

数字光处理制备金刚石结构多孔氮化硅陶瓷的孔径调控与力学性能

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摘要:背景 氮化硅(silicon nitride, Si₃N₄)是前景广阔的牙科生物陶瓷材料, 数字光处理(digital light processing, DLP)是高精度先进3D打印技术, 目前尚缺乏DLP成型Diamond型三周期极小曲面(triply periodic minimal surfaces, TPMS)多孔氮化硅牙科种植体的相关研究。目的 通过DLP技术制备300~600 μm孔径梯度的Diamond(金刚石)型TPMS结构多孔氮化硅陶瓷, 分析其微观结构与力学性能, 筛选适配牙科种植体的最优孔径参数。方法 基于Diamond型TPMS拓扑结构特征, 固定单元晶胞尺寸, 设计四组不同孔径(300、400、500、600 μm)的三维模型, 将各组总孔隙率波动严格控制在5%以内, 构建以孔径为唯一变量的单因素实验体系; 通过DLP技术打印坯体并经高温烧结制备样品, 每组设置5~6个平行样本。采用X射线衍射仪(X-ray diffractometer, XRD)、扫描电子显微镜(scanning electron microscope, SEM)表征材料物相组成与微观形貌, 通过阿基米德排水法测定总孔隙率, 依据ISO 14704-2016、GB/T 10700-2006标准, 借助万能力学试验机测试样品的抗弯强度与弹性模量, 采用单因素方差分析(one-way ANOVA)进行组间数据统计学比较。结果 所制备样品的主晶相为β-Si₃N₄, 宏观结构完整, 孔隙呈三维连通状态。随设计孔径增大, 样品总孔隙率从45.04%呈梯度升高至50.06%, 抗弯强度从(110.69±1.25) MPa单调降至(74.47±1.05) MPa, 弹性模量呈阶梯式非线性升高, 变化过程中存在小幅波动趋势, 所有核心力学指标组间差异均具有显著统计学意义($P<0.001$)。其中300 μm孔径组展现出最优的力学性能组合, 抗弯强度达到最高值(110.69±1.25) MPa, 同时弹性模量低至(13.45±0.20) GPa, 与人体颌骨皮质骨的力学参数范围高度匹配。结论 调控Diamond型TPMS结构的孔径参数, 可有效协同调控多孔氮化硅陶瓷的孔隙特征与力学响应特性。300 μm孔径结构实现了高强度与低模量的最佳平衡, 具备优异的骨组织力学适配性, 为高性能“致密核心——多孔表层”梯度氮化硅口腔种植体的多孔表层结构优化设计提供了实验依据与数据支撑。

关键词:氮化硅; 数字光处理; 三周期极小曲面; 牙种植体; 力学性能

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Aperture regulation and mechanical properties of diamond-structured porous silicon nitride ceramics fabricated by digital light processing

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Abstract: Background Silicon nitride (Si₃N₄) is a promising bioceramic material for dental applications, and digital light processing (DLP) is an advanced 3D printing (additive manufacturing) technology with high fabrication precision. However, there is a paucity of systematic investigations on DLP-fabricated porous Si₃N₄ dental implants with Diamond-type triply periodic minimal surfaces (TPMS) architecture. **Objective** To fabricate porous Si₃N₄ ceramics with Diamond-type TPMS architecture and a pore size gradient ranging from 300 to 600 μm via DLP technology, characterize their microstructure and mechanical properties, and identify the optimal pore size parameters suitable for dental implant applications. **Methods** Based on the topological characteristics of Diamond-type TPMS structure, three-dimensional models with four different pore sizes (300, 400, 500, and 600 μm) were designed with a fixed unit cell size. The fluctuation of total porosity among each group was strictly controlled within 5% to establish a single-factor experimental system with pore size as the sole variable. Green bodies were printed via DLP technology and sintered at high temperature to prepare specimens, with 5 - 6 replicate specimens set for each group. The phase composition and microstructure of the materials were characterized by X-ray diffractometer (XRD) and scanning electron microscope (SEM). Total

