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热处理对激光选区熔化成型纯钛钛瓷结合强度的影响

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【摘要】 **目的** 探讨热处理对激光选区熔化（selective laser melting，SLM）成型纯钛钛瓷结合强度的影响。**方法** 利用SLM技术制作ISO 9693标准要求的纯钛试件96个，分为热处理组（A）和未热处理组（B）。A、B组试件根据瓷粉种类分为Super Ti22（a）、Titankeramik（b）、Triceram（c）组。再根据喷砂压力0.25 MPa(1)，0.45 MPa（2），最终分为Aa1、Aa2、Ab1、Ab2、Ac1、Ac2及Ba1、Ba2、Bb1、Bb2、Bc1、Bc2组。使用激光扫描共聚焦显微镜观察喷砂试件表面形貌及测量粗糙度；烤瓷后测试三点弯曲钛瓷结合强度；使用体视显微镜观察瓷层剥脱后的钛表面形貌并分析其断裂模式。**结果** A组维氏硬度（188.21 ± 11.94）HV低于B组维氏硬度（204.48 ± 6.32）HV（$P < 0.05$）。粗糙度值A1组（2.90 ± 0.32）μm，A2组（3.43 ± 0.43）μm，差异有统计学意义（$P < 0.05$）；B1组（2.62 ± 0.08）μm，B2组（3.01 ± 0.06）μm，差异有统计学意义（$P < 0.05$）。A组结合强度均高于B组[Aa1组(33.75 ± 2.31)MPa，Aa2组(36.32 ± 1.44)MPa，Ab1组(39.82 ± 2.28)MPa，Ab2组(33.74 ± 1.53)MPa，Ac2组(38.63 ± 1.36)MPa；Ba1组(29.65 ± 1.10)MPa、Ba2组(27.17 ± 2.24)MPa、Bb1组(27.29 ± 1.61)MPa、Bb2组(23.85 ± 0.97)MPa、Bc2组(35.75 ± 1.93)MPa（$P < 0.05$）]；随着喷砂压力的增大，钛瓷结合强度Aa2组高于Aa1组，Ab2组低于Ab1组（$P < 0.05$）；Ba2、Bb2、Bc2组结合强度低于对应的Ba1、Bb1、Bc1组结合强度。A组及Bc1、Bc2组为混合断裂，Ba1、Ba2、Bb1、Bb2组为界面断裂。**结论** 热处理可降低SLM纯钛的维氏硬度；SLM纯钛热处理后有利于三种瓷粉与纯钛的结合；喷砂压力对钛瓷结合强度有影响。

【关键词】 激光选区熔化； 固定修复； 纯钛； 瓷粉； 热处理； 喷砂； 钛瓷结合； 结合强度； 粗糙度； 维氏硬度； 三点弯曲； 断裂模式
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Effects of heat treatment on the bonding strength of pure titanium processed *via* selective laser melting and porcelain

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【Abstract】 **Objective** To evaluate the effect of heat treatment on the bonding strength of pure titanium formed by selective laser melting (SLM) and porcelain. **Methods** Ninety-six pure titanium specimens were laser machined to meet ISO 9693 standards. The specimens were divided into a heat treated group (A) and a nonheat treated group (B). According to the porcelain type, the specimens in groups A and B were divided into Super Ti22 (a), Titankeramik (b), and Triceram (c) groups. Then, according to sandblasting pressures of 0.25 MPa (1) and 0.45 MPa (2), they were further divided into Aa1, Aa2, Ab1, Ab2, Ac1, Ac2, Ba1, Ba2, Bb1, Bb2, Bc1, and Bc2 groups. The surface morphology and roughness of the sandblasted specimens were assessed using a laser scanning confocal microscope. After the porcelain

