

中国激光
Chinese Journal of Lasers
ISSN 0258-7025, CN 31-1339/TN

《中国激光》网络首发论文

题目：激光冲击强化对激光增材制造 TC4 钛合金组织和性能的影响
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网络首发日期：2022-07-18
引用格式：陈雪鹏，张凌峰，熊毅，罗高丽，武永丽. 激光冲击强化对激光增材制造 TC4 钛合金组织和性能的影响[J/OL]. 中国激光.
<https://kns.cnki.net/kcms/detail/31.1339.tn.20220713.1850.100.html>



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激光冲击强化对激光增材制造 TC4 钛合金 组织和性能的影响

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摘要 对激光增材制造 (Laser additive manufacturing, LAM) TC4 钛合金表面进行激光冲击强化 (Laser shock peening, LSP), 对比研究了 LSP 处理对 LAM-TC4 钛合金微观组织、力学性能和断口形貌的影响。LAM-TC4 钛合金原始组织由大量粗大的 α 板条及一定体积分数的板条间 β 相构成。经 LSP 处理后, 表层组织在高速冲击波的作用下, 原始粗大的 α 板条被破碎细化, 形成了大量位错、形变孪晶, 导致晶格畸变。LSP 处理, 使 LAM-TC4 钛合金的残余应力由 LAM 形成的残余拉应力转变成残余压应力。LSP 处理后 LAM-TC4 钛合金表面存在最大残余压应力 (-190 MPa), 显微硬度提高了 16.5%, 且呈现沿深度梯度变化的特征。此外, 经 LSP 处理后 LAM-TC4 钛合金的屈服强度和抗拉强度与原始相比分别提高了 46.3%, 32.3%, 塑性基本维持不变。LSP 处理可使 LAM-TC4 钛合金获得更好的强度和塑性匹配。

关键词 激光增材制造; 激光冲击强化; 钛合金; 微观组织; 力学性能; 断口形貌

中图分类号 TN249 **文献标志码** A

1 引言

TC4 钛合金比强度高、耐热及耐蚀性好, 在航空领域被广泛应用^[1-2]。随着航空装备的不断发展, 对关键主承力结构, 提出了大型化、整体化、复杂化等迫切需求^[3], 传统锻造和铸造方法工艺复杂、成材率低、后续加工困难等难以满足此需求。近年来, AM 技术正成为工程、制造、材料、光学等学科的研究热点^[4]。LAM 具有数字、设计、制造一体化等优势, 能极大地提高原材料的利用率, 特别适合用于制造钛合金结构件, 已成为提升高性能复杂构件设计与制造能力的核心技术之一^[5-10]。

然而, 在钛合金 LAM 过程中, 熔池与基板之间会存在较大的温度梯度, 导致成形件综合力学性能不佳^[11-13]。为了改善 LAM 钛合金的综合力学性能, 研究者对 LAM 钛合金工艺参数、微观组织做了大量研究工作。齐振佳等^[14]研究发现硼元素对 LAM 钛合金的微观组织具有显著的细化效果。Zhan 等^[15]研究发现热处理可以有效的控制和消除 LAM 过程中较大

基金项目: 国家自然科学基金(U1804146, 52111530068)

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的温度梯度导致的残余应力。Ji 等^[16]研究表明可以通过优化工艺参数来消除增材制造产生的宏观缺陷。

LSP 是金属零件后处理的一种重要方法，利用高功率短脉冲激光作用于材料表面，使材料表面产生残余压应力和加工硬化层，具有可控性强、强化效果显著等突出优点^[17]。Martinez 等^[18]研究了 LSP 对激光熔覆 S275 和 316 钢的残余应力缓解效果，他们发现 LSP 处理可以引入显著的压应力。Sun 等^[19]将 LSP 技术应用于 2319 铝合金增材制造，他们发现 LSP 可以细化表面组织，提高显微硬度和拉伸性能。Shiva 等^[20]比较了激光退火和 LSP 对增材制造的 Ni-Ti 记忆合金的影响，结果表明，LSP 可以诱导残余压应力，防止表面裂纹的形成。Guo 等^[21]研究了 LSP 对等轴组织增材制造 Ti6Al4V 钛合金的影响，结果表明，LSP 使表层组织得到细化，延伸率有所提高，但屈服强度和极限抗拉强度变化不大。说明，LSP 技术对于 LAM 材料在关键应用中的应用是非常有益的^[22]。目前，关于 LSP 对 LAM 钛合金方面的研究还较少，因此，LSP 对 LAM-TC4 钛合金组件微观结构和性能的影响值得深入研究。

本文在 LAM-TC4 钛合金上，采取 LSP 表面处理，对 LSP 前、后的材料进行实验，系统地研究了 LSP 对 LAM-TC4 组织和性能的影响机制，期望通过改善 LAM 钛合金表层的力学性能进而改善其综合力学性能，为优化 LAM 钛合金组织及力学性能提供试验依据。

2 实验步骤

2.1 实验材料与方法

实验所选用的材料是 LAM-TC4 钛合金，化学成分如表 1 所示。

表 1 LAM-TC4 钛合金的化学成分（%，质量分数）

Table 1 Chemical composition of LAM-TC4 alloy（%，mass fraction）

Element	Al	V	Fe	C	Ti
Mass fraction	6.25	4.12	0.05	0.01	Bal.