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porosity was measured by the Archimedes method. The flexural strength and elastic modulus of the specimens were tested using a universal mechanical testing machine in accordance with ISO 14704-2016 and GB/T 10700-2006 standards. One-way analysis of variance (ANOVA) was used for statistical comparison of data among groups. **Results** The main crystalline phase of the as-prepared samples was β - Si_3N_4 , with an intact macrostructure and a three-dimensionally interconnected pore network. As the designed pore size increased, the total porosity of the samples increased in a gradient manner from 45.04% to 50.06%, the flexural strength decreased monotonically from (110.69 ± 1.25) MPa to (74.47 ± 1.05) MPa, and the elastic modulus exhibited a stepwise non-linear increase accompanied by minor fluctuations throughout the variation. There were statistically significant differences in all core mechanical parameters among the groups ($P < 0.001$). Notably, the group with a designed pore size of 300 μm presented the optimal combination of mechanical properties, with the highest flexural strength of (110.69 ± 1.25) MPa and a low elastic modulus of (13.45 ± 0.20) GPa, which was highly consistent with the mechanical parameter range of human mandibular cortical bone. **Conclusion** Tailoring the pore size parameters of Diamond-type TPMS structure can effectively and synergistically regulate the pore characteristics and mechanical response properties of porous Si_3N_4 ceramics. The 300 μm pore size structure achieves the optimal balance between high strength and low modulus, with excellent mechanical compatibility with bone tissue. It provides core experimental basis and data support for the optimal design of the porous surface structure of high-performance "dense core-porous surface" gradient Si_3N_4 dental implants.

Keywords: silicon nitride; digital light processing; triply periodic minimal surfaces; dental implant; mechanical properties

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口腔种植修复的核心目标是实现种植体——骨界面的早期稳定与长期骨整合,理想材料需满足生物安全性、功能适配性与长期稳定性要求。钛及其合金作为临床“金标准”材料,存在弹性模量不匹配引发应力屏蔽^[1]、抗疲劳性不足^[2]、致敏风险^[3]及无固有抗菌性^[4]等固有缺陷。多孔结构虽可缓解应力屏蔽^[5]并促进骨整合^[6],但多孔钽、氧化锆、PEEK等替代材料均难以兼顾力学稳定性与骨整合性能^[7-8]。因此,兼具优异力学性能^[9]、生物相容性^[10]、成骨活性^[11]、抗菌效能^[12]与影像学阻射性^[13]的氮化硅成为新一代种植体重点研发材料,数字光处理(digital light processing, DLP)则是制备多孔氮化硅陶瓷的理想技术^[14]。

孔径是多孔植入物的核心参数,决定骨整合效能^[15]。目前关于最优孔径的研究已从单一固定值转向多因素协同调控,受研究体系差异影响尚未形成统一标准^[16]。体外实验证实,100~300 μm 孔径更利于成骨细胞早期黏附、铺展及分化^[17];300~600 μm 为兼顾成骨、血管化与力学适配的最优区间,该区间可上调RUNX2、ALP等成骨基因表达,保障深层营养供应并诱导新骨长入^[18-20]。

三周期极小曲面(triply periodic minimal surfaces, TPMS)可精准调控孔隙参数^[21],匹配颌骨模量^[22],避免应力集中^[23],并提供成骨微环境^[24];其中金刚石(diamond)型是骨植入体多孔结构的优选^[25],在相同孔隙率下力学与成骨性能更优^[26]。TPMS可通过数学参数精准调控孔隙特征,使弹性模量匹配骨组织,缓解应力屏蔽^[27-28];其光滑曲面可均化应力,提升抗疲劳性,三维连通孔

