

• 高熵合金的非平衡凝固制备:工艺、组织与性能

特邀专栏

**Preparation of High-entropy Alloys via Non-equilibrium Solidification:
Processes, Microstructures and Properties •**

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激光粉末床熔融胞状组织对中/高熵合金 变形行为的影响

汤伟哲, 卢健麟, 王松宇, 何 峰

(西北工业大学 凝固技术全国重点实验室, 陕西 西安 710072)

摘 要: 激光粉末床熔融技术为中/高熵合金的设计和制备提供了新的工艺途径, 激光粉末床熔融特有的快速凝固和反复热循环特性赋予了金属材料兼具元素偏析和高密度位错的胞状组织。胞状组织影响金属材料变形行为, 并最终决定材料力学性能, 是增材制造金属材料性能调控的关键。激光粉末床熔融胞状组织影响位错行为并可触发额外变形机制, 与传统制备工艺中/高熵合金相比, 展现出高强度塑性协同。因此, 系统综述激光粉末床熔融胞状组织对变形行为的影响机理, 对完善增材制造金属材料的性能调控理论及推动其工程应用具有重要意义。

关键词: 激光粉末床熔融; 中熵合金; 高熵合金; 胞状组织; 变形行为; 力学性能

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Effect of Cellular Structure on the Deformation Behavior of Medium/high-entropy Alloys Fabricated by Laser Powder Bed Fusion

TANG Weizhe, LU Jianlin, WANG Songyu, HE Feng

(State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, China)

Abstract: Laser powder bed fusion (LPBF) technology provides a novel processing route for the design and preparation of medium/high-entropy alloys (M/HEAs). The unique rapid solidification and repeated thermal cycling characteristics of LPBF endow the metallic material with a cellular structure featuring both elemental segregation and high-density dislocations. This cellular structure influences the deformation behavior of the metallic material and ultimately affects its mechanical properties, making tailoring the performance of additively manufactured materials key. Compared with conventionally processed M/HEAs, the cellular structure of LPBF affects dislocation behavior and can trigger additional deformation mechanisms, resulting in high strength-ductility synergy. Therefore, systematically reviewing the influence mechanism of the LPBF cellular structure on deformation behavior is highly important for improving the performance control theories of additively manufactured metallic materials and promoting their engineering applications.

Key words: laser powder bed fusion; medium-entropy alloy; high-entropy alloy; cellular structure; deformation behavior; mechanical property

中/高熵合金 (medium/high-entropy alloys, M/HEAs) 多主元化的成分设计理念为开发新型高性能合金开辟了新的道路, 具有广阔的工程应用前景^[1]。

其宽广的成分空间结合热机械处理, 赋予材料微观组织高度的可调性, 使位错滑移、变形孪生、应变诱发相变等多种机制得以协同或竞争激活^[2-4], 为调控

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作者简介: 汤伟哲, 2001 年生, 博士生. 研究方向为中/高熵合金变形机理. Email: tangweizhe@mail.nwpu.edu.cn

通信作者: 何 峰, 1991 年生, 博士, 教授. 研究方向为高熵合金设计及其非平衡凝固制备. Email: fenghe1991@nwpu.edu.cn

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并实现优异机械性能提供关键基础。目前,金属增材制造(additive manufacturing, AM)技术已成为现代航空航天、国防等具体工程应用的核心制造技术,占据极为重要的战略发展地位^[5-9]。其中,激光粉末床熔融(laser powder bed fusion, LPBF)是金属材料增材制造领域的代表性方法,其快速凝固的成形特点引入了独特的非平衡微观组织^[10]。这一特性为中/高熵合金的微观组织设计和性能提升提供了具有颠覆性潜力的新技术平台。

激光粉末床熔融过程的快速凝固和多次往复热循环工况形成了兼具元素偏析和高密度位错的胞状组织,是激光粉末床熔融成形合金的共性组织特征,对材料的力学性能具有决定性作用^[11-13]。已有研究指出,胞状组织使增材制造合金能够突破强度-塑性倒置关系^[14-15],然而,其内在机理仍有待揭示。现有研究关注了胞状组织对位错滑移/孪生/相变行为的影响^[14, 16-17],但研究对象涉及多种合金体系,微观组织和变形机制各有不同。胞状组织对变形行为的影响规律与机理尚存争议,当前仍缺乏共性结论,导致增材制造金属材料性能调控受到严重制约。

1 中/高熵合金的变形行为和力学性能

中/高熵合金是一类新型多组元合金,具有广阔的成分设计空间和可观的性能潜能^[18-19]。与传统以单一组元为溶质,并添加少量合金元素作为溶剂的设计策略不同,中/高熵合金将合金成分选择扩展至相图中心,形成无序单相固溶体。本文以面心立方(FCC)中/高熵合金为主要的讨论对象,是一类以Cr、Mn、Fe、Co、Ni等3d过渡族元素为主构成的固溶体合金,典型体系包括CoCrFeMnNi^[20-21],四元CoCrFeNi^[22],以及三元的CoCrNi^[23]、NiCoV^[24]等。

如图1a所示,广泛的成分空间赋予了中/高熵

合金丰富多样的性能组合,其范围几乎涵盖现有报道的各类铝合金、镁合金、钛合金及先进高强钢。多主元体系带来了复杂的合金化效应和灵活的热机械处理组合,从而具备了优异的综合力学性能^[25]。一般而言,FCC中/高熵合金通常表现为低强度和高塑性的特点。以CoCrNi中熵合金为例,其在室温和低温环境下表现出优异的力学性能^[26-27]。Gludovatz等^[26]发现,等原子比CoCrNi合金室温屈服强度为437 MPa,抗拉强度为877 MPa,塑性为70%,且断裂韧性超过200 MPa·m^{1/2};低温下屈服强度达655 MPa,抗拉强度突破1300 MPa,塑性提升至90%且断裂韧性高达275 MPa·m^{1/2}。如图1b所示,等原子比CoCrNi中熵合金强度及韧性组合超越了许多中/高熵合金及传统合金^[1]。Laplanche等^[28]研究指出,CoCrNi中熵合金优异的拉伸性能主要归因于其较低的层错能(stack-fault energy, SFE),能够在较宽的应变范围内激活孪生诱发塑性(twinning induced plasticity, TWIP)效应,从而获得持续增强的加工硬化能力。此外,通过调整原子比例改变CoCrNi体系的SFE,可将变形机制扩展至位错主导或相变诱发塑性(transformation induced plasticity, TRIP)^[29]。变形机制的灵活可调性为中/高熵合金性能优化打开了更为广阔的研究窗口。

1.1 中/高熵合金变形行为

变形机制是调控中/高熵合金力学性能的核心。如图2所示,中/高熵合金表现为与传统金属与合金相似的位错结构。以CoCrFeMnNi合金为例,位错在77~873 K的宽温域范围内表现在{111}滑移面上发生1/2<110>平面滑移的特征^[30]。同样在(CoCrNi)₉₃Al₇Ti₂Nb合金中也观察到来自双{111}滑移面上的1/2<110>位错发生平面滑移。在低层错能体系中,可观察到位错扩展。例如,1/2<110>位错扩展形成两个1/6<112>不全位错及层错^[31]。Xu等^[32]在变形的

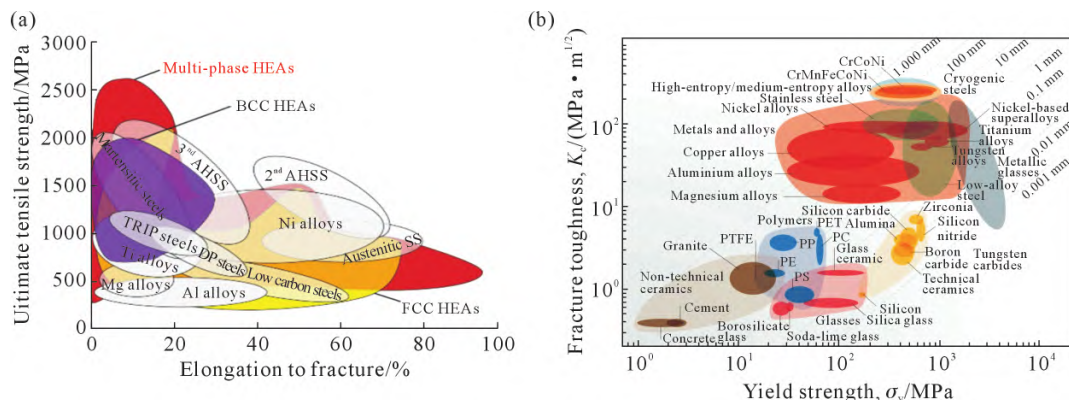


图1 各材料机械性能 Ashby 图:(a) 强度和塑性比较;(b) 强度和韧性比较^[1, 25]