在 LAM-TC4 钛合金试样上，用 DK7732 型线切割机先切出尺寸为 40mm×12mm×1.8 mm 的板状样，其几何尺寸如图 1 所示。对板状样依次用 400#-2000# 的砂纸进行手工打磨，把精磨后的板状样进行超声波清洗，保证待加工表面的清洁。采用 YS80-R200B 型激光冲击强化设备，对板状样进行单面 LSP 处理，如图 1 所示白块区为 LSP 区域（25mm×6 mm），斜线阴影区域为未 LSP 区，光斑直径 3 mm，激光能量 7 J，能量密度 4.95 GW/cm²，波长 1064 nm，脉宽 20 ns，搭接率 50%，约束层为去离子水，吸收保护层为黑胶带，冲击次数 1 次。在 LSP

处理后的板状样中心区域部分切出符合拉伸标准的板状试样，板状拉伸试样的尺寸如图 2 所示，拉伸试样原始标距为 6 mm，平行长度为 8 mm。在 LSP 处理后的板状样的 LSP 区域与未 LSP 区域切出几何尺寸为 6mm×6mm×1.8 mm 的块状试样。

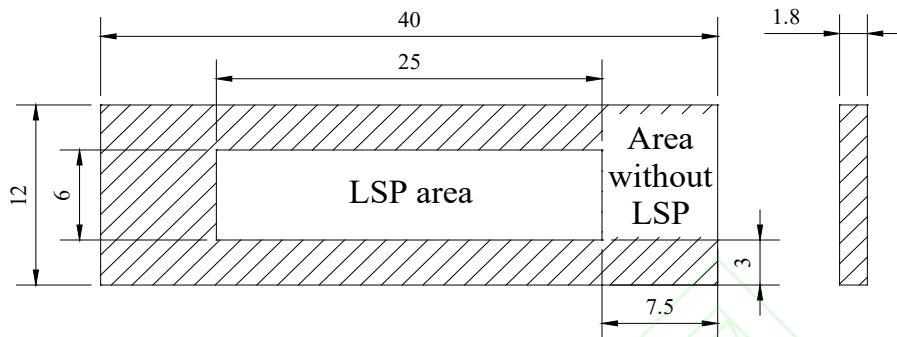


图 1 板状样尺寸(mm)

Fig. 1 Size of plate sample (mm)

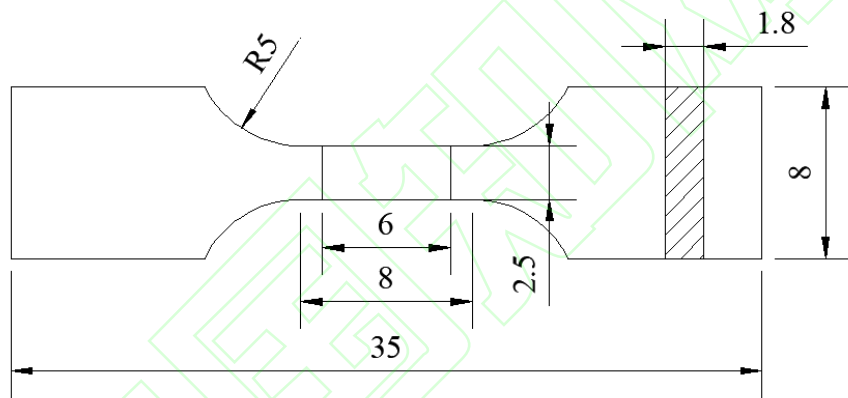


图 2 拉伸试样尺寸 (mm)

Fig. 2 Dimension of tensile sample (mm)

2.2 显微结构表征与性能测试

制备 LSP 与未 LSP 的块状试样的金相，经过粗抛、细抛后成镜面，之后再用清水冲洗干净，用由氢氟酸、硝酸、蒸馏水按体积比为 2: 5: 43 配制而成的腐蚀剂进行腐蚀处理，最后用 OLYMPUS PMG3 型光学显微镜 (OM) 进行微观组织观察。

将 LSP 与未 LSP 的块状试样用不同型号的砂纸手工打磨至 50 μm ，之后用 Gatan 691 离子减薄仪减薄，冲裁得到尺寸为 $\phi 3\text{ mm}$ 的透射样，采用 JEM-2010 型高分辨透射电镜 (TEM) 进行组织结构分析，加速电压为 200 kV。

分别对 LSP 与未 LSP 的块状试样使用 D8 ADVANCE 型 X 射线衍射 (XRD) 分析仪进行测试，采用 Cu-K α 射线，加速电压 40 kV，采用步进扫描模式，扫描速度为 2°/min，步长 0.02°，扫描角度为 30°~90°。

用 X-350A 型 X 射线应力分析仪测量 LSP 前、后 LAM-TC4 钛合金试样距表层不同深

度处的残余应力, 应力衍射仪参数选择: Cu K α 射线, X 射线管电压为 27 kV, 电流为 7 mA, 衍射晶面 $\alpha(213)$ 。用 MH-3 型显微硬度计对 LSP 与未 LSP 的块状试样表面和深度方向上进行硬度测试, 为了减少测量误差, 每个深度方向测量 5 个点的显微硬度, 计算出平均值作为显微硬度值。硬度测试点轨迹如图 3 所示, 施加载荷为 0.1 kgf, 加载时间 10 s。