道利于营养交换与血管化^[29-30]。此外,TPMS体外可诱导成骨分化,体内骨整合优于传统晶格结构,且高比表面积支持表面功能化改性^[26,31-32]。

目前尚缺乏基于DLP成型的Diamond型TPMS多孔氮化硅种植体的孔径调控研究,本研究制备了孔径为300~600 μm 的梯度样品,探究孔径对其微观结构与力学性能的影响,以筛选最优孔径参数。

1 材料与方法

1.1 原料及设备

氮化硅(UBE,日本);氧化铝、氧化钇(上海阿拉丁生化科技股份有限公司,上海);光敏树脂单体1,6-己二醇二丙烯酸酯(HDDA)、乙氧基季戊四醇四丙烯酸酯(PPTTA)、环三羟甲基丙烷甲缩醛丙烯酸酯(CTFA)、光引发剂苯基双(2,4,6-三甲基苯甲酰基)氧化膦[德国良制化学(中国)有限公司,上海];分散剂 SOLSPERSE45000(艾尼韦尔,天津);数字光处理成型陶瓷打印机(LITHOZ,奥地利);超声震荡机(洁盟,深圳);管式炉(纳博热,德国);马弗炉(纳博热,德国);气氛压力烧结炉(岛津,日本);万能力学试验机(岛津,日本);扫描电镜(蔡司,德国);X射线衍射仪(布鲁克D8,德国);激光粒度分布仪(马尔文,英国)。

1.2 制备过程

选取300~600 μm 孔径区间,该范围兼顾牙科种植体的成骨、血管化与力学适配性,同时可规避DLP打印300 μm 以下孔径时易出现的固化不全、孔道粘连等成型缺陷。

基于 TPMS 原理, 采用隐式函数(公式 1)构建 Diamond 拓扑结构基础模型, 通过调整曲面水平集常数 C 精准调控孔径与孔隙率参数, 初始设计孔隙率为 50%, 式中 x 、 y 、 z 为三维空间坐标, C 为曲面水平集常数。

$$\sin(x)\sin(y)\sin(z) + \sin(x)\cos(y)\cos(z) + \cos(x)\sin(y)\cos(z) + \cos(x)\cos(y)\sin(z) = 0$$

利用三维建模软件设计不同孔径的 Diamond 结构多孔氮化硅试件, 力学性能测试用长条状试件尺寸统一设定为 3 mm×4 mm×36 mm, 根据设计孔径分别命名为 D300(300 μm)、D400(400 μm)、D500(500 μm)、D600(600 μm)。采用数字光处理陶瓷打印机进行坯体成型, 设定曝光能量为 900 mJ/cm³, 切片层厚为 20 μm; 坯体成型后于 600℃ 下进行脱脂处理, 随后以 2℃/min 的升温速率升至 1 670℃, 保温 2 h 后以 2℃/h 的冷却速率降至室温, 完成烧结过程。

1.3 表征与微观结构

采用激光粒度分布仪表征 Si₃N₄ 造粒粉末的粒度分布特征; 通过扫描电子显微镜 (scanning electron microscope, SEM) 观察烧结样品的微观形貌及孔隙结构; 利用 XRD 表征材料的物相组成; 采用阿基米德排水法测定试件的实际体积密度。采用 Image-Pro Plus 6.0 软件对 SEM 图像进行定量分析, 测定各组试件的实际孔径、支柱宽度与节点连接面积; 每组测试 5 个平行样本, 每个样本随机选取 5 个无重叠视场, 每个视场测量 20 个有效数据, 最终结果以 $\bar{x} \pm s$ 表述。

1.4 力学性能测试

采用万能力学试验机对多孔氮化硅试件进行力学性能表征, 重点测试抗弯强度与弹性模量。所有试件均经 2000 目砂纸精细抛光以消除表面缺陷及边缘效应, 每组测试至少包含 5 个有效样本, 以确保统计结果的可靠性。