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was fused, the three-point bending titanium-porcelain bonding strength was tested. A stereomicroscope was used to characterize the titanium-porcelain interfaces and determine the mode of failure. **Results** The Vickers hardness of group A specimens (188.21 ± 11.94) was significantly lower than that of group B specimens (204.48 ± 6.32) HV (P < 0.05). The roughness value in group A1 (2.90 ± 0.32) μm was significantly lower than that in group A2 (3.43 ± 0.43) μm (P < 0.05). Specimens in group B1 (2.62 ± 0.08) μm were significantly smaller than those in group B2 (3.01 ± 0.06) μm (P < 0.05). The bonding strength in group Aa1 was (33.75 ± 2.31) MPa, group Aa2 was (36.32 ± 1.44) MPa, group Ab1 was (39.82 ± 2.28) MPa, group Ab2 was (33.74 ± 1.53) MPa and group Ac2 was (38.63 ± 1.36) MPa, which was significantly higher than that in the corresponding groups Ba1 (29.65 ± 1.10) MPa, Ba2 (27.17 ± 2.24) MPa, Bb1 (27.29 ± 1.61) MPa, Bb2 (23.85 ± 0.97) MPa, and Bc2 (35.75 ± 1.93) MPa (P < 0.05). With increasing sandblasting pressure, the bonding strength of the titanium ceramic in group Aa2 was significantly higher than in group Aa1, while that in group Ab2 was significantly lower than that in group Ab1 (P < 0.05). In groups A, Bc1 and Bc2, the fracture model showed mixed failure, while in groups Ba1, Ba2, Bb1, and Bb2, the model showed interfacial failure. **Conclusion** The Vickers hardness of SLM titanium can be significantly reduced by heat treatment. SLM pure titanium after heat treatment is beneficial to combination of the three porcelain types and titanium. The titanium-porcelain bonding strength may be affected by sandblasting pressure.

【Key words】 selective laser melting; fixed prosthesis; pure titanium; porcelain; heat treatment; sandblasting; titanium-porcelain bonding; bonding strength; roughness; Vickers hardness; three-point bending; fracture mode

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增材制造(additive manufacturing，AM)技术又称3D打印，可成型复杂且精度高的三维形状物件[1]。激光选区熔化(selective laser melting，SLM)是利用激光束的热作用使粉末快速熔化、快速凝固成型的一种AM技术，其制作的钛修复体性价比高、生产效率高、质量高[2-3]。与传统铸造工艺涉及繁复的加工操作和低的材料利用率相比，具有明显优点，可替代钛的传统铸造工艺。然而SLM技术由于快速冷却过程中存在较大的温度梯度差，导致了热应力的积累，易导致试件变形、卷翘[4]。而理想的热处理工艺可以消除金属晶体结构缺陷，使内部结构更加均匀，部分或完全释放成型过程中产生的残余应力，是目前解决SLM技术残余应力问题的有效方法，可达到稳定组织、优化性能的目的[5]。

纯钛因其良好的机械性能和优异的生物相容性，是牙科金属烤瓷修复理想的基底材料，在种植修复可避免异种金属电化学腐蚀情况。然而目前对于SLM工艺以及后热处理对其钛瓷结合强度的影响尚未明确，因此本实验主要研究热处理对SLM纯钛钛瓷结合强度的影响，以期为获得SLM纯钛相关所需性能的后热处理标准的制定打下基础。

1 材料和方法

1.1 主要材料和设备

钛专用烤瓷粉Super Porcelain Ti22(Noritake，日本)；Titankeramik(Vita，德国)；Triceram(Dentuarum，德国)；纯钛粉末TA1(15 ~ 53 μm，江苏威拉里新材料科技有限公司，中国)；激光3D打印设备(Tr150，南京前知智能科技有限公司，中国)；纯钛热处理炉(RZF1100-14-230S，上海热凡高温设备有限公司，中国)；110 μm氧化铝喷砂剂(Renfert，德国)；笔式喷砂机(P-G 400，Harnisch+Rieth，日本)；蒸汽清洗机(STEAMER X3，德国)；数控超声波清洗器(KQ-250DE，昆山超声仪器有限公司，苏州)；烤瓷炉(Programat P700，Ivoclar，列支敦士登)；维氏硬度仪(WHW Microcre Optics-Mech，上海研润光机科技有限公司，中国)；万能测试机(MTS，MTS Systems，美国)；体式显微镜(SMZ 1500，Nikon，日本)；激光扫描共聚焦显微镜(laser scanning confocal microscope，LSCM)(LSM800，Zeiss，德国)。

1.2 实验方法试件制备

应用Rhino5.0软件设计25.0 mm×3.0 mm×0.6 mm的三维模型，排版后打印。制备96个纯钛试件。热处理参数为700 ℃，2 h，炉冷。线切割试件，去支撑，打磨支撑突，SIC砂纸240、320、480、

