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增材制造金刚石颗粒增强金属基复合材料 研究进展

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摘要: 金刚石颗粒增强金属基复合材料同时具备金属和金刚石的优良特性, 作为功能材料、耐磨材料等在众多领域有着重要应用。增材制造技术为金属/金刚石复合材料复杂构件的直接成形提供新途径, 极大地增加构件设计的自由度。本文基于常用于制备金属/金刚石复合材料的激光选区熔化、激光熔覆、冷喷涂等几种典型增材制造技术, 从粉末原料和成形过程的主要技术难点、应用场景等方面, 介绍增材制造金属/金刚石复合材料的研究进展。着重讨论成形过程中金刚石飞溅及石墨化的产生原因、影响及主要解决方法, 最后总结增材制造金属/金刚石复合材料面临的挑战和未来发展方向。面临的挑战主要表现在增材制造过程中, 出现金刚石飞溅、金属与金刚石颗粒界面控制、金刚石石墨化、金刚石颗粒破损等问题。其中, 需要解决的问题侧重于优化成形工艺, 实现复合材料致密性、界面结合、金刚石防护等几方面协同控制。

关键词: 增材制造; 金属/金刚石复合材料; 飞溅; 金刚石石墨化

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Research progress in diamond-reinforced metal matrix composites fabricated by additive manufacturing

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Abstract: Diamond-reinforced metal matrix composites, which exhibit unique properties of both metals and diamonds, are used as functional and wear-resistant materials in various fields. Additive manufacturing technology provides a novel approach for fabricating complex components of metal/diamond composites, significantly enhancing the design versatility of components. Based on several key additive manufacturing techniques, including selective laser melting, laser cladding and cold spraying, this paper introduces the research progress in the additive manufacturing of metal/diamond composites. It covers powder raw materials, core processing technologies and practical applications. Emphasis is placed on discussing the causes, consequences and potential solutions for sputtering and diamond graphitization that may occur during the manufacturing process. Finally, the main challenges and future development directions of metal/diamond composites in additive manufacturing are summarized. The main manifestations are as follows: in the additive manufacturing process, problems such as diamond splashing, interface control between metal and diamond particles, graphitization of diamond and damage to diamond particles occur. The key issues to be addressed focus on optimizing the forming process to achieve coordinated control of the composite material's density, interface bonding and diamond protection.

Key words: additive manufacturing; metal/ diamond composites; sputtering; diamond graphitization

金刚石因独特的晶体结构及强的C—C键使其具有优良的耐磨性、耐腐蚀性、导热性及化学稳定性,广泛用于硬质合金、光学玻璃、半导体材料、磨削工具等重要的超硬耐磨表面^[1-3]。其中以金属材料为黏结相,金刚石为增强颗粒的金属/金刚石复合材料,因具有成本低、金刚石颗粒把持力高、导热性高等特点,在众多领域取得重要应用^[4]。传统金属/金刚石复合材料通常采用高温高压烧结、真空热压烧结、微波烧结、熔体浸渗等方法制备^[5-10]。但是,由于工艺技术的限制,对于异型、超薄、内部流道等复杂结构的零件,需要进行复杂的模具设计及机加工处理,而金属/金刚石类复合材料的加工难度和成本较高,制约着材料的发展。

增材制造(additive manufacturing, AM),俗称3D打印技术,是一种以粉末、液体、丝材等为原料,基于零件的三维模型,逐点-逐线-逐层堆积的材料成形技术^[11]。较传统材料制备技术,增材制造技术具有材料总体利用率高、工序少、生产效率高、成品率高、可制造复杂结构零件等优势^[12-13]。目前主要用于金属材料成形的增材制造技术有激光/电子束选区熔化、电子束/电弧熔丝、激光熔覆等。另外,广义的金属增材制造技术还包括热喷涂成形、冷喷涂成形、气相沉积、电化学沉积、摩擦焊块体组焊/堆焊等。金属增材制造技术已在钛合金、镍基合金、铝合金、铁基合金等多种合金体系制备上取得应用^[14-18],因此,众多科研团队关注到增材制造技术在制备金属/金刚石复合材料复杂构件上的潜力,开展了相关研究工作^[19-21]。本工作介绍几种常用的金属/金刚石复合材料增材制造技术,并从原料、成形过程的技术难点、主要应用等方面介绍增材制造金属/金刚石复合材料的研究进展。