抗弯强度测试依据国际标准 ISO 14704-2016 测定, 采用三点弯曲法。将试件水平置于跨距为 30 mm 的两个支撑圆柱上, 支撑圆柱与加载压头直径均为 5 mm, 确保试件长度方向与支撑辊轴线垂直; 上压头以 1 mm/min 的恒定速率垂直加载于试件跨距中点, 直至试件完全断裂, 同步记录最大断裂载荷 (P), 并通过公式 $\sigma = \frac{3PL}{2bh^2}$ 进行计算, 式中: σ 为试件抗弯强度 (MPa), P 为试件最大断裂载荷 (N), L 为三点弯曲支座跨距 (mm), b 为试件横截

面宽度 (mm), h 为试件横截面厚度 (mm)。

弹性模量测试参照中国国家标准 GB/T 10700-2006 (弯曲法), 在与抗弯强度测试相同的三点弯曲装置上完成测试。测试前, 在试件跨距中央的受拉面粘贴对应栅长的电阻应变片, 采用高精度应变采集仪同步采集试件应变数据, 以 0.1 mm/min 的恒定速率匀速加载, 加载上限为同组试件预测试抗弯强度的 70%, 确保加载全过程中载荷-应变曲线处于材料的线性弹性阶段, 同步采集线性段内的载荷与对应实测应变数据, 利用公式 $E = \frac{3L(P_2 - P_1)}{2bh^2(\varepsilon_2 - \varepsilon_1)} \times 10^{-3}$ 计算得出弹性模量, 式中: E 为试件弹性模量 (GPa); P_1 、 P_2 分别为线性弹性段内的起始与终止载荷 (N); L 为三点弯曲支座跨距 (mm); b 为试件横截面宽度 (mm); h 为试件横截面厚度 (mm); ε_1 、 ε_2 分别为 P_1 、 P_2 对应的试件跨中实测应变 (mm/mm)。

1.5 统计学分析

采用 SPSS 26.0 统计学软件对本研究所有定量实验数据进行处理与分析。体积密度、总孔隙率测试每组设置 6 个平行样本, 抗弯强度、弹性模量测试每组设置 5 个有效平行样本, 所有计量资料均以 $\bar{x} \pm s$ 形式表述。数据先行采用 Shapiro-Wilk 检验验证正态分布性, 采用 Levene 检验验证方差齐性; 符合正态分布且方差齐性的多组间计量资料比较, 采用单因素方差分析, 组间两两多重比较采用 Tukey 检验。以 $P < 0.05$ 为差异有统计学意义。

2 结果

2.1 表征与微观结构

氮化硅造粒粉末的粒度分布结果及造粒粉末 SEM 图像显示, 其体积平均粒径为 2.672 μm, 中位粒径 (D50) 为 2.353 μm, 且 52.48% 的颗粒集中于 2.000 ~ 5.000 μm, 该粉末粒度分布相对集中, 有利于 DLP 打印工艺中粉末的均匀分散与流动, 为复杂 TPMS 结构的高精度成型奠定基础。见图 1。

X 射线衍射分析结果表明, 经 DLP 成型及后续烧结处理的多孔氮化硅材料, 其衍射谱图与 β-Si₃N₄ 标准卡片 (PDF #33-1160) 完全吻合, 未检测到明显杂质相, 证实材料主晶相为 β-Si₃N₄, 烧结过程实现了 Si₃N₄ 的充分晶化。见图 2。