600目依次湿润打磨。根据热处理与否分为热处理组(A)、未热处理组(B)。根据瓷粉种类,细分为Super Ti22(a)、Titankeramik(b)、Triceram(c)。根据喷砂压力0.25MPa(1组),0.45MPa(2组),最终分为Aa1、Aa2、Ab1、Ab2、Ac1、Ac2组及Ba1、Ba2、Bb1、Bb2、Bc1、Bc2组,每组8个试件。

1.3 维氏硬度测量

A组与B组,每组5个试件,每个试件测试5个相距明显的位点的维氏硬度。加压载荷10 kg,保压时间15 s。

1.4 喷砂后钛表面粗糙度测量

使用LSCM测量A组与B组打磨至600目后0.25 MPa、0.45 MPa压力喷砂的纯钛试件表面粗糙度;各组随机选择三个试件,每个试件测量两个位点。

1.5 喷砂及低熔瓷粉的烧结

在0.25 MPa、0.45 MPa压力下使用110 μm Al_2O_3颗粒对钛表面喷砂,喷嘴与试件表面呈45°,间距10 mm喷砂15 s。喷砂后蒸汽清洗,丙酮、75%酒精、去离子水超声清洗5 min,干燥备用。瓷粉为Super Ti22、Titankeramik、Triceram。将调匀的瓷粉均匀涂覆于试件中央长8 mm喷砂处,依次烧结粘接瓷(0.15~0.2 mm)、遮色瓷(0.15~0.2 mm)、体瓷并进行自上釉,瓷层总厚度1.0 mm,操作均由一人完成。

1.6 钛瓷结合强度测试

采用三点弯曲法测试试件的钛瓷结合强度,将试件置于万能试验机加载台,瓷面朝向地面,两支点半径为1.0 mm,支点间距为20.0 mm,加载头半径为1.0 mm,施加垂直向金属面的力,加载速度为0.5 mm/min。记录最大加载力F,计算弯曲强度(τ),即结合强度,τ=k×F(MPa),k为常数。

1.7 钛-瓷结合界面断裂模式观察

对已测试过结合强度的试件,使用尼龙小毛刷刷除残留在钛表面的碎瓷块。利用体式显微镜放大7.5倍观察已断裂试件的断裂模式。断裂模式分类:①界面断裂:断裂发生在钛、瓷或涂层的交界面上;②内聚断裂:即断裂发生在钛基底、瓷层或者涂层内部;③混合断裂:即存在以上两种断裂模式。

1.8 统计学分析

使用Graphpad 8.0软件进行统计分析,采用独立样本t检验对各组瓷粉的结合强度进行比较,采用配对样本t检验对热处理前后试件的结合强度及维氏硬度进行比较,检验水准α=0.05。

2 结 果

2.1 维氏硬度

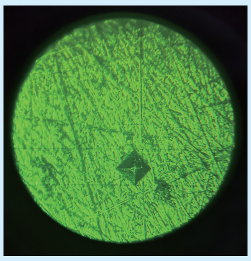
维氏硬度值见表1。A组的维氏硬度值为(188.21 ± 11.94)HV,B组的维氏硬度值为(204.48 ± 6.32)HV,差异有统计学意义(t=3.926, P=0.017)。维氏硬度压痕可见中央为"+"字的菱形压痕,测量"+"字的两条长轴长度,用以计算维氏硬度(图1)。

表1 热处理组与未热处理组纯钛表面维氏硬度

Table 1 Vickers hardness of specimens in the heat-treated group and the nonheat-treated group

Groups	Vickers Hardness/HV					$\bar{x} \pm s$	t	P
A	182.02	173.76	184.46	202.84	197.96	188.21 ± 11.94	3.926	0.017
B	208.68	193.30	206.08	206.84	207.48	204.48 ± 6.32		

A: heat-treated group; B: nonheat-treated group



The diamond shape is vickers hardness indentation

Figure 1 Indentation for Vickers hardness test

图1 维氏硬度压痕

2.2 钛表面粗糙度

钛表面粗糙度值见表2。A组组间粗糙度值差异有统计学意义(t=2.477, P=0.033),B组组间粗糙度值差异有统计学意义(t=9.674, P < 0.001)。表面粗糙度三维形貌可见A组及B组0.45 MPa喷砂纯钛表面凹陷较0.25 MPa深,三维形貌图中黄色及红色区域较多(图2)。