1 金属/金刚石复合材料增材制造技术

目前报道的用于制备金属/金刚石复合材料的增材制造技术主要基于粉末原料,包括激光选区熔化、激光熔覆、冷喷涂等。

1.1 激光选区熔化技术

激光选区熔化(selective laser melting, SLM)技术是研究较为广泛的一种增材制造技术^[22],它根据零件的三维模型切片信息,利用激光束对粉末层进行选择性地熔化,形成内部含有金刚石颗粒的细小微熔池,逐层累积最终形成零件。由于激光能量密度高,光斑尺寸小,在金属/金刚石复合材料精密复杂结构的直接成形方面较有优势。

1.2 激光熔覆技术

激光熔覆(laser cladding, LC)技术是利用激光束将熔覆材料与基材熔凝在一起,在基材表面形成一层性能更为优异的表面层。通过激光熔覆技术,在不影响基体材料性质的前提下,可以在金属基材上制备出含有耐磨金刚石颗粒的金属/金刚石耐磨层。相较于传统的电镀、钎焊等工艺,激光熔覆技术可有效提高表层与基体的结合力。激光熔覆技术按熔覆材料的供给方式可分为两类,即预置式激光熔覆和同步式激光熔覆。预置式激光熔覆是将熔覆材料事先置于基材表面的熔覆部位,然后采用激光束辐照扫描熔化。同步式激光熔覆则是将粉末材料经过喷嘴在熔覆过程中同步送入熔池中。

1.3 冷喷涂技术

作为新兴的增材制造和涂层技术,冷喷涂(cold spraying, CS)技术是一种基于高速粒子固态沉积的涂层制备方法。在制备过程中,微粉被加速后撞击到基材上形成涂层或沉积物。加速气体通常预热到低于喷涂材料熔点的温度以提高粒子速度与变形能力,进而提高沉积效率。因为全程处于低温状态,冷喷涂技术可有效避免高温沉积过程中金刚石的热损伤、残余热应力、相变等问题,在金刚石耐磨层制备上具有很大潜力^[23-24]。

2 增材制造用粉末原料

金属/金刚石复合材料通常以粉末作为成形原材料,粉末质量是决定成形零件品质的重要前提条件。制备所用粉末通常由金刚石微粉与金属粉末机械混合而成。

2.1 金属粉末的选择

为保证金属元素的均匀性和粉末的流动性,金属粉末多选用球形或近球形预合金粉末。目前增材制造领域常用的金属粉末粒度范围为15~53 μm(细粉)和53~150 μm(粗粉)。常用金属球形粉末的制备技术主要有雾化法(以气雾化为主,包括真空气雾化技术、电极感应雾化技术等)和等离子法(包括等离子旋转电极雾化技术、等离子熔丝雾化技术、等离子球化技术等)^[25]。根据应用环境对金属材料的要求来确定合金成分,通常遵循两个原则:一是合金体系对于增材制造过程应具备较好适应性,有利于优化成形工艺;二是合金与金刚石应具有较好亲和性,有利于金属材料与金刚石表面结合,提高复合材料的力学性能与工艺性能^[26]。基于上述原则目前用于增材制造金属/金刚石复合

数(激光功率和扫描速度)下复合材料的表面形貌、孔隙率、孔隙形貌和弯曲强度,通过考虑激光束流、熔池和金刚石颗粒之间相互作用,从理论上分析金刚石飞溅产生的机制,飞溅过程示意图和实际观测情况分别如图1和图2所示。飞溅主要分为两类:一类是蒸发引起的飞溅(vapor-driven diamond spattering),一类是夹带引起的飞溅(entrainment-driven diamond spattering)。蒸发引起的飞溅主要与金属蒸气和反冲压力有关。高能束流与金属粉末作用时,金属粉末熔化形成细小的金属熔池,其顶部表面发生剧烈蒸发,蒸气在熔体中产生反向压力,金属蒸气的流动速度高于150 m/s,高速金属蒸气导致金刚石飞溅。金刚石颗粒与液体熔池之间的结合力远小于金属蒸气的推力,因此,金刚石飞溅比金属粉末飞溅更容易触发。另外,金属蒸气诱导产生向上的气流场驱动附近的粉末朝向熔池,形成所谓的夹带现象。夹带的粉末向熔体移动并在高速金属蒸气的冲击下被吹走,引起夹带飞溅。通常夹带驱动和蒸气驱动的金​​刚石飞溅会同时发生,研究表明,蒸气引发的飞溅占总飞溅的小部分,而夹带引发的飞溅占比更大^[39]。