宏观形貌观察显示, 所制备的 Diamond 型 TPMS 结构多孔试件与原始三维设计模型高度一致, 呈现出完整、连续且均匀的三维互穿网络结

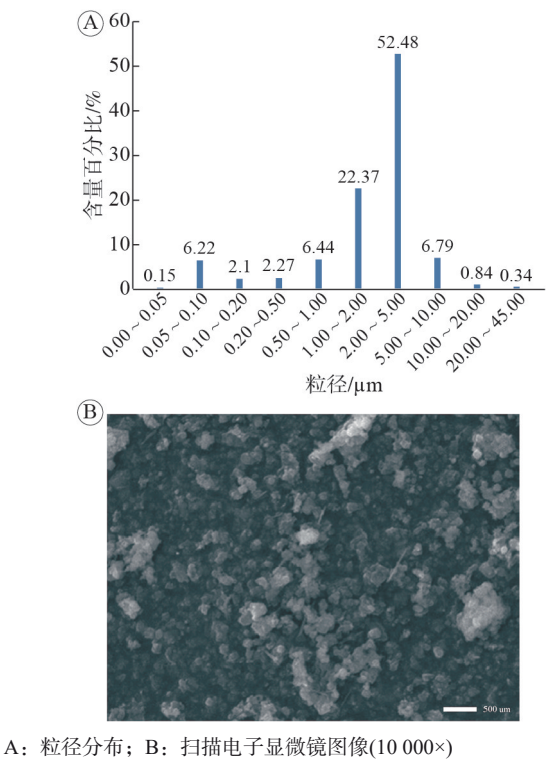


图 1 氮化硅造粒粉末表征

Fig. 1 Characterization of Si₃N₄ granulated powder

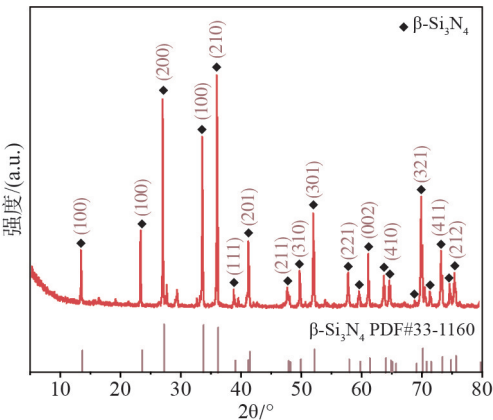


图 2 DLP成型氮化硅陶瓷烧结体的 X 射线衍射图谱

Fig. 2 X-ray diffraction pattern of DLP-fabricated Si₃N₄ ceramic sintered body

构,无明显宏观缺陷(如裂纹、坍塌),表明DLP技

表 1 不同孔径样品的物理与力学性能测试结果

Tab.1 Physical and mechanical properties of samples with different pore sizes

变量	D300	D400	D500	D600	F 值	P 值
体积密度/(g/cm ³)	1.787±0.004	1.652±0.005 ^a	1.643±0.004 ^{ab}	1.627±0.004 ^{abc}	1826.23	<0.001
总孔隙率/%	45.04±0.13	49.19±0.18 ^a	49.45±0.15 ^{ab}	50.06±0.19 ^{abc}	1027.58	<0.001
实际孔径/μm	252.0±44.8	355.0±60.0 ^a	444.6±70.7 ^{ab}	540.0±75.0 ^{abc}	213.47	<0.001
抗弯强度/MPa	110.69±1.25	91.25±1.31 ^a	81.76±1.18 ^{ab}	74.47±1.05 ^{abc}	942.65	<0.001
弹性模量/GPa	13.45±0.20	46.51±0.58 ^a	42.61±0.72 ^{ab}	58.47±0.65 ^{abc}	5219.32	<0.001

结果以 $\bar{x}\pm s$ 表示,所有指标组间整体差异经ANOVA分析,组间两两多重比较采用Tukey检验;^a表示与同指标D300组相比($P<0.05$);^b表示与同指标D400组相比($P<0.05$);^c表示与同指标D500组相比($P<0.05$)。所有核心指标组间整体差异均有显著统计学意义($P<0.001$)。