采用 Instron 5587 型拉伸试验机测试拉伸性能, 拉伸速度为 0.5 mm/min, 并借助 JSM-IT200 型扫描电子显微镜对拉伸试验后的 LSP 前、后 LAM-TC4 钛合金拉伸样的断口形貌进行分析, 加速电压为 20 kV。

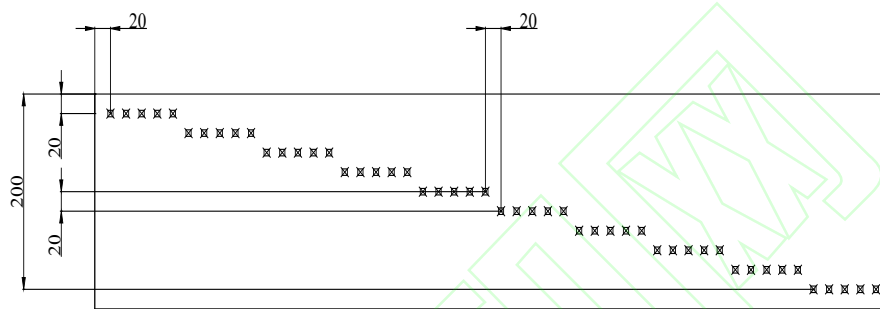


图 3 硬度轨迹图 (μm)

Fig. 3 Hardness trajectory diagram (μm)

3 实验结果与分析

3.1 LAM-TC4 钛合金 LSP 前、后微观组织形貌 (OM)

LAM 合金的组织特征与激光束作用下金属材料的热过程密切相关^[23]。LAM 过程中存在多层多道熔覆沉积热效应, 会对已沉积层产生往复加热/冷却热影响, 直接决定了 LAM 合金晶粒形貌和微观组织与传统锻件明显不同^[3]。图 4 (a) 给出了 LAM-TC4 钛合金的金相组织形貌, 可见其宏观组织由呈外延生长的粗大 β 柱状胞晶构成, 晶内微观组织是由大量的 α 板条及一定体积分数的板条间 β 相组成, 箭头所指为粗大的 β 柱状晶晶界^[23]。形成这种组织特征的原因是, 高温凝固时, 外延初生 β 柱状枝晶生长, 在往复的加热/冷却过程中快速地进行 β 相到 α 相的固态相变, 同时大量的初生 α 相, 继续生长成细长的板条状^[24-26]。图 4 (b) 为 LSP 后的金相组织形貌, 与 LSP 前相比, 原始粗大的 β 柱状晶晶界变的不明显, α 板条更细小。

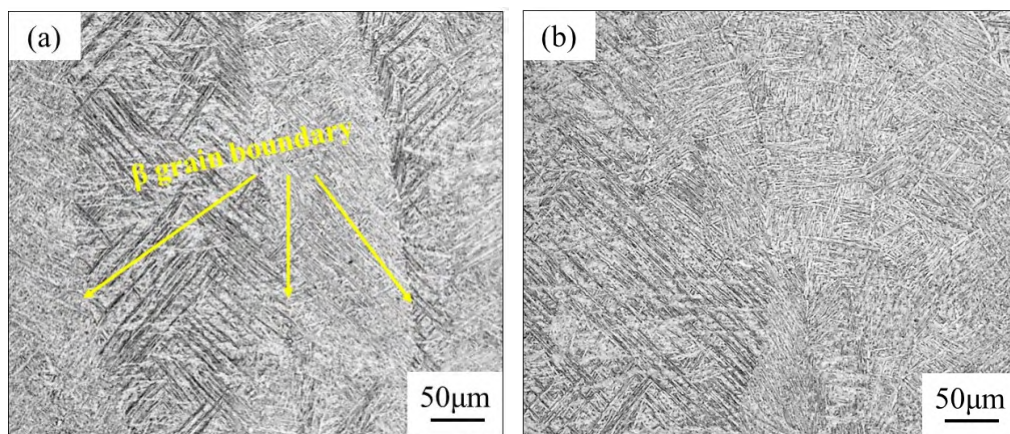


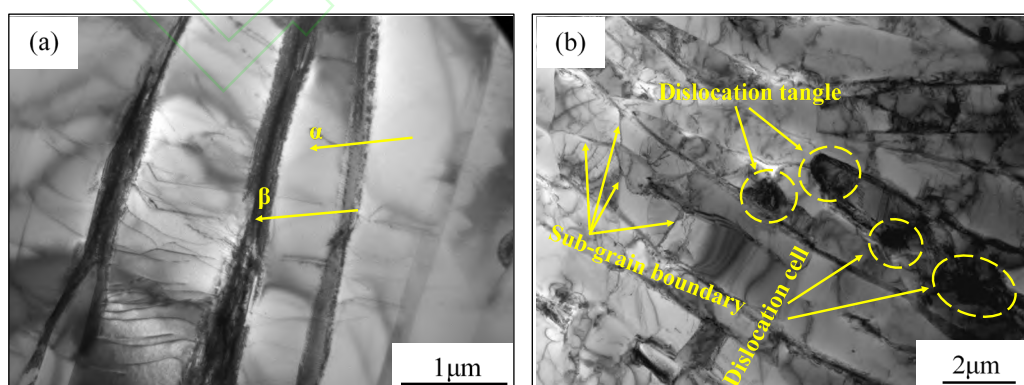
图 4 LAM-TC4 钛合金 LSP 前后微观组织形貌 (OM)。(a) LSP 前; (b) LSP 后

Fig. 4 OM microstructure morphology of LAM-TC4 titanium alloy before and after LSP.