据报道,增加激光能量密度可提高成形件的致密度,但是对飞溅的控制作用有限^[40]。

Constantin等^[35]通过单层内重复铺粉及重复熔化两种方式对工艺进行改进,研究表明,粉层厚度是影响金刚石飞溅的主要原因。图3^[35]为分别使用常规熔化、重复铺粉以及重复熔化3种熔化策略得到不同成形高度时粉末床和成形样品的表面情况。常规熔化和重复铺粉方法得到的熔化表面粗糙,不平整的表面会导致粉层厚度的变化。通过重复熔化可降低表面粗糙度,有效抑制金刚石飞溅,进而抑制成形缺陷形成。

3.2 增材制造过程中金属与金刚石颗粒界面控制

当金刚石与高温熔池接触时,在金属熔体瞬间熔化又快速凝固的过程中界面处会发生复杂的反应,界面反应情况直接影响基体材料对金刚石的把持力、复合材料导热等物理性能。目前增材制造金属/金刚石复合材料的金属基体合金主要包括AlSi系材料、NiTi系材料、CuSnTi系材料、Ti/TiC系材料等,不同合金体系的界面反应不同。研究表明^[36],对于AlSi系基体材料,激光能量密度对AlSi/金刚石复合材料的界面反应有显著影响,在低激光

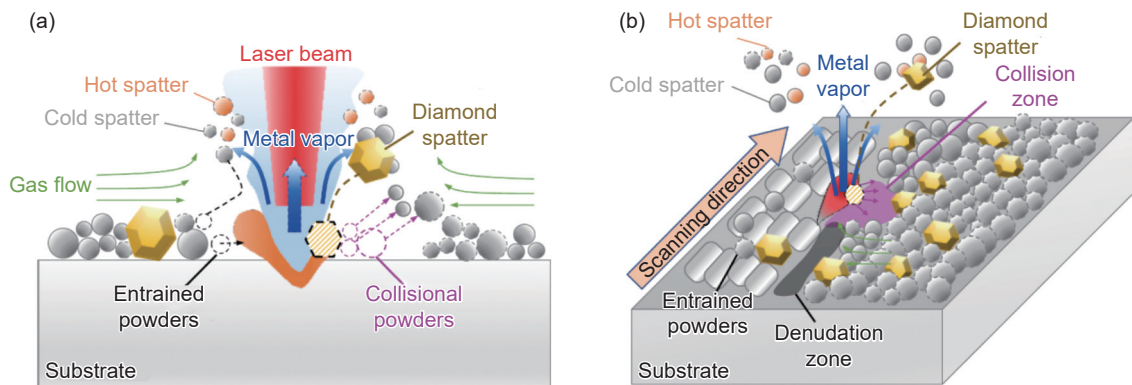


图1 激光束扫描金刚石飞溅过程二维(a)和三维(b)示意图^[38]

Fig. 1 Schematic illustrations of the diamond spattering in two dimensions (a) and three dimensions (b) as the laser beam scans by a diamond grain^[38]

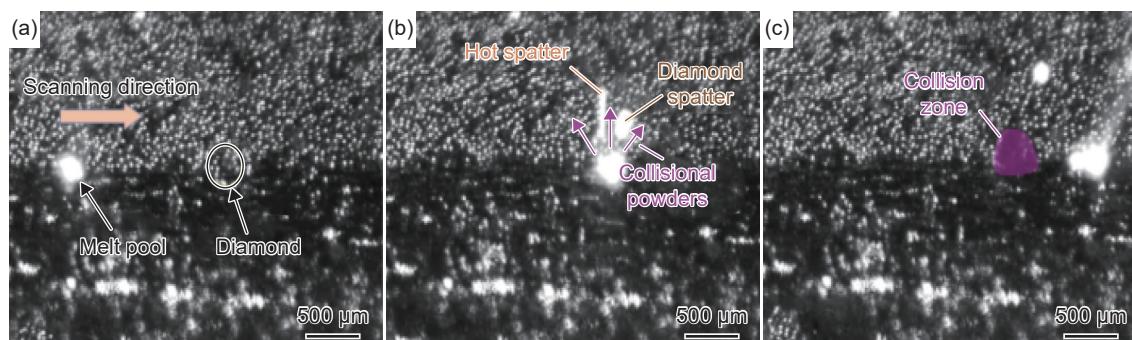


图2 金刚石飞溅过程中的粉末碰撞^[38] (a)扫描方向;(b)碰撞粉末;(c)碰撞区域

Fig. 2 Collision of powder caused by diamond spattering^[38] (a)scanning direction;(b)collisional powder;(c)collision zone

