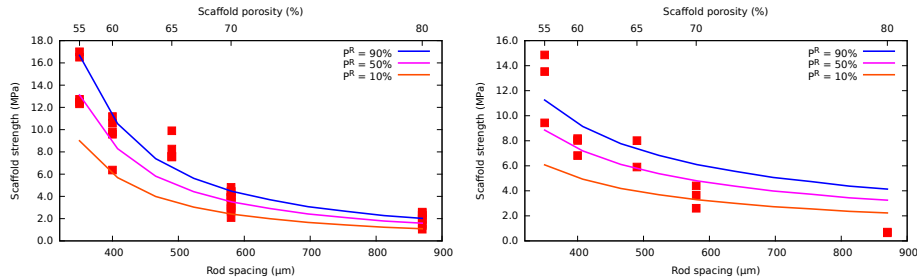
(a) Batch  $S_{d=200\mu m}^{HA}$ , *i.e.* HA scaffolds with constant rods diameter ( $200 \mu m$  before sintering) (b) Batch  $S_{d=250\mu m}^{HA}$ , *i.e.* HA scaffolds with constant rods diameter ( $250 \mu m$  before sintering)

Figure 8: Comparison of the HA scaffolds' out-of-plane strength predicted through the proposed two-scale Weibull model and measured experimentally. It can be seen that for a given diameter, the model can predict scaffold strength for a large range of porosities, outside the calibration domain.

1 was used to calibrate the model's parameters. Table 4 presents the outcome  
 2 of this identification process, once again made through the best-fit method.  
 3 Figure 9 presents the scaffolds' out-of-plane and in-plane strength predicted  
 4 by the proposed model based upon this identification, compared with the  
 5 experimentally measured ones (see Section 4.1.1). Once again the model is  
 6 shown to suitably predict the strength of scaffolds with a large range of rods  
 7 spacing, *i.e.* of scaffold porosity. Moreover, it is shown to provide satisfy-  
 8 ing predictions for different loading condition with regard to the calibration  
 9 loading condition.

$m$	$( )$	$\sigma_0$	$(MPa)$
5		38	

Table 4: HA/TCP rods' Weibull coefficients characterizing the population of critical defects in the scaffolds.



(a) Batch  $S_{d=250\mu m}^{HA/TCP}$ , *i.e.* HA/TCP scaffolds with constant rods diameter ( $250 \mu m$  before sintering), loaded vertically  
 (b) Batch  $S_{d=250\mu m}^{HA/TCP}$ , *i.e.* HA/TCP scaffolds with constant rods diameter ( $250 \mu m$  before sintering), loaded horizontally

Figure 9: Comparison of the HA/TCP scaffolds' out-of-plane strength predicted through the proposed two-scale Weibull model and measured experimentally. It can be seen that the model can predict scaffold strength for a large range of porosities and several loading conditions, outside the calibration domain.

## 10 5. Conclusion

11 In this paper, we have introduced a new two-scale model of the mechanics  
 12 of hierarchical quasi-brittle materials, by the example of calcium phosphate  
 13 scaffolds made by robocasting for bone-tissue engineering applications. The  
 14 domain of validity and the different possibilities offered by this model have  
 15 been studied based on two sets of experimental data: uniaxial compression

1 tests on scaffolds and three-point bending tests on single rods. While the  
2 former has been detailed in [Houmard et al., 2012a], the latter is presented  
3 here for the first time, and leads to an interesting fact: rods' Weibull coeffi-  
4 cients are actually rod diameter-dependent, meaning that the population of  
5 defects is different in rods printed with different tip diameters.

6 At the core of the proposed model, the rod and scaffold scales are linked  
7 through the Sanchez-Palencia's theory of periodic homogenization [Sanchez-  
8 Palencia, 1974]. Furthermore, scaffold strength is predicted through the  
9 Weibull's theory of failure probability [Weibull, 1939, 1951]. The Weibull  
10 integral is computed on the rod scale, and transferred at the scaffold scale  
11 thanks to the computational bridge established between microscopic and  
12 macroscopic strain and stress fields. This allows to account for both mi-  
13 crostructural and structural effects, and then to study with a single model  
14 the influence of *e.g.* rod diameter, rod spacing, scaffold size, *etc.*

15 The model has been calibrated in two ways: directly from the rod scale  
16 experimental data (*i.e.* following a bottom-up approach) and by fitting the  
17 scaffold scale experimental data (*i.e.* following a top-down approach). Both  
18 ways lead to very different answers, meaning that two different populations  
19 of defects have been identified. When calibrating the rods' Weibull coeffi-  
20 cients based on the rods three-point bending tests, one actually characterizes  
21 the population of rods' largest defects. Transferred to the scaffold scale, this  
22 leads to very conservative predictions that are not compatible with experi-  
23 mental data, as it is necessary to take into account the successive failures of  
24 rods within the scaffolds from the onset of damage to the final failure. This  
25 process can be implicitly handled in the proposed model by calibrating the  
26 rods' Weibull coefficients from the uniaxial compression tests on scaffolds,  
27 which means that one actually characterizes the population of critical de-  
28 fects, *i.e.* those responsible for the scaffold's final failure. This allowed us  
29 to make the model compatible with all available experimental data on the  
30 scaffold's scale.

31 It is important to point out that if the rods Weibull modulus was very  
32 high, then the failure of a single rod would trigger the failure of all other rods,  
33 and would induce the failure of the scaffold, and modeling the link between  
34 rod and scaffold failure would be more straightforward. However, in the  
35 more realistic case investigated here where rods Weibull modulus are pretty  
36 low, multiple single rod failures, all at different stress levels, are necessary  
37 to break the scaffolds, and the modeling becomes more involved. Thus, the  
38 present model appears to be a good compromise between modeling effort and  
39 prediction capabilities. Indeed, even though numerical simulation is required  
40 on the rod scale, and experimental calibration from scaffold scale data is

1 required for each rod diameter, the model has been shown to accurately  
2 predict the strength distributions of a wide range of scaffolds, with given  
3 rod diameter but varying porosities, far away from the calibration domain,  
4 and under loading conditions different from the calibration ones.

5 Consequently, the present model could be used to find optimal printing  
6 patterns, with regard to stiffness and strength, for specific geometries and  
7 mechanical loads. Indeed, novel scaffold designs are now being investigated  
8 for application in multiple domains such as tissue engineering. For instance,  
9 there were attempts to mimic the structure of real bone, with low porosity at  
10 the scaffold's surface, and higher porosity toward its core. (Other example  
11 are presented in [Houmard et al., 2012a].) Thus, with graded porosity, the  
12 scaffold design domains become too vast to be explored without the help  
13 and guidance from a computational model such as the one introduced in  
14 this paper. Numerical optimization (see for instance [Allaire et al., 2002])  
15 will have to be used, based on multiscale models as the one presented here,  
16 in order to process truly optimal scaffold for each application.

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21 her help in robocast scaffolds and single rods processing.

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