(a) Before LSP; (b) After LSP

3.2 LAM-TC4 钛合金 LSP 前、后微观组织形貌 (TEM)

通过透射电镜观察,可以更好地了解 LSP 前、后 LAM-TC4 的微观组织演变。图 5 为 LSP 前、后 LAM-TC4 钛合金的 TEM 图像及选区电子衍射花样。其中图 5 (a) 所示为试样未 LSP 时的 TEM 照片,可以看出片状 α 相和残余 β 相构成的基体中几乎没有位错。图 5 (b-f) 所示为试样经过 LSP 后的 TEM 照片,从图 5 (b) 中可以看出粗大 α 板条被破碎细化,这归因于 LSP 诱导各种位错结构的发展形成 (亚) 晶粒^[27]。从图 5 (c-d) 中可以看出 α 板条中产生了形变孪晶,位错密度大幅增加,形成了位错网、位错缠结,相界处产生胞状位错结构^[28]。图 5 (e-f) 为形变孪晶的明场像与暗场像,可以看出, α 板条上存在大量位错和与形变孪晶交互作用产生的高密度位错缠结。通过对图 5 (f) 中的区域进行选区电子衍射斑点标定[图 5 (f) 右上角],可确定其为形变孪晶,孪晶面为 $\{0002\}$ 。



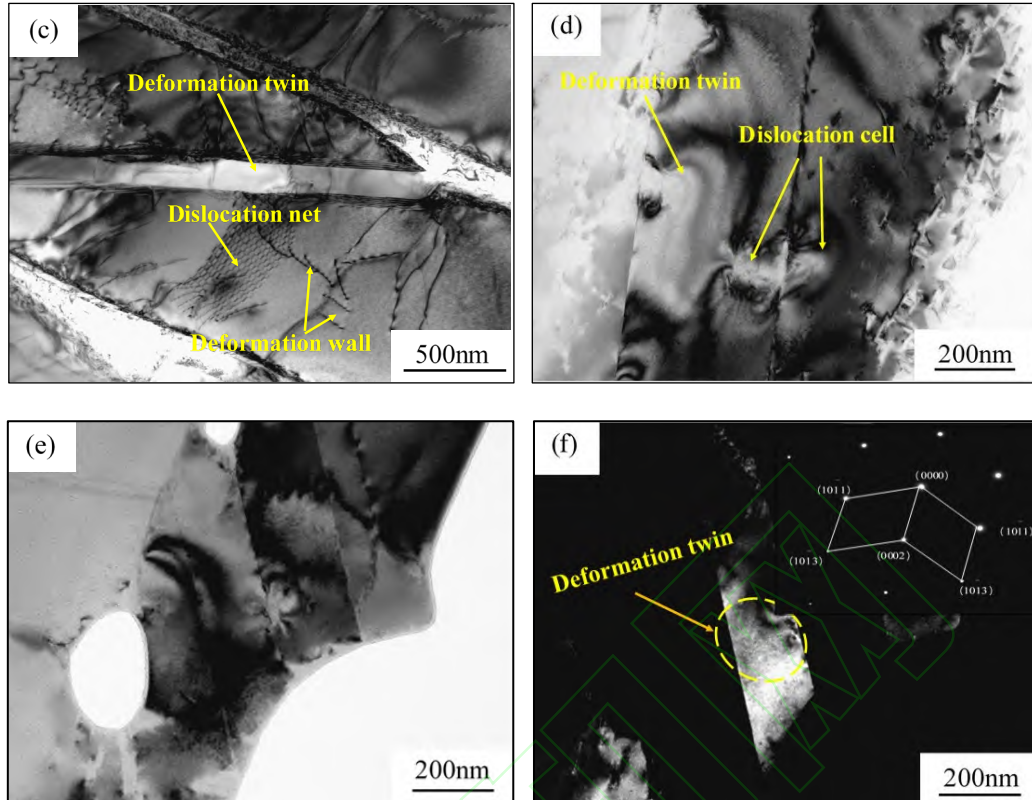


图 5 LAM-TC4 钛合金 LSP 前后微观组织形貌（TEM）。（a）LSP 前；（b-f）LSP 后

Fig. 5 TEM microstructure morphology of LAM-TC4 titanium alloy before and after LSP.