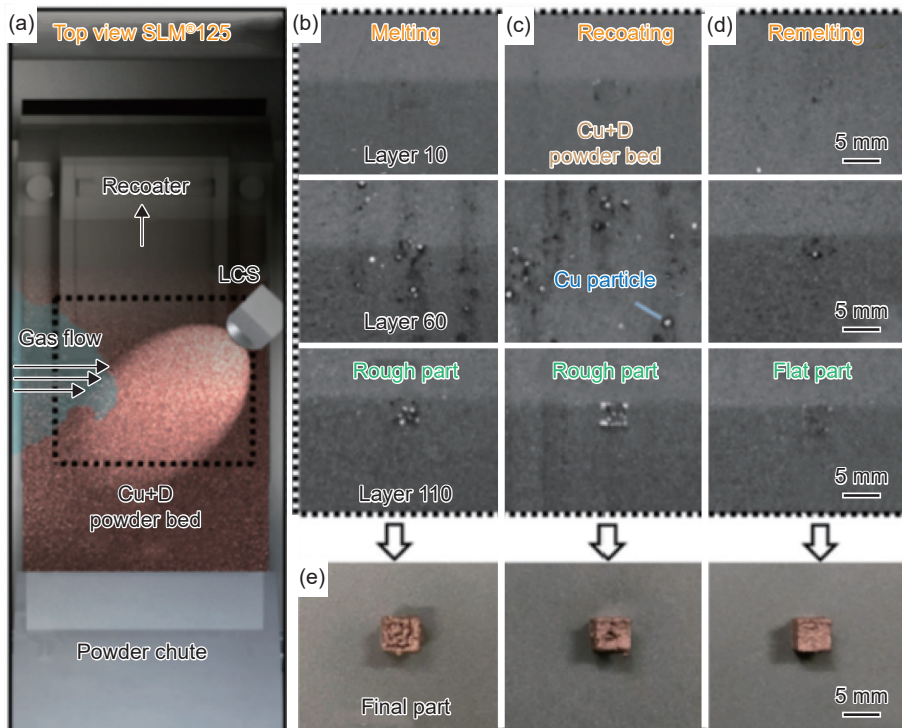


图 3 熔化策略对粉末床和成形样品表面质量的影响^[35] (a) SLM 成形机中单层控制系统示意图; (b)常规熔化后的粉末床图像; (c)重复铺粉后的粉末床图像; (d)重复熔化后的粉末床图像; (e)经过不同熔化策略得到的成形部件

Fig. 3 Effect of the printing strategy on the powder-bed and surface quality of composites^[35] (a) illustration of the layer control system in the SLM printer; (b) image of the powder bed using the melting; (c) image using the recoating; (d) image using the remelting strategy; (e) photos of the printed part using various printing strategies

能量密度下,成形窗口内可得到洁净界面。但是在高激光能量密度下,金刚石表面会形成一层平均厚度约为 15 nm 的氧化物层,同时还会与基体金属元素生成一层厚度不均匀(几十到几百纳米)的 Al_4C_3 碳化物层(如图 4 所示),在金刚石附近还会出现细小棒状 Al_4C_3 碳化物颗粒,降低复合材料导热性能。氧化物的形成主要是由于金刚石表面存在吸附氧或金属粉末原料表面存在氧化层。碳化物的形成主要是由于长时间高温促进金刚石表面

游离碳产生,同时,细小熔池的马格尼对流作用带动表面带有游离碳的金刚石颗粒在熔池内运动,当到达适宜的热力学温度区域时反应生成碳化物。Denkena 等^[33]进行了分别以预合金粉末和元素粉末为原料的 NiTi/金刚石复合材料激光选区熔化成形研究,结果表明,当金属基体为 NiCu 预合金粉末时,成形致密度较高,同时在界面处会形成不同厚度的 TiC 过渡层。在同样含有 Ti 元素的 CuSnTi 系材料及 Ti/TiC 系材料中界面反应同样以生成

