Mechanical Properties of ZrO₂ TPMS Structures Prepared by DLP 3D Printing

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Abstract: Compressive strength is one of the most important mechanical properties for cellular ceramics. But the dependence of compressive strength of highly porous cellular ceramic structures on porosity remains unclear due to the limit of available methods for making such strong structures based on specific structural designs. In this paper, the TPMS structures, namely P-cell, and neovius structure, were prepared based on the DLP 3D printing technology. Samples with various unit cells were fabricated with zirconia. The relative density of the sintered samples exceeds 99%. The effects of cell number on the compressive strength and deformation of the model were investigated. Samples with similar relative densities exhibited comparable mechanical property in aspect of compressive strength. It is very interesting that the cell number eventually only influence the total strain.

1. Introduction
Lightweight has always been the target of engineering-materials. To achieve this goal, researches has been investigated[1-4]. These special materials have been widely applied on the automotive and aerospace fields with their lightweight and the ability to absorb compressive energy effectively[5-7], heat transfer efficiently[8]. Topology optimization, that is, the preparation of cellular materials, is an effective way to achieve lightweight. Cellular structures have been found to consistently have more desirable specific properties (properties by unit mass/volume) than their bulk material counterparts[9].

Researchers are inspired from nature frequently in the preparation of cellular structures. Recently, triply periodic minimal surfaces (TPMS) as a bionic structure, has been widely studied by researchers. The TPMS are surfaces with zero mean curvature and are characterized by local area minimizing which means any sufficiently small patch taken from the TPMS has the smallest area among all patches created under the same boundaries[10-13]. A typical example of TPMS in nature is soap film. TPMS with the open cell structure deemed to facilitate cell migration and vitalization, while retaining a high degree of structural stiffness can also be found from butterfly, beetle shells[14-16]. This characteristic of high porosity and high surface to volume ratio is of great significance for the preparation of biological scaffolds.[17, 18]. Porous scaffolds based on TPMS structure can well simulate host tissue, the porous part promotes cell infiltration, and the solid part play a role in load bearing. The porosity and stiffness are negatively correlated, so finding a compromise point is critical.
The research of H. Montazerian et al showed that when the relative density of TPMS structure is 30%, good biological permeability and mechanical property can be maintained simultaneously [19].

The material and geometry play a key role on the mechanical properties of cellular structure. Cellular solids: ceramics, polymers, metal of the three classes, polymer materials are the most widely-publicized. Oraib Al-Ketan et al fabricated polymer TPMS metamaterials, and they found that polymer microgrids based on TPMS wafers exhibit better mechanical properties than other microgrids containing metallic and ceramic coated polymer bases, and are less affected by density changes [20]. And they studied the mechanical properties of composite phase of TPMS structure, adding the composite phase model can reduce stress concentration, and mainly showed the bending-dominated [21, 22]. Diab W. Abueidda et al. printed four TPMS structures: pcell, gyroid, iwp and neovius via selective laser sintering (SLS), and tested their mechanical properties. The results showed that pcell had the worst mechanical performance, gyroid followed, iwp and neovius with similar mechanical behaviors, which were better than the other two structures [23, 24]. I. Maskery et al. studied the structural deformation mode of some TPMS, and they found that pcell is bending-dominated while gyroid and diamond are stretching-dominated [25].

Metal materials are widely studied for their excellent properties. Oraib Al-Ketan et al. prepared TPMS structure and truss structure by SLS sintering technology based on maraging steel powder. Their results show that the mechanical properties of TPMS are better than those of truss structure, which is consistent with the results of TPMS prepared by Han et al. [26-28]. I. Maskery et al. prepared double gyroid based on Al-Si10-Mg alloy and had an investigate on their deformation mechanism and absorption energy, they found that cell size plays an important role in determining the failure mechanism of metal additive manufacturing lattices [29]. Yan et al. also realized the printing TPMS structure by using 316L stainless steel [30], Ti-6Al-4V [31].

Of the three classes, ceramic materials are the least well studied. Due to the constraints of preparation methods, there are few studies on the preparation and properties of TPMS structure based on ceramic materials. With the development of ceramic 3D printing technology, the preparation of TPMS structure has become a reality [32].