2.3 钛瓷结合强度

三点弯曲实验模式图见图3 。A组(Aa1,Aa2,Ab1,Ab2,Ac1,Ac2)钛瓷结合强度均高于对应的B组(Ba1,Ba2,Bb1,Bb2,Bc1,Bc2)(表3),随着喷砂压力的增加,A组三种钛专用瓷粉与SLM纯钛的

表2　热处理组与未热处理组0.25 MPa、0.45 MPa压力喷砂的纯钛试件表面粗糙度

Table 2　Surface roughness of pure titanium specimens in the heat-treated group and the nonheat-treated group with 0.25 MPa and 0.45 MPa sandblasting pressure　$\bar{x} \pm s$, n=6

Groups	Surface roughness/Sa	t	P
A1	2.90 ± 0.32	2.477	0.033
A2	3.43 ± 0.43		
B1	2.62 ± 0.08	9.674	< 0.001
B2	3.01 ± 0.06		

A: heat-treated group; B: nonheat-treated group; 1: 0.25 MPa sandblasting pressure; 2: 0.45 MPa sandblasting pressure

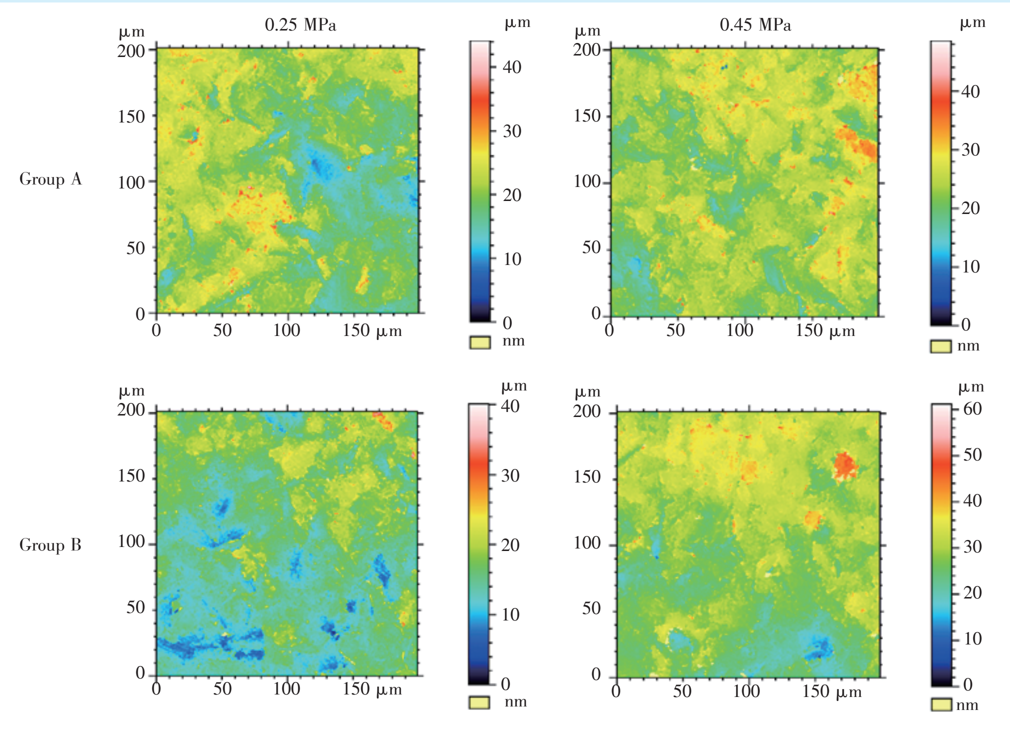
结合强度呈现不同的趋势：喷砂压力的增大，增加了Aa组的钛瓷结合强度，降低了Ab组钛瓷结合强度，对Ac组钛瓷结合强度无显著影响；B组的钛瓷结合强度降低，Ba2、Bb2、Bc2组结合强度低于对应的Ba1、Bb1、Bc1组结合强度（$P < 0.05$，图4）。

2.4　断裂模式

Ba组与Bb组主要偏向于界面断裂，断裂的钛表面基本无瓷块残留；A组的三种瓷粉组及Bc组的断裂模式为混合断裂，钛表面有白色瓷块残留，同时Ab组有明显的黑色粘接瓷残留（图5）。