术对复杂三周期极小曲面结构具有良好的成型保真度(图3)。

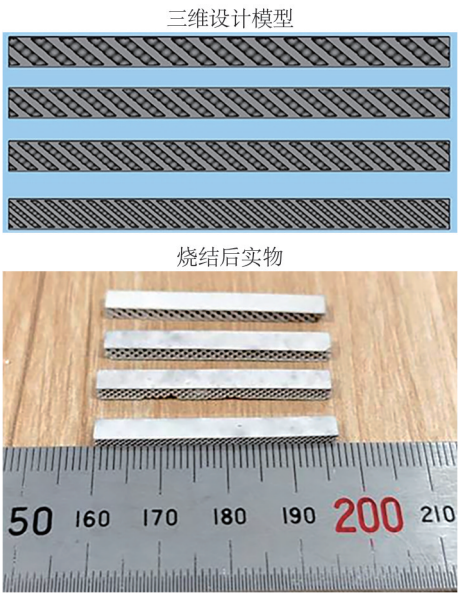


图 3 Diamond型TPMS结构三维设计模型与烧结后实物对比图

Fig. 3 Comparison of the 3D design model and the sintered physical specimen of Diamond-type TPMS structure

SEM观察结果(图4)显示,烧结后的Diamond结构表面光滑、拓扑曲面连续,孔隙连通性良好;随孔径增大,孔壁厚度同步增加,孔壁致密化程度提升,小孔径组孔隙边缘可观察到少量亚微米级微孔及未完全致密化的原料附着颗粒,提示后续可通过优化粉体预处理工艺及烧结参数减少表面缺陷。

2.2 不同孔径样品的物理与力学性能

Diamond型TPMS结构的孔径参数对多孔氮化硅陶瓷的物理与力学性能具有极显著的调控作用(所有核心指标组间差异均 $P<0.001$),见表1。随设计孔径从300 μm增至600 μm,试件体积密度呈梯度递减,孔隙率与实际成型孔径同步梯度增大,抗弯强度呈单调显著下降趋势,弹性模量整体呈非线性波动上升趋势,仅在500 μm孔径组出现小

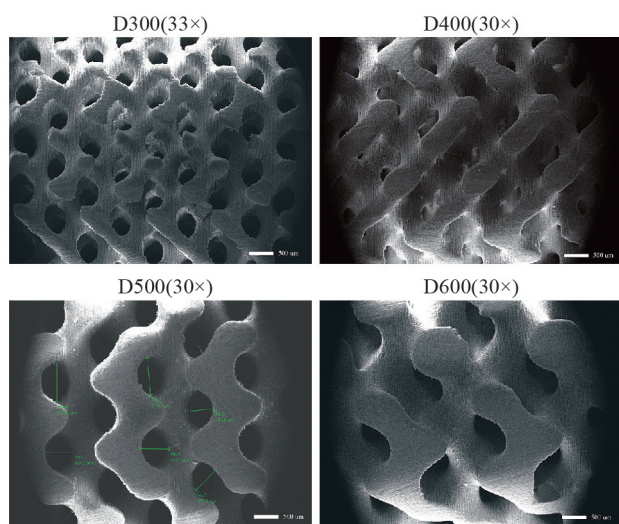


图4 不同孔径Diamond型TPMS结构多孔氮化硅陶瓷的扫描电子显微镜图像

Fig. 4 Scanning electron microscopic images of porous Si_3N_4 ceramics with Diamond-type TPMS structures and different pore sizes