Fig.1 Ashby plot comparing the mechanical properties of various materials: (a) strength versus ductility; (b) strength versus fracture toughness^[1, 25]

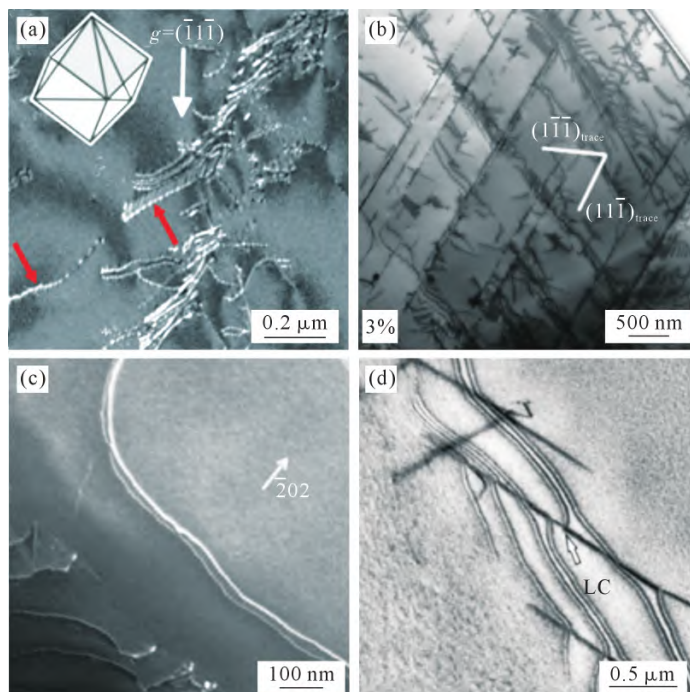


图2 各类 FCC 中/高熵合金位错结构:(a) CoCrFeMnNi 合金在小变形量下为 $\{111\}$ 滑移面上 $1/2\langle 110 \rangle$ 位错的平面滑移;(b) $(\text{CoCrNi})_{93}\text{Al}_4\text{Ti}_2\text{Nb}$ 双 $\{111\}$ 滑移面上交截的 $1/2\langle 110 \rangle$ 位错;(c) CoCrNi 合金中位错扩展;(d) 扩展位错交截形成 Lomer-Cottrell 锁^[25,31-32]

Fig.2 Dislocation structures in various FCC medium/high-entropy alloys: (a) planar slip of $1/2\langle 110 \rangle$ dislocations on $\{111\}$ slip planes under small strain in CoCrFeMnNi alloy; (b) intersecting $1/2\langle 110 \rangle$ dislocations on double $\{111\}$ slip planes in $(\text{CoCrNi})_{93}\text{Al}_4\text{Ti}_2\text{Nb}$ alloy; (c) observed dislocation extension in CoCrNi alloy; (d) formation of a Lomer-Cottrell lock by extended dislocations^[25,31-32]

$\text{Al}_{0.1}\text{CoCrFeNi}$ 合金观察到交截的扩展位错形成 Lomer-Cottrell (L-C)位错锁。即来自不同滑移面的扩展位错相互交截形成不可动的压杆位错,成为后续滑移位错的阻塞。

由于 FCC 中/高熵合金特有的浓度波动,其位错行为具有区别于传统稀溶质合金的特征。Smith 等^[33]统计中/高熵合金中层错的扩展宽度,表现出高度离散性。位错倾向于避开高层错能区域而在低层错能区域发生扩展,从而展现出成分依赖的层错宽度离散性。中/高熵合金独特的化学起伏还控制了位错类型和变形机制^[34]。举例而言,密度泛函理论(density functional theory, DFT) 计算结果得出 CoCrNi、CoCrFeMnNi 和 NiCoV 合金 SFE 均为负值,分别为 -30、-23 和 -11 mJ/m^2 ^[35]。然而,三者的变形机制却不尽相同。CoCrNi 合金表现为位错+孪晶,仅在低至 20 K 下观察到 TRIP 效应^[27];CoCrFeMnNi 合金同样为位错+孪晶的变形机制^[36];而 NiCoV 合金则完全为位错主导的变形,未观察到孪晶或新相生成^[37]。这一特性亦将影响增材制造中/高熵合金位错的滑移行为及变形机制,下文进行具体讨论。

另一方面,如表 1 所示,以 Cr、Mn、Fe、Co、Ni 等为主元成分的 FCC 中/高熵合金因普遍具有较低的层错能^[38-41]。因此,研究者借鉴了传统奥氏体钢激活

TWIP 效应和 TRIP 效应的设计思路,开发机械亚稳型中/高熵合金^[42-44]。例如, Laplanche 等^[28]对 CoCrFeMnNi 合金的研究表明,在 77 K 低温下, TWIP 效应被激活(图 3),为变形提供额外的加工硬化能力。Li 等^[42]通过调整 $\text{Fe}_{80-x}\text{Mn}_x\text{Co}_{10}\text{Cr}_{10}$ 体系的 Fe/Mn 原子比例,降低 FCC 相稳定性,持续的诱发了 FCC 向 HCP 马氏体相变(图 4)。由不全位错滑动形成的层错作为变形孪晶和 HCP 马氏体相形成的核胚,是机械亚稳效应的基本结构起源^[45]。研究表明,层错自身作为典型的面缺陷,也能有效的阻碍位错滑移^[46]。Tong 等^[47]发现,在 $\text{FeCoCrNiTi}_{0.2}$ 合金中变形层错阻碍了位错的平面滑移,大量的层错交织形成网状层错束,进一步限制位错的运动(图 5)。

1.2 中/高熵合金力学性能

前文指出中/高熵合金广阔的成分空间赋予了其变形机制灵活的可调性,能够覆盖包涵位错、层错、孪晶、应变诱发马氏体相等多种变形机制。这一特性激发了学者对性能调控的广泛探索,并获得了一系列重要的进展,分类总结如下。

(1)基于位错的性能调控策略 研究者致力于通过成分和微观组织设计,以期调控位错行为,提升合金综合力学性能。通过引入化学短程有序^[48]和异质晶粒尺寸^[49]等组织调控策略,可促进塑性变形过

表1 不同方法获得FCC中/高熵合金层错能
Tab.1 Stacking fault energies of FCC medium-/high-entropy alloys captured by different methods

Composition	T/K	SFE/(mJ·m ⁻²)	Technique to capture SFE
CoCrNi ^[38]	0	-62	DFT calculation
CoCrNi ^[38]	293	18±4	TEM
CoCrFeNi ^[39]	0	31.6±0.9	DFT calculation
CoCrFeNi ^[40]	298	27±4	TEM
CoCrFeMnNi ^[39]	0	29.7±1.2	DFT calculation
CoCrFeMnNi ^[40]	298	26.5±4.5	TEM
CoCrFeNiCu _{0.5} ^[39]	0	29.0±1.9	DFT calculation
CoCrFeNiCu ^[39]	0	27.5±1.3	DFT calculation
CoCrFeNiCuAl _{0.3} ^[39]	0	33.8±2.2	DFT calculation
CoCrFeNiCuAl _{0.5} ^[39]	0	32.0±2.1	DFT calculation
CoCrFeNiCuTi _{0.5} ^[39]	0	37.4±2.4	DFT calculation
Ni ₂₆ Fe ₂₀ Cr ₁₄ Co ₂₀ Mn ₂₀ ^[41]	0	57.7	EMTO+CPA+VASP
Ni ₂₃ Fe ₂₀ Cr ₁₇ Co ₂₀ Mn ₂₀ ^[41]	0	19.7	EMTO+CPA+VASP
Ni ₁₄ Fe ₂₀ Cr ₂₆ Co ₂₀ Mn ₂₀ ^[41]	0	3.5	EMTO+CPA+VASP
CoCrFeNiAl _{0.25} ^[39]	0	38.7±2.6	DFT calculation
CoCrFeNiAl _{0.3} ^[39]	0	35.2±2.3	DFT calculation
CoCrFeNiAl _{0.375} ^[39]	0	33.7±2.3	DFT calculation
CoCrFeNiAl _{0.5} Ti _{0.1} ^[39]	0	42.4±3.0	DFT calculation
CoCrFeNiAl _{0.3} Mo _{0.1} ^[39]	0	37.2±2.6	DFT calculation
CoCrFeNiMnCu ^[39]	0	27.0±1.2	DFT calculation