3　讨　论

SLM制作的单冠边缘适合性与铸造纯钛冠无差异甚至效果更佳[6]。有研究表明，SLM快速冷却产生针状马氏体相细化晶粒[7]。由于细晶强化现象[8]，该技术成型纯钛具有较好的强度和韧性[9]，力学性能能够达到ASTM标准对于锻造态Ti-6Al-4V合金的要求[10]。然而，SLM试件内部存在较大的残余热应力，多层小熔池的快速凝固会产生亚稳定组织及内部缺陷[11]，使试件塑性和疲劳性能降低，表面硬度升高。(700 ± 10)℃下进行2 h热处理（退火）的条件被广泛应用于锻造的钛材料[12]。热处理使SLM纯钛内部发生再结晶，结构更加均匀，使晶相转变为延相α相[2]，延伸率得以提高，硬度降低[13]。高性价比SLM纯钛通过合适的热处理



Group A: heat-treated group; Group B: nonheat-treated group. The surface depressions of 0.45 MPa sandblasted pure titanium were deeper in group A and group B than in specimens sandblasted at 0.25 MPa, and more yellow and red areas were observed in the 3D topography (×200, LSCM); LSCM: laser scanning confocal microscope

Figure 2　Surface morphology of pure titanium specimens in the heat-treated group and nonheat-treated group sandblasted with 0.25 MPa and 0.45 MPa sandblasting pressure

图2　热处理组与未热处理组0.25 MPa、0.45 MPa压力喷砂的纯钛试件表面形貌

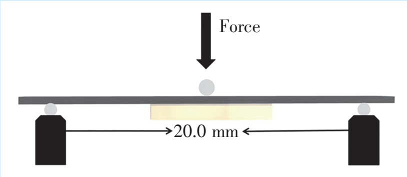
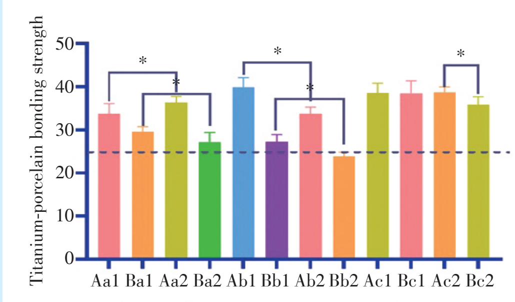


Figure 3 Pattern diagram of the three-point bending test
图3 三点弯曲试验模式图

表3 热处理与未热处理纯钛试件与三种饰瓷结合强度
Table 3 The bonding strength of heat-treated and unheat-treated pure titanium specimens with three types of veneering porcelain $\bar{x} \pm s$, n=8, MPa

Groups	Bonding strength		t	P
	A	B		
a1	33.75 ± 2.31	29.65 ± 1.10	4.228	0.004
a2	36.32 ± 1.44	27.17 ± 2.24	8.701	< 0.001
b1	39.82 ± 2.28	27.29 ± 1.61	11.200	< 0.001
b2	33.75 ± 2.31	23.85 ± 0.97	25.820	< 0.001
c1	38.54 ± 2.28	38.48 ± 2.89	0.044	0.967
c2	38.63 ± 1.36	35.75 ± 1.93	4.471	0.003

A: heat-treated group; B: nonheat-treated group; a: Super Ti22; b: Titankeramic; c: Triceram; 1: 0.25 MPa sandblasting pressure; 2: 0.45 MPa sandblasting pressure