(a) Before LSP; (b-f) After LSP

3.3 LSP 前、后 LAM-TC4 钛合金的 XRD 图谱

图 6 为 LSP 前、后 LAM-TC4 钛合金表面的 XRD 衍射图。从图 6（a）可看出，其主要物相为 $\text{hcp}\alpha\text{-Ti}$ ，经 LSP 处理后的 XRD 图谱中没有出现新的衍射峰，即无相变。从衍射峰局部 $34^\circ\sim 42^\circ$ 放大图[图 6（b）]可以看出 $\alpha(100)$ 及 $\alpha(002)/\beta(110)$ 衍射峰强度增大，说明经 LSP 处理后这些晶面上出现了变形织构^[29]，LSP 处理后半峰全宽明显宽化，由 Jade 估算出 LSP 处理后试样晶粒细化至 39.9 nm。衍射峰宽化是晶粒细化和微观应变共同作用的结果^[30]。此外还可以看到 LSP 后衍射峰的位置略向右偏移，这是由于在 LSP 过程中，LAM-TC4 钛合金表面在高能冲击波作用下产生了较大的残余压应力，引起晶格畸变和晶面收缩^[31-32]。这也与 Guo 等^[21]的研究结果相吻合。

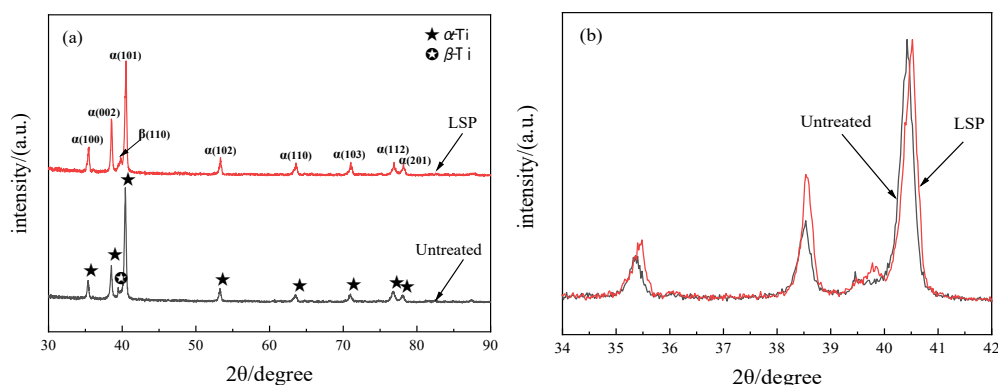


图 6 LSP 前后 LAM-TC4 钛合金的 XRD 图谱。(a) 30°~90°; (b) 34°~42°

Fig. 6 XRD patterns of LAM-TC4 titanium alloy before and after LSP. (a)30°~90°;(b)34°~42°

3.4 LSP 对 LAM-TC4 钛合金性能的影响

图 7 为 LSP 前、后 LAM-TC4 钛合金不同深度的残余应力分布图。可看到，在 LSP 之前，LAM-TC4 钛合金表层存在残余拉应力。残余拉应力是热应力和相变应力竞争的结果，其中前者有利于压应力的形成，后者有利于拉应力的形成^[33]。由此可以推断，LAM 过程中， β 相到 α 相的固态相变产生的拉应力大于高温梯度产生的压应力，导致原始样中存在残余拉应力，在 140 μm 深度处产生的最大拉应力为 210 MPa。经 LSP 处理后，在 LAM-TC4 钛合金表层形成了残余压应力，最大值为 -190 MPa，残余压应力影响深度为 180 μm 。残余压应力的形成是塑性变形和体积限制的组合作用^[34]。LSP 高能冲击波使合金表层发生严重的塑性变形，诱导表层形成残余应力场^[35-36]。距离表面越远，残余压应力值越小，这与激光冲击波能量随着深度的增加逐渐衰减有关，因此残余压应力的分布呈现深度梯度变化特征。这在 Yan 等^[37]的研究中有相同的发现。

图 8 为 LSP 前、后 LAM-TC4 钛合金表面和深度方向上显微硬度分布。结果表明，LSP 提高了 LAM-TC4 钛合金试样表面的显微硬度。其中未 LSP LAM-TC4 钛合金表面和深度方向的显微硬度值大致相同，为 326.7HV_{0.1}。经过 LSP 后，试样表面显微硬度值显著提高，达到最大值 380.7 HV_{0.1}，提升幅度为 16.5%。显微硬度的提高是位错、形变孪晶、细晶强化等共同作用的结果^[38]。经过 LSP 处理，LAM-TC4 钛合金表面发生塑性变形，产生大量的位错缠结，阻碍位错的运动，导致表面加工硬化^[14]。同时，在高能冲击作用下， α 相结构被破坏，得到细化，有效提升了表面硬度。

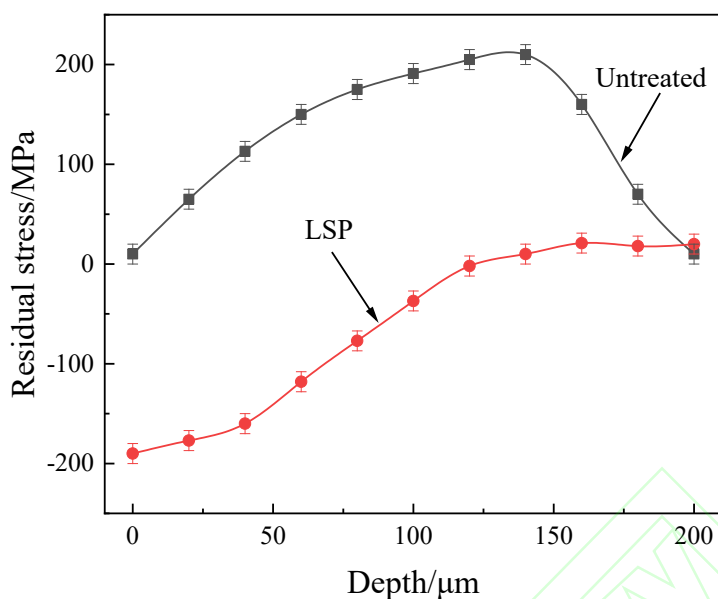


图 7 LSP 前后 LAM-TC4 钛合金不同深度的残余应力

Fig. 7 Residual stress in LAM-TC4 titanium alloy with different layer depths before and after LSP