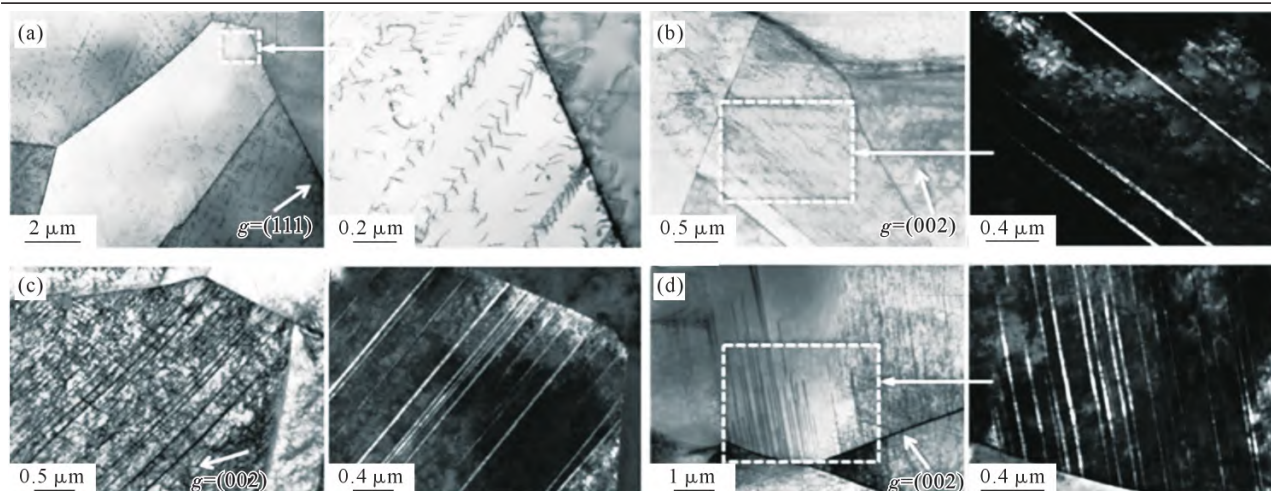


图3 低温变形下应变诱发孪生 CoCrFeMnNi 合金组织演化规律: (a) $\varepsilon=2.1\%$; (b) $\varepsilon=6.0\%$; (c) $\varepsilon=8.8\%$; (d) $\varepsilon=12.1\%$ ^[28]
Fig.3 Microstructural evolution of the TWIP effect in CoCrFeMnNi alloys under cryogenic deformation: (a) $\varepsilon=2.1\%$; (b) $\varepsilon=6.0\%$; (c) $\varepsilon=8.8\%$; (d) $\varepsilon=12.1\%$ ^[28]

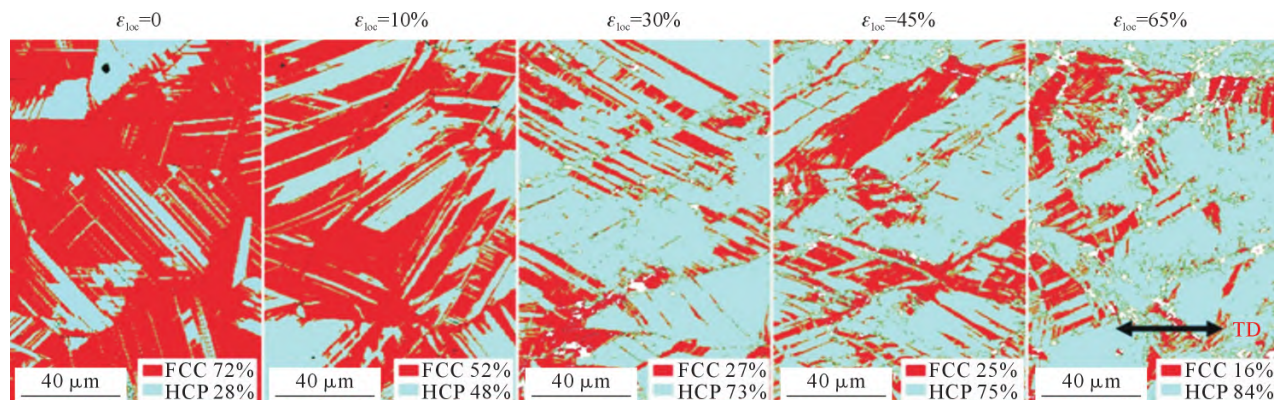


图4 应变诱发相变 Fe_{80-x}Mn_xCo₁₀Cr₁₀ 合金组织演化规律^[42]
Fig.4 Microstructural evolution of the TRIP effect in the Fe_{80-x}Mn_xCo₁₀Cr₁₀ alloy^[42]

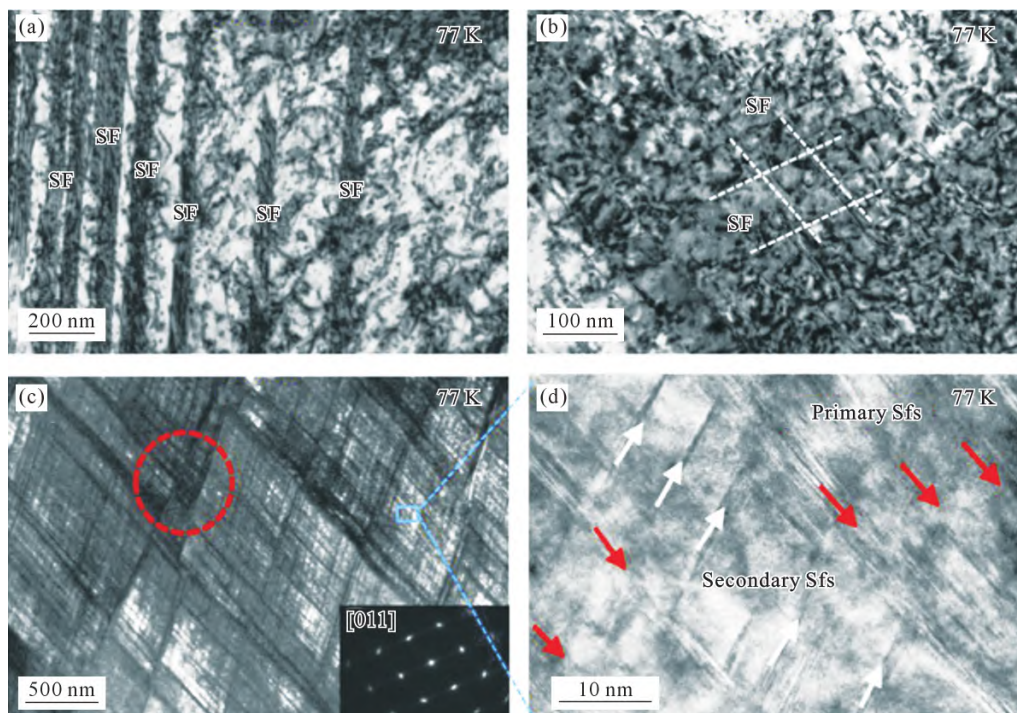


图 5 FeCoCrNiTi_{0.2} 合金在不同变形量下微观组织中中层错演化规律: (a) $\varepsilon=2.5\%$; (b) $\varepsilon=10\%$; (c) $\varepsilon=36\%$; (d) (c)局部放大图^[47]
Fig.5 Deformation stacking faults and the formation of intersecting stacking fault bundles in FeCoCrNiTi_{0.2} alloy: (a) $\varepsilon=2.5\%$; (b) $\varepsilon=10\%$; (c) $\varepsilon=36\%$; (d) locally enlarged view of (c)^[47]