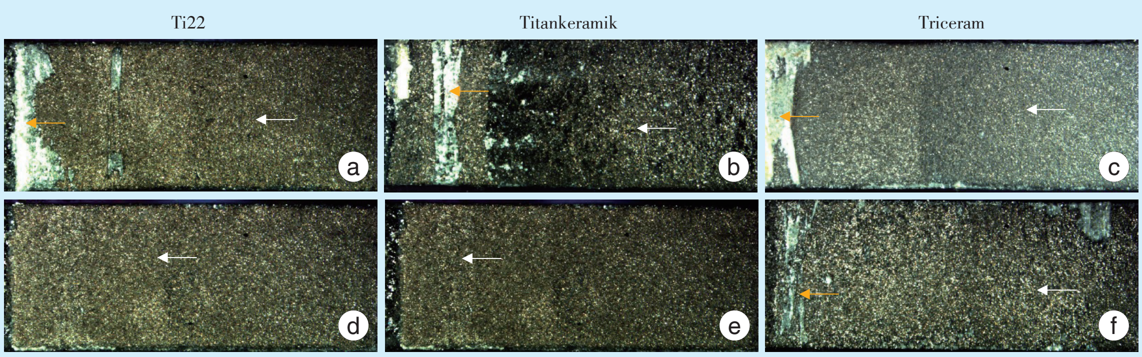


A: heat-treated group; B: nonheat-treated group; a: Super Ti22; b: Titankeramic; c: Triceram; 1: 0.25 MPa sandblasting pressure; 2: 0.45 MPa sandblasting pressure; *: $P < 0.05$
Figure 4 Titanium-porcelain bonding strength in each group
图4 钛瓷结合强度

既保留了较高的强度亦获得了合适的延展性，有利于钛材料在牙科固定及可摘修复领域的进一步发展。如种植冠方修复中央螺丝的主动就位，一定的弹性形变可避免刚性就位带来的不利影响，并且钛瓷修复体是种植修复的理想材料，可避免异种金属在口腔内的电化学腐蚀。由于市售的钛专用瓷粉种类不一，成分存在差异，通过研究相关后热处理对3种钛专用瓷粉的钛瓷结合强度的影响，为临床瓷粉选择提供参考。

Iseri等[14]比较了铸造、铣削和激光烧结钛与牙科饰瓷的剪切结合强度，应用Titankeramik饰瓷及Triceram饰瓷时，结合最佳的均为激光烧结钛合金组。李瑶[15]发现Titankeramik饰瓷与SLM纯钛烤瓷的三点弯曲强度高于传统失蜡铸造技术。这与有关学者认为陶瓷与钛合金的结合强度受金属加工工艺影响的观点一致，认为激光加工钛可获得更佳的钛瓷结合强度[16]。然而这些涉及钛瓷结合的研究均未说明打印试件的热处理情况。目前关于激光加工纯钛的热处理方面研究多集中在其力学性能和显微晶相变化方面，尚未见热处理对SLM纯钛钛瓷结合强度的影响。

本研究热处理后的SLM纯钛维氏硬度显著低于未进行热处理的纯钛，同时热处理后试件的钛



a-c: heat-treated pure titanium specimens, the fracture model showed mixed failure; d-f: nonheat-treated pure titanium specimens, the model showed interfacial failure; yellow arrow shows the remaining veneering porcelain; white arrow shows the titanium substrate (×7.5)
Figure 5 Typical fracture modes of the three types of porcelain
图5 三种瓷粉的典型断裂模式

瓷结合强度高于未热处理组的钛瓷结合强度。Li 等[17]研究表明,金瓷结合强度与合金表面层的硬度成反比。在金瓷界面层硬度较低的区域,裂纹沿合金延性较高的区域扩散时需要消耗额外的塑性性能,因此金瓷界面区的结合强度较高。Tulga[18]认为由于热处理后钴铬合金延展性提高,材料软化,金瓷结合强度得以提高。这可能也是提高钛瓷结合强度的原因之一。本研究随着喷砂压力的增大,钛表面粗糙度增大,B组的钛瓷结合强度却随着喷砂压力的增大显著降低,可能原因是未进行热处理的纯钛表面硬度较高,喷砂后存在尖锐边嵴,易造成应力集中;而A组3组瓷粉的钛瓷结合强度表现出不同的趋势,可能与瓷粉成分相关,如促进熔融瓷流动的元素种类及含量等[19]。Zhou等[20]发现利用SLM制备的钛合金的抗氧化性能优于铸造钛合金。而热处理使SLM纯钛晶粒尺寸增大,结构均匀[7],也可能有利于钛瓷结合。

未热处理的SLM纯钛维氏显微硬度高于热处理后的SLM纯钛。在同一喷砂压力下,热处理后的SLM纯钛与三组瓷粉结合强度均高于未进行热处理的SLM纯钛的钛瓷结合强度,热处理可提高SLM纯钛与三种瓷粉的钛瓷结合强度。未来将进一步研究不同热处理参数对钛瓷结合强度以及对SLM纯钛显微结构及力学性能的影响。

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（编辑　罗燕鸿）

官网

·短讯·

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