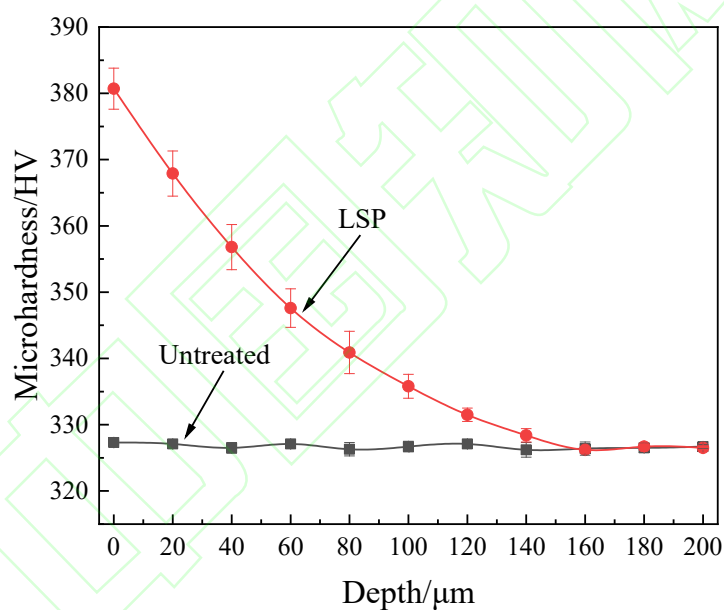


图 8 LSP 前后 LAM-TC4 钛合金深度方向上显微硬度分布

Fig. 8 Microhardness distribution in the direction of layer depth of LAM-TC4 titanium alloy before and after LSP

与锻造材料相比，增材制造 TC4 的平均屈服强度和极限抗拉强度略低，塑性相似^[39]。

LSP 前、后 LAM-TC4 钛合金的拉伸性能如图 9 所示。从图中可以看出，未 LSP 处理的 LAM-TC4 钛合金的屈服强度为 720 MPa，抗拉强度为 940 MPa，伸长率为 15.5%。经 LSP 处理一次后，LAM-TC4 钛合金的屈服强度为 1053 MPa，抗拉强度为 1244 MPa，断后伸长率为 15.0%。屈服强度和抗拉强度均有显著提升，断后伸长率基本维持不变。可见，LSP 处理可以消除增材制造引起的负面影响，并提高样品的拉伸性能。经 LSP 处理后，LAM-TC4 钛合金表面发生严重塑性变形，形成高幅值残余压应力，可以抑制裂纹的产生与扩展，使强

度提高^[40]。LSP 处理使 LAM-TC4 钛合金表层产生细晶强化效果，而内部粗晶组织仍具有很高的拉伸应变和加工硬化能力，可以有效抑制表层结构可能产生的应变集中和早期颈缩^[41]。晶粒细化提高材料的强度是基于晶界的影响，晶界会阻碍位错运动，增加位错穿过晶界和从一个晶粒移动到另一个晶粒的难度^[21]。前人的研究表明，细晶强化到纳米尺度后，经典位错活动不再存在，纳米晶粒尺寸小，没有位错滑移的空间^[42]，塑性变形的调节被晶粒旋转和晶界滑动所取代^[43-45]。LSP 处理使 LAM-TC4 钛合金表层组织产生大量形变孪晶，有研究发现，孪晶界可以提供位错成核位置，作为位错发射源，提供更多的可移动位错，使晶体取向有利于晶粒间的协调变形改变，从而有效地增韧材料，提高材料的塑性^[46-47]。在这些因素的综合作用下，LAM-TC4 钛合金经 LSP 处理后获得了更佳的力量和塑性匹配。