程位错的交叉滑移。交叉滑移能够缓解位错塞积引发的应力集中,从而推迟塑性失稳的发生,提升合金的塑性。然而,交滑移缓解应力集中的同时,不可避免的促进位错湮灭,降低了位错存储,将在一定程度的削弱合金的应变硬化。相反的,He 等^[50]通过 Ti、Al 微合金化在 Ni₂CoCrFe 合金引入短程有序结构,有效调控了位错滑移模式,提出反常的固溶强韧化机制。位错平面滑移形成密集的滑移带,滑移带间距的持续细化促进了晶内位错的均匀分布。合金在提升强度(屈服强度由 177 MPa 提升至 252 MPa,抗拉强度由 531 MPa 提升至 682 MPa)的同时保持高加工硬化能力,获得良好的塑性(总伸长率由 59%提升至 69%)。

(2)基于扩展位错的性能调控策略 机械亚稳型中/高熵合金借鉴了来自奥氏体钢的成功经验。基于大量实验实践总结获得出普适性规律,SFE 可用作机械亚稳机制的判定指标:当 $SFE>35 \text{ mJ/m}^2$,倾向于位错主导变形^[51];当 $SFE=18\sim35 \text{ mJ/m}^2$,倾向发生 TWIP 效应;当 $SFE<18 \text{ mJ/m}^2$,合金倾向于发生 TRIP 效应。本质上,TWIP 和 TRIP 效应是通过产生额外的界面阻碍位错滑移,从而促进位错存储^[30]。因此,降低 SFE 成为实现合金强韧化的一种直接手段。在 FeMnNiCoCr 体系中调整 Fe/Mn 含量,激活了 TRIP 效应,获得约 350 MPa 屈服强度,850 MPa 抗拉强度和 75%总伸长率的优异综合性能^[42]。Laplanche 等^[23]研究认为,由于 CoCrNi 中熵合金的层错能低于

FeMnCoCrNi 高熵合金($\text{CoCrNi}:(22\pm4) \text{ mJ/m}^2$, $\text{FeMnCoCrNi}:(30\pm5) \text{ mJ/m}^2$)而激活了广泛的变形孪晶,从而获得更为优异的综合力学性能(CoCrNi 中熵合金屈服强度为 250 MPa,抗拉强度为 870 MPa,总伸长率为 38%; FeMnCoCrNi 高熵合金屈服强度为 360 MPa,抗拉强度为 760 MPa,总伸长率为 30%)。此外,Li 等^[52]在 $\text{Fe}_{50}\text{Mn}_{30}\text{Co}_{10}\text{Cr}_{10}$ 合金中添加 C 间隙原子实现增加体系 SFE,从而提升 FCC 相的稳定性。尽管合金变形的 TRIP 效应受抑制,但额外的 TWIP 效应和 TRIP 效应协同作用带来更优异的应变硬化能力,实现了合金的综合力学性能提升(屈服强度由约 350 MPa 提升至 520 MPa,抗拉强度由 850 MPa 提升至 960 MPa,总伸长率由 75%降低至 60%)。由此可见,合金力学性能的提升并非与层错能的降低呈简单的线性关系。以 TRIP 效应合金为例,形成的 HCP 相由于有限的滑移系和显著的各向异性,削弱了合金进一步塑性变形的调节能力^[53-54]。同时,FCC 和 HCP 相边界的应变不相容容易促进微裂纹的萌生,从而导致快速的失效^[55]。

Wei 等^[3]在 CoCrNiW 合金中另辟蹊径的提出层错主导变形的亚稳强韧化策略,获得约 500 MPa 的屈服强度,1 100 MPa 的抗拉强度和 61%的总伸长率。广泛的层错增殖及其交互作用提供了有效的应变硬化,同时避免了因 HCP 相形成而牺牲的应变协调能力,为机械亚稳合金的强韧化提供一种新思路。这更新了传统的认知,层错不仅仅作为相变和孪晶

的核胚,其本身亦可作为单一的晶体塑性载体。总结层错引发加工硬化的微观机制具体包括:①非平行层错相交形成不动位错;②平行层错间的弹性互斥^[3]。具体而言,当来自相交滑移面上的层错相互交截形成压杆位错,构成 L-C 位错锁(图 6)。另一方面,当层错在已有层错之间形核和扩展时,将对两侧的 Shockley 不全位错产生 Peach-Koehler 力^[56]。原则上,变形层错诱发应变硬化机制普遍适用于任何金属材料。然而,在机械亚稳型合金的强韧化机理研究中,层错的作用长期被理解为变形孪晶或应变诱发 HCP 相的核胚,其自身作为独立的晶体塑性载体尚未引起足够的重视。针对层错的强韧化策略仍有待探索与开发。

2 激光粉末床熔融中/高熵合金胞状组织及其变形机理

激光粉末床熔融技术以高能激光束作为输入源,依据三维模型切片规划的扫描路径,对粉末床铺展的金属粉末原料进行逐点逐层的快速熔化和凝固,进而实现目标形状的构建。与传统铸造、锻造、机械加工结合等制造工艺技术相比,具有高柔性、响应迅速和周期短的特点,在成形复杂结构、零件精度、表面质量等方面具有优势^[10]。

研究表明,激光粉末床熔融过程极高的温度梯度、极快的凝固速度和循环受热的成形过程,促使材料形成独特的具有微观偏析、高密度位错的微米/亚微米级胞状组织^[58]。以 Kumar 等^[15]报导的沉积态 CoCrNi 合金为例,其强度较传统制备合金显著提升。如图 7 所示,激光粉末床熔融技术形成具有高密度位错的胞状组织,使沉积态 CoCrNi 合金屈服强度

达到 691 MPa、抗拉强度达到 926 MPa,同时保持 38%塑性。其屈服强度相较于传统制备工艺合金提升 57%,并拥有 $184 \text{ MPa} \cdot \text{m}^{1/2}$ 的断裂韧性。Sun 等^[57]的研究表明,激光粉末床熔融 CoCrFeMnNi 合金在具有 550 MPa 室温屈服强度的同时仍保持 18% 的塑性。同时,得益于胞状组织高热稳定性,实现在 77 K 至 873 K 宽温域保持优异的力学性能。上述研究结果一致表明,激光粉末床熔融成形合金具有优异的综合力学性,胞状组织形成机理与强韧化机制息息相关,将在后文进行系统论述。

2.1 胞状组织特征

胞状组织是激光粉末床熔融成形合金的共性组织特征,普遍存在于 Ni 基、Co 基、Fe 基以及中/高熵合金中^[13-14,59]。针对胞状组织的研究主要集中于构型尺寸、元素偏析、位错组态及密度等特征要素。

(1)胞状组织构型尺寸 激光粉末床熔融胞状组织构型尺寸为凝固胞状晶的一次胞晶间距,大小取决于合金的凝固条件^[60]。胞状组织普遍呈现沿晶向的择优取向生长^[58]。图 8a 展示了胞状组织的三维结构形状,呈现平行轴向为柱状结构和垂直轴向为蜂窝状结构的特征。调控工艺参数可有效改变胞状组织尺寸。Liu 等^[61]通过改变扫描速度,成功实现了胞状组织尺寸在 300~1 200 nm 的有效调控。

(2)胞状组织元素偏析 胞状组织的元素偏析源于快速凝固过程的溶质再分配。如图 8b 所示,胞状组织胞壁处的偏析元素种类根据合金体系而异。举例而言,激光粉末床熔融 316L 不锈钢胞壁偏析元素为 Cr 和 Mo^[12,62];CoCrNi 合金偏析元素为 Cr^[63]; (CoCrNi)₉₄Ti₃Al₃ 合金偏析元素为 Ti^[64]。Zhang 等^[65]结合多尺度模拟和实验验证发现,偏析随增材制造