In this work, primitive cell, neovius (two types of TPMS) were been fabricated based on the zirconia suspension, and their linear and nonlinear mechanical responses were studied experimentally. Pcell with similar relative density was prepared, and the mechanical behavior of pcell under different binding conditions of x, y, z axis was investigated, and neovius is the same. One advantage of using TPMS structures to create cellular materials is to overcome the common weaknesses of truss/strut-based structures because joints and struts do not exist in the TPMS. The connection of TPMS allows a smoother transfer of loads which leads to a better stress dispersion compared to truss/strut-based cellular materials possessing joints.

2. Experimental procedure

2.1. Methodology

The primitive cell and neovius cell belong to the family of triply periodic minimal surfaces (TPMS), a subset of the larger class of constant mean curvature surfaces. In particular, TPMS are categorised by their zero mean curvature at every point. TPMS equations describe 3D surfaces which, for the purpose of additive manufacturing, can be taken as the boundary between void and solid material. Matrix phase pcell, neovius structures with arbitrary numbers of cells and volume fractions can be generated by finding the \( U = 0 \) isosurface of the TPMS equation [33, 34].

\[
U_p = \cos(k_x x) + \cos(k_y y) + \cos(k_z z) \tag{1}
\]

\[
U_{\text{neovius}} = \{3(\cos(k_x x)) + \cos(k_y y) + \cos(k_z z) + 4 \cos(k_x x) \cos(k_y y) \cos(k_z z)\} - t \tag{2}
\]

For each of the above, \( k_i \) are the TPMS function periodicities, defined by
\[ k_i = 2\pi \frac{n_i}{L_i} \quad \text{(with } i = x, y, z) \]  

where \( n_i \) are the numbers of cell repetitions in the directions \( x, \) \( y \) and \( z \), and \( L_i \) are the absolute sizes of the structure in those directions. In equations (1,2), \( t \) can be used as a variable to control the volume fraction (relative density).

### 2.2. Fabrication

3D printing based on sinking digital light processing technology with the raw materials of zirconia was employed in fabricating the TPMS structures at room temperature. Zirconia suspension and printer are purchased from Jiashan CeramPlus material co., LTD. The thickness of the printer is 50 \( \mu m \) and the pixel is 50 \( \mu m \times 50 \mu m \). After the printing process, it is sintered in a muffle furnace to 1550°C. Figure 1 is the temperature protocol. Holding on 80°C for an hour to dehumidify all the specimens should be sintered immediately after printed to avoid moisture and surface crack.

![Figure 1. Thermogravimetric analysis (TGA) of zirconia: weight (black line) and DSC (blue line).](image)

![Figure 2. The sintering conditions of the green body.](image)
Each unit cell is $4 \times 4 \times 4$ mm$^3$. The thickness of the two structures (pcell, neoVius) is 0.2 mm. In order to investigate the influence of cell number in three directions of $x$, $y$, $z$ on compressive strength and deformation of the model, nine different structures with the same theoretical relative density have been designed. Unit cell of the samples are varied: $1 \times 1 \times 1$, $1 \times 1 \times 2$, $1 \times 1 \times 3$, $2 \times 2 \times 1$, $2 \times 2 \times 2$, $2 \times 2 \times 3$, $3 \times 3 \times 1$, $3 \times 3 \times 2$, $3 \times 3 \times 3$. For printing convenience, a 0.2 mm thickness perforated plate is added to the bottom of each model. Figure 2, 4 are the CAD design, and the Figure 3, 5 are the samples.

Figure 3. CAD image of pcell.