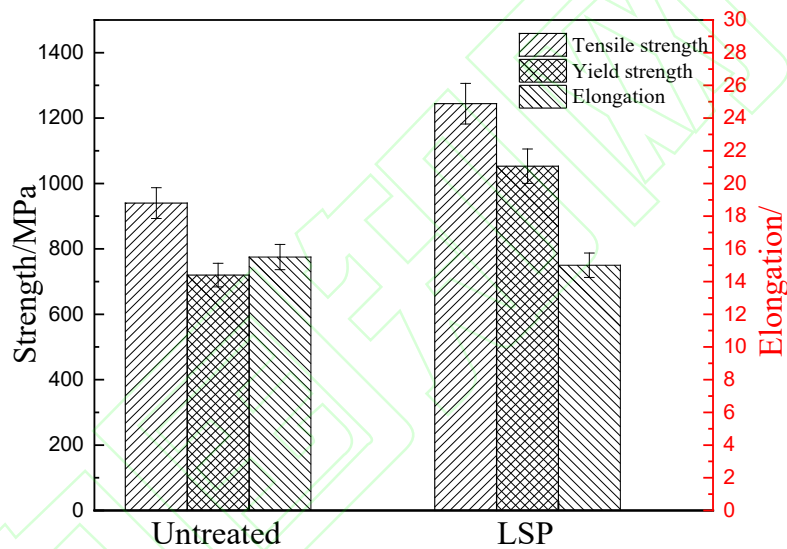


图 9 LSP 前后 LAM-TC4 钛合金的拉伸性能

Fig. 9 Tensile properties of LAM-TC4 titanium alloy before and after LSP

3.5 LSP 前、后 LAM-TC4 钛合金的拉伸断口形貌

拉伸试验后 LSP 前、后 LAM-TC4 钛合金的断口形貌如图 10 所示。断口形貌以深等轴韧窝为主，断裂机制均为典型的韧性断裂。图 10（a）和（b）为未经 LSP 处理的 LAM-TC4 钛合金表层和心部的拉伸断口形貌，可以看出，表层拉伸断口包含大量大小不一的韧窝和少量孔洞，与心部断口形貌无明显区别。图 10（c）和（d）为经 LSP 处理的 LAM-TC4 钛合金表层和心部的拉伸断口形貌，与图 10（a）和（b）中的韧窝相比，可以看出，经 LSP 处理后，表层强化区域的韧窝小且浅，韧窝分布均匀，这是由于表层晶粒在**高能冲击波**的作用下充分破碎，晶粒细化；经 LSP 处理的钛合金心部断口韧窝与未经 LSP 处理的钛合金心部断口韧窝相比也较细且浅，但其变化幅度低于表层，是由于心部所选区域也受到一定冲击波

影响所致。

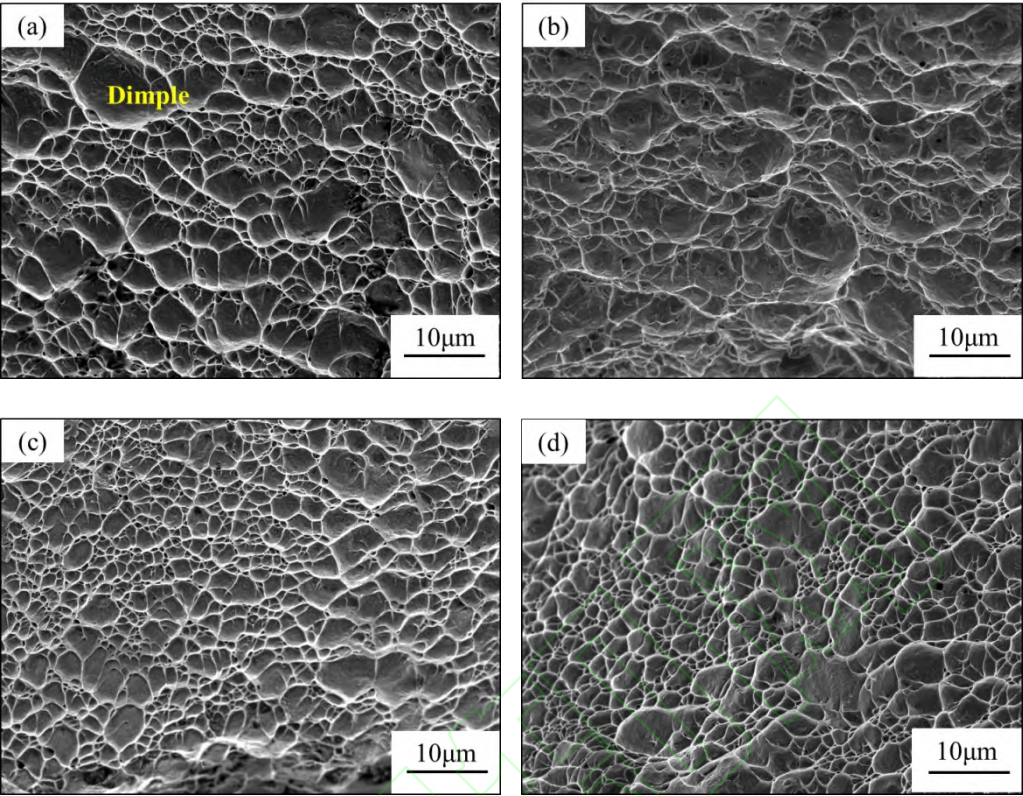


图 10 LSP 前后 LAM-TC4 钛合金的拉伸断口形貌。(a) 未 LSP 试样的表层断口形貌；(b) 未 LSP 试样的心部断口形貌；(c) LSP 试样的表层断口形貌；(d) LSP 试样的心部断口形貌

Fig. 10 Tensile fracture morphology of LAM-TC4 titanium alloy before and after LSP. (a) Surface fracture morphology of the sample before LSP; (b) Heart fracture morphology of the sample before LSP; (c) Surface fracture morphology of the sample after LSP; (d) Heart fracture morphology of the sample after LSP

4 结 论

本文通过采用 LSP 对 LAM-TC4 钛合金进行表面处理，系统地研究了 LSP 对 LAM-TC4 钛合金组织演变和性能的影响，得到以下结论：

LSP 处理使 LAM-TC4 钛合金表层发生严重的塑性变形，表层基体中产生大量形变孪晶，位错密度大幅增加，各种位错结构的交互作用发展形成（亚）晶粒，从而使晶粒细化。