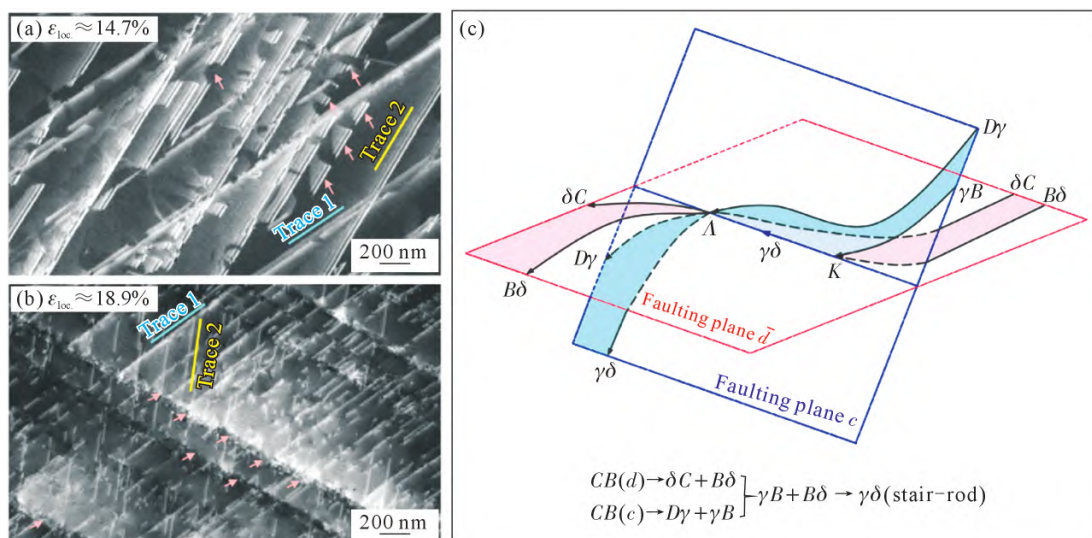


图 6 非平行层错交互作用机理:(a, b) 不同变形量下非平行层错交截形成 Lomer-Cottrell 锁;(c) 不同滑移面层错反应示意图^[3]
Fig.6 Mechanism of non-parallel stacking fault interaction: (a, b) formation of Lomer-Cottrell locks through the intersection of non-parallel stacking faults under different deformation levels; (c) schematic of stacking fault reactions on different slip planes^[3]

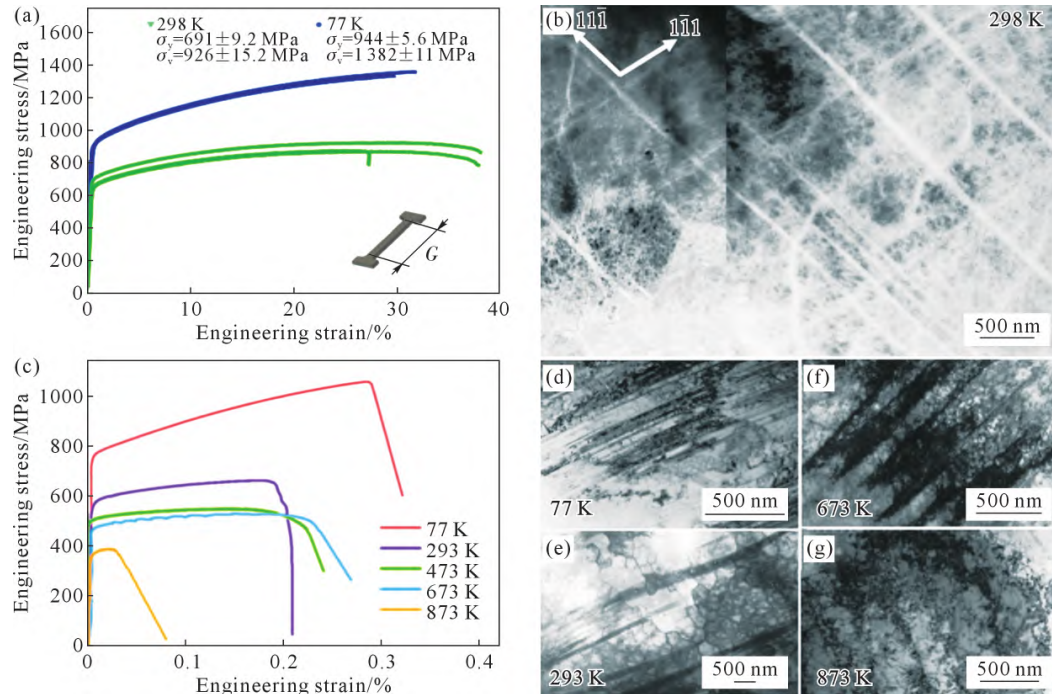


图7 激光粉末床熔融中/高熵合金力学性能:(a) 沉积态 CoCrNi 合金室温(298 K)和低温(77 K)的力学性能;(b) 对应室温变形微观组织;(c) 沉积态 CoCrFeMnNi 合金宽温域力学性能;(d~g) 对应不同温度的变形微观组织^[15,57]

Fig.7 Mechanical properties of medium/high-entropy alloys fabricated via LPBF: (a) mechanical properties of the as-built CoCrNi alloy at room temperature (298 K) and cryogenic temperature (77 K); (b) corresponding room-temperature deformation microstructures; (c) mechanical properties of the as-built CoCrFeMnNi alloy over a wide temperature range; (d~g) deformation microstructures at corresponding temperatures^[15,57]

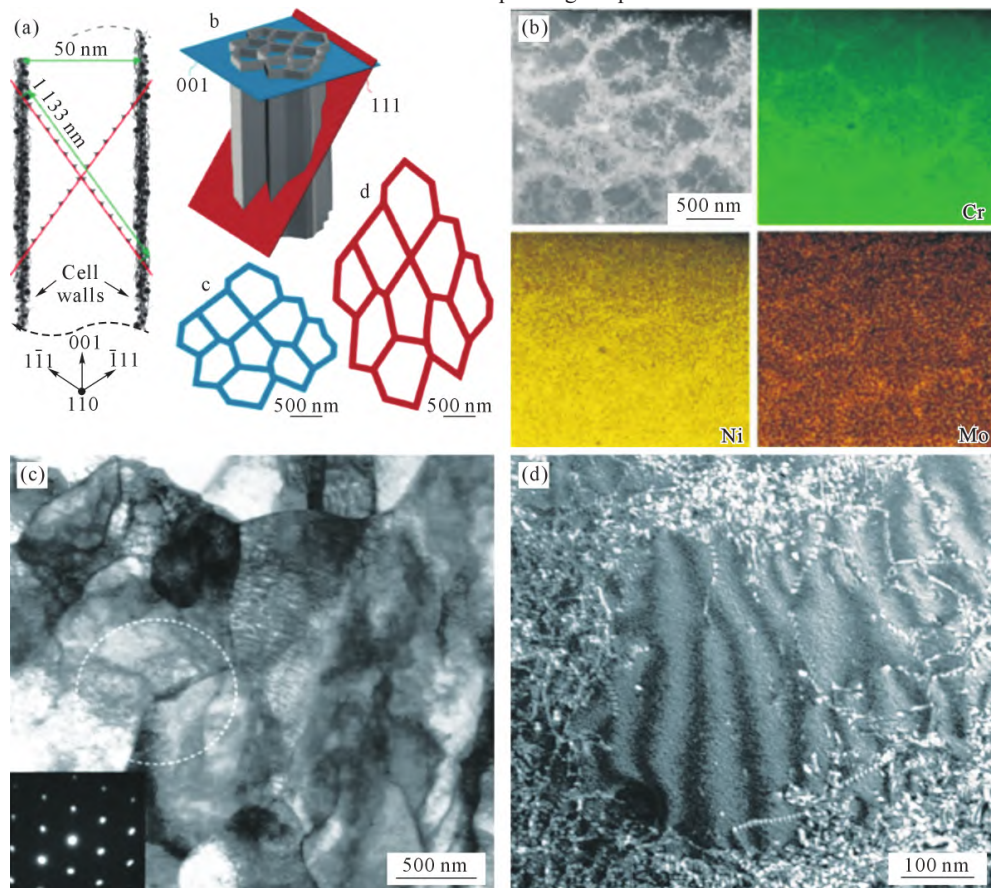


图8 激光粉末床熔融胞状组织特征:(a) 胞状组织示意图;(b) 胞壁元素偏析特征;(c) 纯铜合金胞状组织位错结构;(d) 有偏析-位错胞状组织^[12,17,58]

Fig.8 Cellular structure characteristics of LPBF-fabricated alloys: (a) schematic of the cellular structure; (b) elemental segregation characteristics at cell walls; (c) dislocation structures within the cellular structure of pure copper alloy; (d) cellular structure with segregation-dislocation^[12,17,58]