Figure 4. Sintered samples of the pcell.
2.3. Mechanical test
The mechanical behavior of ceramics is strongly dependent on the applied strain rate, material type, and sintering schedule. The loading direction was equivalent to printing direction. The compressive deformation rate was 0.2mm/min. This can be considered a quasi-static experiment. The base material is zirconia. It was tested to characterize its mechanical response and to use the obtained stress-strain curves to calibrate the constitutive models adopted in computations. In the case of compression loading, the specimens were tested until cracked.
3. Result and discussion

3.1. Characterization

Figure 7(a) shows the SEM image of sintered samples. There are no obvious pores on the surface of sintered samples. The grain boundary is obvious, and the size of the zirconia particles is uniform,
about 2 μm. Figure 7(b) shows that organics acted as impurities and might have formed porous structures during the debinding and sintering processes. The actual density of the samples measured by archimedean drainage method is all greater than 6.01g/cm³, in other words: the relative density is exceeded 99%. The theoretical relative density of pcell, neovius is 11.58%, 17.09%, respectively. Due to the limitation of printer resolution and the light scattering and absorption, the measured relative density is higher than the theoretical value. The actual relative density ρ is marked in the figure 8 and figure 9.

![Figure 9. The stress-strain curve of different neovius number.](image)

### 3.2. Mechanical property

Figure 8 shows the stress-strain curve of each specimen in the quasi-static mode composed of different unit cells. The length of side of the unit cell is 4 mm. For each row (a-c, d-f, g-i), the layers in Z axis is constant, and cells in X-Y plane was changed systematically. It can be seen from the figure that the compressive strength of each model is similar when the relative density ρ (volume ratio) is similar. When, X-Y plane remains unchanged, in other words, the stressed area remains unchanged and only the Z-axis is changed, it has no obvious influence on the deformation and compressive strength of the model. When the Z-axis is fixed, the increase of the stressed area will increase the deformation capacity of the model. In Fig.8 (i), there is an obvious plateau region, which has a significant effect on the increase of absorption energy. This is because the cell in the middle is constrained in three dimensions, which promotes stress dispersion. While, in other models, the cells are totally free from the boundary confinement or partially constrained by the neighboring cells, their strength and deformation are not as good as 3×3×3 samples. In general, the constraints induced by the neighboring cells has a positive effect on the compressive strength of the model and the increase of deformation.
Figure 9 shows the stress-strain curve of the neovius model, and each sample has the similar relative density. In each row of figure 6 (a-b-c, d-e-f, g-h-i), it is shown that when the number of unit cell in the Z-axis is constant, the deformation of the model increases with the increase of the number of unit cell in the X-Y plane. This is similar to the results of pcell. In each column of figure 6 (a-d-g, b-e-g, c-f-i), it is shown that when the X-Y plane has the same number cell, the deformation of neovius decreases with the increase of the number of unit cell in the Z-axis.

![Figure 10. The mechanical property of neovius compared with pcell.](image)

Figure 10 shows the comparison of compressive strength of neovius and pcell per gram under the same number of single cells. It showed that the mechanical strength of neovius was higher than that of pcell. The specific strength of only one single cell on the X, Y, Z axis was the highest. Under the condition of the same Z-axis, the specific strength of both models decreases with the increase of the number of X-Y planes. When the number of units in the X-Y plane is the same, the specific strength of the model decreases with the increase of the number of units in the Z-axis, but the rate of reduction is lower than that mentioned above.

4. Conclusions
For pcell, samples with similar relative densities exhibited comparable mechanical property in aspect of compressive strength. For the structure density range of 18-22%, a compressive strength of 3-7MPa can be achieved. The cell numbers included in the designed TPMS lattice structure can affect the strain (deformation) obviously. Linked with the constraint implemented on the cells, the increased number of cell arrays in X-Y plane can lead to a general increase in the strain before fracture. A more constrained structure can further increase the strain up to 7% when a $3\times3\times3$ lattice was built. Ceramics TPMS lattice can be very promising for potential energy absorption applications. For neovius, samples with relative density of 26%-30%, a 35MPa-65MPa compressive strength can be achieved. The influence of X-Y plane cell numbers on deformation is the same as the pcell. When the cell numbers on the X-Y plane is constant, the deformation of neovius structure decreases with the increase of the number of Z-axis cells, which is different from P-cell. The mechanical property of neovius is higher than pcell. In conclusion, the TPMS show superior properties when compared with other cellular structure, and thus are promising candidates for various technological applications.
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