LSP 处理后 LAM-TC4 钛合金表面存在最大残余压应力（-190 MPa），试样表面显微硬度值显著提高，达到最大值 380.7 HV_{0.1}。残余应力和显微硬度值均呈梯度变化趋势，随着距表层距离的增大，相应的数值减小。

LSP 处理后 LAM-TC4 钛合金的屈服强度和抗拉强度与原始相比分别提高了 46.3%，32.3%，塑性基本维持不变，断口形貌仍以深等轴韧窝为主，合金获得了更好的强度和塑性

匹配。

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Effect of Laser Shock Peening on the Microstructure and Properties of Laser Additive Manufacturing TC4 Titanium Alloy

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Abstract

Objective TC4 titanium alloy is widely used in the aerospace industry due to its high specific strength, good heat and corrosion resistance. Laser additive manufacturing has the advantages of digital, design and manufacturing integration, which can greatly improve the utilization rate of raw materials and is particularly suitable for manufacturing titanium alloy structural parts, it has become one of the core technologies to enhance the design and manufacturing capability of high-performance complex components. However, during laser additive manufacturing of titanium alloys, there can be large temperature gradients between the melt pool and the substrate, resulting in poor comprehensive mechanical properties of the formed parts. Therefore, it is very important to find a suitable method to improve its comprehensive mechanical properties to extend its service life. Laser shock peening (LSP) is an important method for post-treatment of metal parts, using a high-power short-pulse laser on the surface of the material to produce compressive residual stresses and work hardening layers on the surface of the material, with the outstanding advantages of controllability and significant strengthening effect. In this paper, the effect mechanism of LSP on the microstructure and properties of laser additive manufacturing (LAM) TC4 titanium alloy is systematically investigated by adopting LSP surface treatment, expecting to improve the mechanical properties of the surface layer of LAM-TC4 titanium alloy and then improve its comprehensive mechanical properties, and provide an experimental basis for optimizing the microstructure and mechanical properties of LAM titanium alloy.

Methods In this work, LSP was first applied to the LAM-TC4 titanium alloy surface. Then, the physical phase of LSP and non-LSP block samples was analyzed by X-ray diffractometer (XRD), and the microstructure of LSP and non-LSP block samples were observed by optical microscope

(OM); the LSP and non-LSP block samples were manually ground to 50 μm with different types of sandpaper, punched into discs of size $\phi 3\text{ mm}$, and then thinned by Gatan 691 ion thinning instrument, and the microstructure was further investigated using a JEM-2010 transmission electron microscope (TEM). Finally, the mechanical properties and fracture morphology of the samples before and after LSP were characterised by X-ray stress analyser, microhardness tester, tensile testing machine and Scanning electron microscope (SEM).

Results and Discussion The original microstructure of LAM-TC4 titanium alloy consists of a large number of thick α laths and a certain volume fraction of inter-lath β phases [Fig.4(a)]. After LSP treatment, the surface layer microstructure was broken and refined by the action of high-energy shock waves [Fig.5(b)], and a large number of dislocation [Fig.5(b-d)] and deformation twins [Fig.5(d-f)] were formed. The LSP treatment made the residual stress of the LAM-TC4 titanium alloy the tensile residual stress formed by the LAM into the compressive residual stress (Fig. 7). After LSP treatment, the surface of LAM-TC4 titanium alloy had the maximum compressive residual stress (-190 MPa) (Fig. 7), the microhardness had increased by 16.5% (Fig. 8), and it shows the characteristics of changing along the depth gradient. In addition, after LSP treatment, the yield strength and tensile strength of LAM-TC4 titanium alloy increased by 46.3% and 32.3% respectively compared with the original, and the plasticity remained basically unchanged (Fig.9). The fracture morphology of LAM-TC4 titanium alloy before and after LSP is mainly composed of deep equiaxed dimples, and the fracture mechanism is typical of ductile fracture (Fig.10).

Conclusion In present study, the effect of LSP on the microstructure evolution and properties of LAM-TC4 titanium alloy was systematically investigated by surface treatment of LAM-TC4 titanium alloy using LSP, and the following conclusions were obtained: Our study shows that the LSP treatment causes severe plastic deformation in the surface layer of LAM-TC4 titanium alloy, generates a large number of deformation twins in the surface matrix, dislocation density increases significantly, and the interaction of various dislocation structures develops to form (sub)grains, resulting in grain refinement. The maximum compressive residual stress (-190 MPa) exists on the surface of LAM-TC4 titanium alloy after LSP treatment, and the surface microhardness value of the specimen increases significantly, reaching a maximum value of 380.7 $\text{HV}_{0.1}$. Both residual stress and microhardness values show a gradient trend, and the corresponding values decrease

with increasing distance from the surface layer. The yield strength and tensile strength of LAM-TC4 titanium alloy after LSP treatment increased by 46.3% and 32.3%, respectively, compared with the original specimens, while the plasticity remained basically unchanged, and the fracture morphology was still dominated by deep equiaxial dimples, and the alloy obtained a better match between strength and plasticity.

Key words laser additive manufacturing; laser shock peening; titanium alloy; microstructure; mechanical properties; fracture morphology