成形参数改变的幅度极小,这是因为凝固分配系数只有在凝固速度变化极大的时候才受到显著影响。

(3)胞状组织位错组态和位错密度 胞状组织的位错组态(位错类型和空间分布)表现为胞壁高密度位错和胞内低密度位错的非均匀分布特征。高密度位错分布通常与凝固偏析胞重叠。除完全位错外,在激光粉末床熔融 316L 不锈钢和中/高熵合金胞状组织中还额外发现了层错^[11-12]。推测层错的形成与胞壁处高密度位错、残余应力和局域化学成分起伏相关^[58]。在无溶质偏析的激光粉末床熔融纯铜^[17]和纯铁^[66]金属中同样观察到高密度位错的胞状组织存在。值得注意的是,由于缺乏与溶质偏析的相互作用,激光粉末床熔融纯金属的胞状组织在形态上与塑性变形形成位错胞更为相近,呈现等轴状而非在合金中普遍观察到的蜂窝状结构。Fan 等^[67]观察到沿沉积方向不同深度的胞状组织具有不同的位错组态。如图 9 所示,沿沉积方向观察,上表面为未发展完全的胞状组织,位错分布松散随机;而随沉积深度增加(经历热循环次数增多),逐渐形成边界清晰的胞状组织。

综合现有实验表征和模拟研究结果可知,胞状组织的形成归结为一个多阶段、多因素耦合的物理

过程:激光粉末床熔融快速凝固阶段的溶质发生再分配形成凝固胞晶;同时,熔池约束和枝晶偏转等因素诱发了位错的形成。热循环过程影响位错的增殖、湮灭和重排,最终形成溶质元素富集和高密度位错共存的胞状组织。现有结论表明了胞状组织的位错组态与位错密度对激光粉末床熔融工艺参数和合金成分的敏感性与可调性,这为激光粉末床熔融成形合金的微观组织设计与性能调控提供了基础条件。

2.2 胞状组织对合金变形的影响

胞状组织的高密度位错和微观偏析是影响材料变形行为及力学响应的关键要素。具体而言,胞状组织中的高密度位错壁可作为位错移动的障碍,缩短了位错滑移平均自由程,产生“类 Hall-Petch 效应”的强化作用^[15]。而 Liu 等^[61]通过 TEM 下的原位观察发现,在应变增加到一定程度后,塞积于胞壁处的位错将穿过胞壁继续滑移。进一步的,Shi 等^[68]通过中子衍射轮廓分析,定量评估了胞状组织的高密度位错满足泰勒模型的位错强化,这一观点逐步被学界普遍接受^[11,69]。

已有研究探讨了胞状组织高密度位错的强化机理。然而,胞状组织如何影响塑性的具体规律与机理尚缺乏系统性的结论。胞状组织对位错滑移/孪生/相变

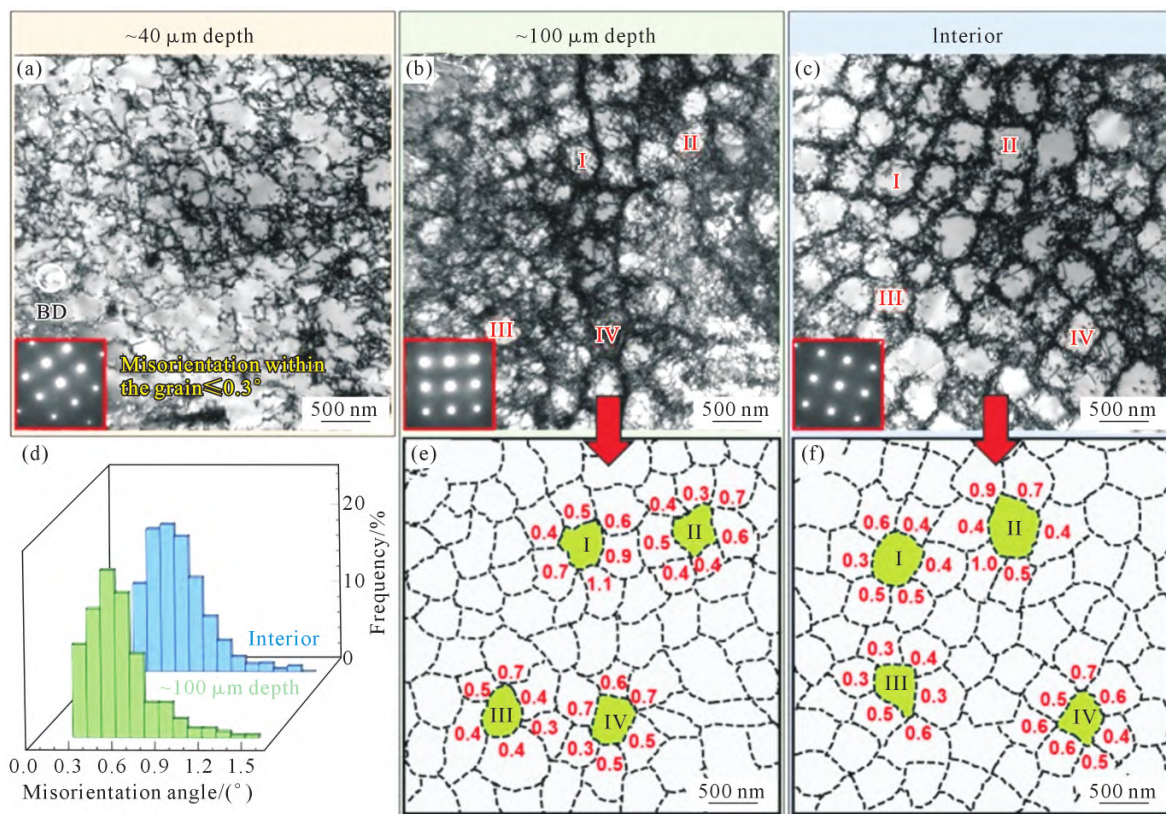


图9 激光粉末床熔融成形 316L 不锈钢组织特征:(a~c)沿沉积方向不同沉积深度胞状组织特征;(d)距沉积表面约 100 μm 及芯部组织的取向差分布;(e, f)距沉积表面约 100 μm 及芯部邻近胞状组织取向差^[67]

Fig.9 Cellular structure characteristics of laser powder bed fusion-fabricated 316L stainless steel at different layer heights: (a~c) cellular structure characteristics at different deposition depths along the building direction; (d) misorientation distribution at ~100 μm depth and interior; (e, f) misorientation of adjacent cellular structures at ~100 μm depth and interior^[67]

行为的影响规律不同时,其位错组态亦呈现出明显区别。依据胞状组织的位错组态,可将其分类讨论。

(1)胞状组织中位错对变形影响 Li等^[17]关于激光粉末床熔融纯铜金属的研究指出,热循环过程完全位错的相互作用在胞壁处形成大量 Lomer 锁,有效阻碍塑性变形过程中的位错滑移,从而提升了材料的加工硬化能力(图 10a 和 b)。然而,这难以完全代表具有凝固偏析特征的胞状组织的变形行为。除对位错的阻碍作用外,胞状组织还将对位错滑移模式造成影响。Cui等^[70]报道了胞状组织促进位错由波状滑移转变为平面滑移,从而提升了合金的疲劳性能。Guo等^[11]针对胞状组织的研究揭示,胞状组织的高密度位错可作为位错源,同时胞壁具有优异的位错存储能力,促进位错亚结构的持续细化。沉积态合金获得屈服强度为 655 MPa 的同时保持总伸长率为 35%。相较于铸态合金强度提升 1 倍(338 MPa),且塑性保持相同(图 10c 和 d)。

(2)胞状组织中扩展位错对变形影响 层错普遍存在于激光粉末床熔融中/高熵合金胞状组织中。预先存在于胞状组织中的层错,是 LPBF 非平衡过程的产物,在传统工艺制备的合金中通常难以形成。尽管其在胞状组织的数量密度远低于完全位错,却