幅回落。其中, 300 μm 孔径组实现了高强度与低弹性模量的最优平衡, 其力学性能与人体颌骨高度匹配, 为高性能口腔种植体的结构优化设计提供了关键实验支撑。

3 讨论

本研究基于DLP技术制备了Diamond型TPMS多孔氮化硅陶瓷, 孔径梯度调控与理想孔径范围高度契合; 总孔隙率为45.04%~50.06%。

孔隙率与材料性能关系的核心调控机制分为3个层面。其一, 总孔隙率升高致基体承载面积缩减是强度下降的核心原因, 多孔脆性陶瓷抗弯强度与有效承载面积正相关^[33]; 本研究总孔隙率虽波动仅5%, 但经孔径放大效应削弱了承载能力, 与多孔陶瓷强度-孔隙率幂函数规律一致^[34]。其二, 孔径增大致缺陷临界尺寸扩张与应力集中加速材料失效, 脆性陶瓷断裂强度由内部最大缺陷尺寸决定^[35]; 本研究梯度孔径构成材料内部规则预制缺陷, 孔径增大直接提升缺陷临界尺寸, 且大孔径孔边曲率半径更小、应力集中系数更高, 更易诱发微裂纹萌生与扩展, 该机制已在氧化铝、氧化锆体系中得到验证^[36]。其三, TPMS支柱变形模式转变放大强度衰减效应, 固定晶胞尺寸下, 孔径增大伴随支柱高径比提升, 弯曲载荷下支柱变形从以轴向承载为主转向以弯曲变形为主, 加剧节点应力集中, 加速支柱屈曲断裂; 该规律已在Diamond型TPMS结构研究中被证实, 也印证了其

骨植入物力学适配优势^[37-38]。

Gibson-Ashby模型证实弹性模量随孔隙率升高呈幂律下降, 但本研究中总孔隙率仅升高不足5%, 样品弹性模量却呈阶梯式非线性升高^[39]。这一反常现象源于Diamond型TPMS拓扑变形主导效应与DLP工艺致密化效应协同作用。Diamond型TPMS拓扑变形主导效应是弹性模量反常升高决定性因素。与随机多孔泡沫的弯曲主导变形机制不同, Diamond为典型的拉伸主导型有序拓扑结构; 在Gibson-Ashby模型中, 随机开孔泡沫弹性模量-相对密度幂律指数 $n \approx 2$, 而拉伸主导型TPMS结构 $n \approx 1$, 对孔隙率小幅变化的敏感性较低; 因此, 本研究中不足5%的孔隙率波动对模量的衰减效应微弱, 为拓扑调控提供了空间。在固定晶胞尺寸下, 孔径增大伴随支柱宽度、节点连接面积同步扩大, TPMS弹性模量核心取决于拓扑节点连接性与应力传导效率^[40]; 节点连接面积扩大可优化应力分布、降低节点应力集中, 使弯曲载荷更高效地经轴向拉伸/压缩传递, 提升结构抗变形能力, 该拓扑优化带来的模量提升效应完全覆盖了孔隙率小幅升高导致的衰减效应, 致使宏观弹性模量显著升高, 印证了拓扑结构是有序多孔材料模量的核心决定因素^[41]。

微观孔壁致密化叠加效应进一步放大了拓扑优化对模量的提升作用。多孔氮化硅陶瓷的宏观弹性模量同时取决于宏观拓扑结构、孔壁致密化程度及微观缺陷数量^[42]。一方面, DLP打印中陶瓷粉末对紫外光的强散射易导致小孔径结构出现孔壁固化不全、孔道粘连及生坯微裂纹等缺陷, 显著削弱孔壁有效刚度^[43]; 而大孔径结构因孔道空间更充足、浆料填充更均匀, 光散射干扰显著降低, 生坯质量与孔壁完整性更优, 为烧结后获得高模量奠定基础, 该现象已在DLP制备羟基磷灰石体系中得到验证^[44]。另一方面, 固定拓扑与晶胞尺寸时, 增大孔径结构可通过降低陶瓷实体相占空比, 缓解DLP打印中陶瓷粉体紫外光侧向散射, 提升生坯质量与孔壁完整性, 进而减少排胶阶段孔壁缺陷, 保障烧结液相扩散、晶粒生长与孔壁致密化^[45]。致密氮化硅陶瓷本征弹性模量可达288~300 GPa, 孔壁致密化提升可充分发挥基体本征刚度, 进一步放大拓扑优化模量提升效应。

本研究制备的多孔氮化硅陶瓷, 其孔径范围与力学性能精准匹配口腔种植体的核心临床需求, 为新型氮化硅种植体的多孔表层结构优化设计提供了实验依据。适配钛合金过敏、应力屏蔽致骨

吸收、种植体周围炎高风险患者的临床需求,同时DLP技术的个性化成型优势也为该材料的临床转化提供了工艺支撑。

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利益冲突 所有作者声明无利益冲突。

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