能够显著提升力学性能(图 10e 和 f)^[11,13]。He 等^[12]报道了胞状组织中预先存在的层错引发额外的变形机制,层错在应力驱使下发生宽化并进一步形成“层错束”结构,提供潜在的加工硬化。除此之外,胞状组织形成的层错可促进 TWIP 和 TRIP 效应激活。Liu 等^[61]观察到纳米孪晶起源于胞壁, TWIP 效应的激活使沉积态合金获得 552 MPa 的屈服强度同时保持 83%总伸长率,均高于锻态下的 244 MPa 和 63%。Park 等^[71]在激光粉末床熔融 $\text{Fe}_{60}\text{Co}_{15}\text{Ni}_{15}\text{Cr}_{10}$ 合金中,发现胞状组织促使变形机制从位错滑移转向应变诱发马氏体相变,合金获得 490 MPa 屈服强度的同时拥有 70%的总伸长率。然而,上述针对胞状组织强韧化机制的研究大多停留在定性描述胞状组织促进亚稳效应的层面。相反的, Woo 等^[72]通过原位中子衍射定量揭示了增材制造与传统制备工艺 CoCrNi 合金的变形位错/孪生竞争机制,认为高位错密度胞壁将阻碍的孪晶的进一步生长。Freeman 等^[73]也指出胞状组织将抑制马氏体的生长。

综上所述,胞状组织在强韧化行为中扮演重要角色,胞状组织中完全位错能够作为位错滑移的“软屏障”,而层错能够促进变形孪生/相变。尽管研究者针对胞状组织高密度位错的强化机理已有相对清晰

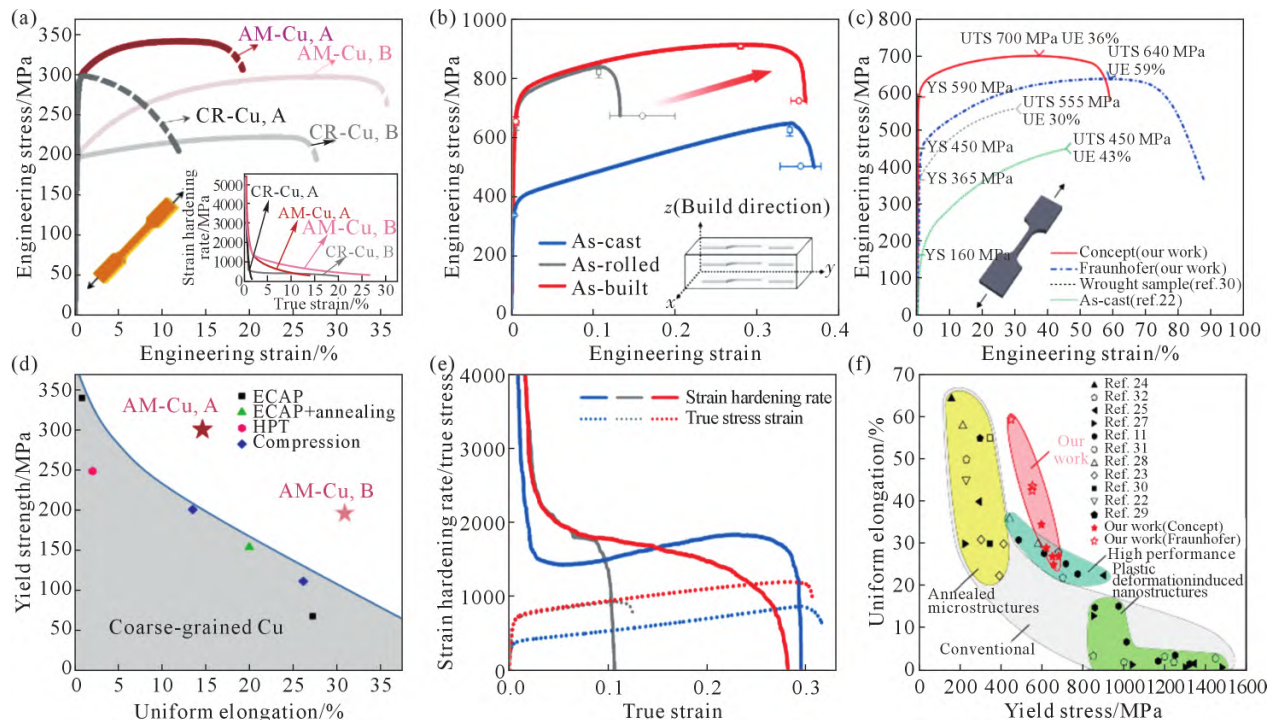


图 10 激光粉末床熔融与传统工艺制备合金力学性能:(a) 激光粉末床熔融和冷轧纯铜拉伸曲线对比;(b) 不同制备工艺纯铜强度和塑性对比;(c) 激光粉末床熔融和铸造及冷轧 $\text{Co}_{35}\text{Cr}_{25}\text{Ni}_{35}\text{Ti}_3\text{Al}_2$ 合金拉伸性能;(d) 不同制备工艺合金加工硬化能力对比;(e) 激光粉末床熔融和铸态及锻态 316L 不锈钢拉伸曲线;(f) 不同制备工艺 316L 不锈钢强度和塑性对比^[11,13,17]

Fig.10 Mechanical properties of alloys fabricated by LPBF and conventional processes: (a) comparison of tensile curves for LPBF-fabricated and cold-rolled pure copper; (b) comparison of strength and ductility of pure copper produced by different processes; (c) tensile performance of LPBF-fabricated, as-cast, and cold-rolled $\text{Co}_{35}\text{Cr}_{25}\text{Ni}_{35}\text{Ti}_3\text{Al}_2$ alloy; (d) comparison of strain hardening capability of the alloy produced by different processes; (e) comparison of tensile curves for LPBF-fabricated, as-cast, and forged 316L stainless steel; (f) comparison of strength and ductility of 316L stainless steel produced by different processes^[11,13,17]

的揭示,但对于胞状组织的增塑机制仍持有不同的观点。根源在于胞状组织不同位错组态显著影响材料的变形机制及力学响应,而相对应规律和机理未得到系统的总结和揭示。一方面,缺乏对于激光粉末床熔融胞状组织与传统变形位错胞的结构特征及变形行为差异的深入认知;另一方面,除高密度位错外,缺乏对于激光粉末床熔融组织中其它晶体缺陷,例如,层错/孪晶/相变与胞状组织的交互作用机制仍不明晰,缺乏对其变形微观机制的深入了解。

3 激光粉末床熔融中/高熵合金力学性能

综上所述,胞状组织是激光粉末床熔融中/高熵合金力学性能提升的关键。因此,如何通过调控胞状组织以实现性能优化,已成为当前的研究热点。现有的激光粉末床熔融合金的性能调控策略,主要围绕工艺参数优化、合金成分调整、掺杂纳米强化相等手段展开,旨在通过沉积态合金晶粒等轴化^[74]、抑制成形裂纹^[75]、获得弥散强化相^[76]等途径实现力学性能优化,尚缺少针对胞状组织的微观结构设计。下文将针对胞状组织的不同调控方法在提升材料性能方面的效果进行评述。

3.1 工艺参数调控合金力学性能

激光粉末床熔融工艺参数通过改变熔池尺寸和

形态,调控沉积态微观组织与冶金缺陷。Gao 等^[77]对比脉冲激光与连续激光成形两种工艺制备 CoCrNi 中熵合金,沉积态合金屈服强度从 692 MPa 提升至 808 MPa,总伸长率由 42.8%提升至 47.1%,这归因于脉冲激光成形改变了位错的分布。更多的研究集中在通过改变激光功率(P),扫描速率(v),道间距(h)和层厚(t),并总结定义了体能量密度($VED=P/vht$)。Wang 等^[78]通过调整 VED 从 69 J/mm³至 139 J/mm³,使得激光粉末床熔融 (CoCrNi)₉₅Mo₅ 合金伸长率从 28%提升至 36%,而强度未发生明显改变,这归因于成形过程冶金缺陷的减少。Li 等^[79]采用机器学习方法确认激光粉末床熔融 CoCrNi 中熵合金高致密度(>0.99)的工艺窗口,实现屈服强度、抗拉强度与伸长率在 580~620 MPa,845~880 MPa,35~53%的调控。然而,这被归因于成形过程冶金缺陷的优化。上述研究表明,尽管工艺参数在一定程度上影响力学性能,但总体而言优化窗口较窄,难以实现突破性提升。

3.2 合金成分调控合金力学性能

依据 3.1 节分析的胞状组织形成机理,胞状组织的位错组态将受到层错能依赖的位错行为影响。Ahn 等^[80]在 CoCrNi 中熵合金中添加 C 元素可提高体系层错能,从而抑制胞状组织中位错的扩展,如图 11a 和 b 所示。合金屈服强度达到 815 MPa,抗拉强度达到 1047 MPa,总伸长率为 19.4%。相反的,如图

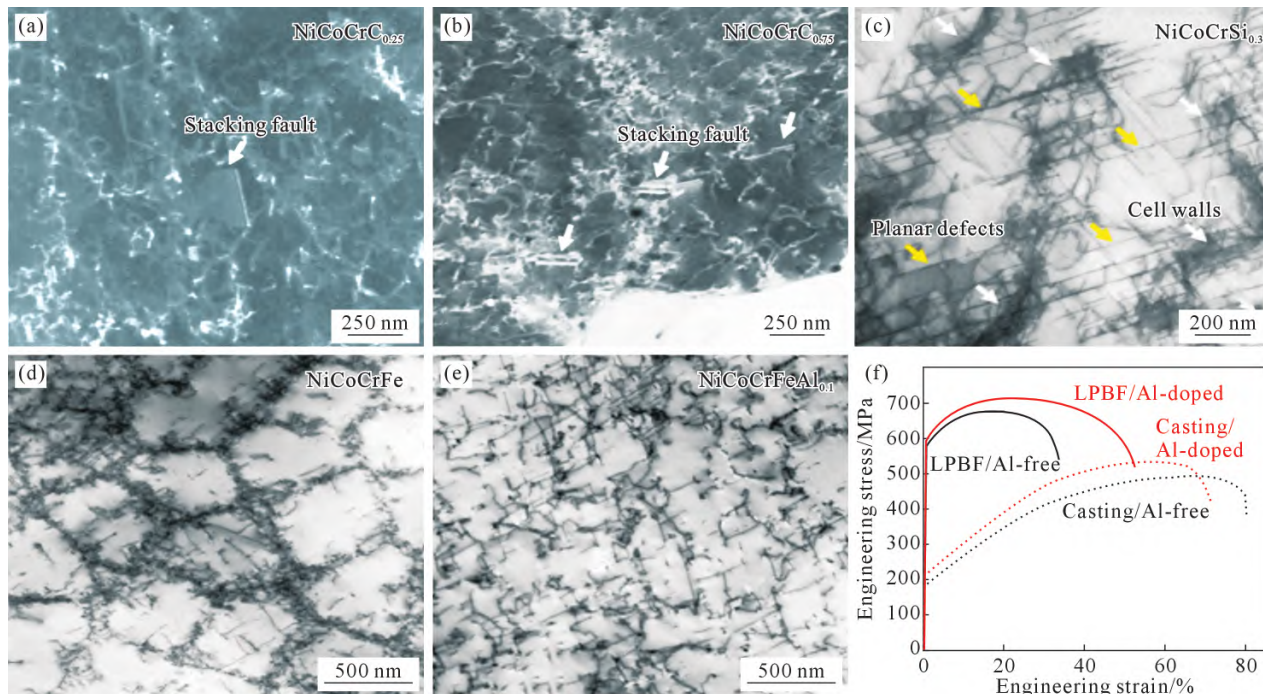


图 11 激光粉末床熔融合金胞状组织及力学性能调控:(a, b)不同 C 含量 CoCrNi 合金胞状组织差异;(c) Si 添加对胞状组织影响;(d~f) Al 添加 NiCoCrFe 合金胞状组织及力学性能差异^[80-82]

Fig.11 Tailoring of the cellular structure and mechanical properties of LPBF-fabricated alloys: (a, b) differences in the cellular structure of CoCrNi alloys with varying C contents; (c) effect of Si addition on the cellular structure; (d~f) evolution of both the cellular structure and mechanical properties of NiCoCrFe alloys with added Al^[80-82]

11c所示,Jung等^[81]指出在CoCrNi中熵合金添加Si元素可降低层错能,在激光粉末床熔融NiCoCrSi_{0.3}合金沉积态组织中观察到大量的层错。合金屈服强度为929 MPa,抗拉强度达到1 264 MPa,总伸长率为14%。Niu等^[82]指出体系层错能降低一方面可使完全位错转变为扩展位错,另一方面促使热循环过程位错由胞状滑移转为平面化滑移,使得位错分布趋向于均匀。针对此,Niu等^[82]在FeCoCrNi合金中加入2.4%(原子分数)的Al实现胞状组织位错组态调控,如图11d,e所示,使得合金屈服强度由560 MPa提升至585 MPa,抗拉强度由673 MPa提升至712 MPa,总伸长率由34%提升至53%。此外,作者指出层错的形成有效的消耗了热循环引起的应力,从而抑制了裂纹的扩展。总结上述规律,胞状组织位错组态的变化与层错能相关。随合金层错能降低,扩展位错成为主导,并促进其空间分布呈现由胞壁聚集向均匀化的演变趋势。尽管如此,胞状组织位错组态变化与力学性能关联关系仍不清晰,仍未建立系统完善的胞状组织位错组织-力学性能的一体化调控理论。

通过添加合金元素引入纳米强化相亦是提升激光粉末床熔融合金力学性能的有效途径。例如,Park等^[83]在Fe₅₀Co₁₈Cr₁₅Ni₁₀合金中通过添加Mo元素增强胞状组织的溶质偏析并引入 μ 相。归因于胞状组织背应力强化和显著提升的滑移位错阻碍能力,合金屈服强的由485 MPa提升至755 MPa,抗拉强度由613 MPa提升至967 MPa,塑性由37.3%提升至45.3%。Jiang等^[84]通过添加N元素在胞状组织中引入(Mo,W)₃Si₃型析出相,同时提升FCC相的稳定性进而有效调控变形过程的相转变动力学。在实现强度提升同时保持了高的加工硬化和塑性(屈服强度由820 MPa提升至983 MPa,塑性由22%降低至19%)。此外,C元素含量的变化改变了沉积态碳化物含量^[80];Si元素的添加引入了 σ 相和纳米夹杂^[81];较多Al元素的添加可能诱发B2相的形成^[85]。然而,过量的第二相也将损害合金塑性,容易引发成形过程的严重开裂^[85]。

4 总结与展望

结合传统制备工艺中/高熵合金的强韧化机理,激光粉末床熔融技术为制备和开发性能优异的中/高熵合金提供了新技术平台。该技术形成的胞状组织,既是实现强塑性协同的核心,也是性能调控的关键特征。近年来,胞状组织的结构特征及其对合金力学性能的影响引起了国内外学者的广泛关注并取得

重要进展。然而,胞状组织对中/高熵合金变形行为的影响及基于胞状组织的性能调控策略仍亟待深入探索。

胞状组织被视为激光粉末床熔融中/高熵合金突破强度-塑性倒置关系的关键。胞状组织贡献高的位错强化并影响合金变形行为和力学响应。尽管现有研究对胞状组织的强化机制已有较为统一的结论,但对于其增塑机制仍存在争议。虽然现有工作初步证实了胞状组织位错组态对强韧化机制的显著影响,但当前尚缺乏针对性的系统研究。在中/高熵合金的强韧化理论基础上,学者总结胞状组织可通过促进位错增殖、激活孪生或相变机制提升合金加工硬化能力,从而提升塑性。然而,胞状组织因合金体系的不同还将对变形产生多重影响,未有统一的结论。尚需根据胞状组织结构特征建立“位错组态-变形行为-力学性能”的关联关系。

此外,现有的激光粉末床熔融中/高熵合金的性能调控策略中尚缺少针对胞状组织的微观结构设计方法。成形参数的调整对胞状组织的影响有限,难以实现力学性能的突破性提升。通过合金成分设计调控激光粉末床熔融合金相组成和微观组织,进而调控胞状组织位错组态或引入纳米强化相,是提升合金力学性能的有效途径。然而,现有针对激光粉末床熔融中/高熵合金胞状组织调控的成分设计准则尚不成熟。研究报导了合金成分优化通过改变胞状组织位错组态实现进一步的力学性能调控,但具体的微观机制仍有待探索。如何发挥胞状组织强韧化效果是实现胞状组织结构设计、提升激光粉末床熔融中/高熵合金力学性能的关键前提。

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