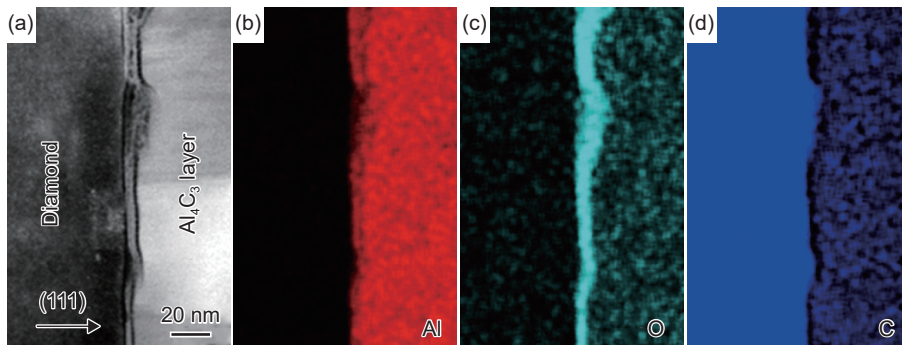


图 4 金刚石(111)/铝合金界面处的金刚石(111) Al_2O_2/Al_4C_3 复合层^[36] (a)界面形貌; (b)Al 的分布; (c)O 的分布; (d)C 的分布

Fig. 4 Diamond (111)/Al interfacial area where a diamond(111)/ Al_2O_2/Al_4C_3 multi-interfacial layer^[36] (a) interface morphology; (b) distribution of Al; (c) distribution of O; (d) distribution of C

TiC 过渡层为主^[32]。除界面处化学反应外,增材制造金属/金刚石复合材料的另一个界面问题是界面处易产生残余应力,由于金刚石与金属基体具有不同热膨胀系数,在增材制造快冷作用下,界面处产生明显的热应力,导致金刚石颗粒周围极易形成裂纹^[35]。

3.3 增材制造过程中金刚石石墨化问题

由于金刚石的热稳定性差,当金刚石受到高能束流冲击或与高温熔池接触时,很可能发生金刚石的石墨化损伤^[41-42]。在金刚石晶体中,每个碳原子都以 sp^3 杂化轨道与另外 4 个碳原子形成共价键,构成正四面体。石墨化是指金刚石原本的 sp^3 杂化键发生断裂,形成石墨 sp^2 杂化键的过程。石墨和金刚石的性质有很大不同,金刚石石墨化将严重影响复合材料的使用性能。通常石墨化行为可采用 XRD、拉曼光谱等方法进行分析, Li 等^[43] 对增材制造石墨烯/金属复合材料拉曼光谱的 D、G 和 2D 峰进行检测,分析轨道杂化情况及石墨烯结构的完整性。目前已有多项研究对增材制造成形金属/金刚石复合材料中金刚石石墨化现象进行报道。Rommel 等^[29,44] 研究金刚石和熔融金属的界面反应,结果表明,热损伤和界面反应仅发生在与熔池接触的金刚石颗粒中,而不是激光直接照射的金刚石颗粒。这是由于激光选区熔化过程中激光高斯热源的快速加热和不均匀的能量分布导致熔池中温度场极不平衡,瞬时温度可能超过 2000 °C,

金刚石颗粒经历严重的高温热传导和热冲击。Zhou 等^[45] 研究指出,激光选区熔化成形过程中,由于层与层之间的二次加热及逐层累积的过程中热量积累增加金刚石的石墨化。同时, Iravani 等^[46] 发现,合金中 Fe 和 Ni 元素可作为催化剂加剧成形过程中金刚石石墨化。Ma 等^[47] 以 CuSn 合金为黏结金属,系统研究增材制造金属/金刚石耐磨材料的温度场-石墨化程度-复合材料耐磨性能之间的定性关系,得到工艺-温度-石墨化的对应情况,如图 5 所示。研究发现,激光的能量输入远低于金刚石石墨化理论阈值,所以激光的直接轰击并不会引起金刚石石墨化,金刚石石墨化主要是由于金刚石与高温熔池的接触。通过模拟熔池中金刚石的温度场演变和对金刚石颗粒微观结构的表征,金刚石石墨化的临界温度确定为 1491.6 °C。在临界温度以下,金刚石不会发生石墨化,复合材料磨损率低于 0.01%。温度升高导致石墨化加剧,同时耐磨性能受到影响,随着温度从 1491.6 °C 升高到 1896.1 °C,摩擦深度由 91.79 μm 增加到 118.29 μm ,同时复合材料磨损机制发生变化。因此,通过调控熔池温度可在一定程度上抑制金刚石石墨化。

3.4 金刚石颗粒的破损

冷喷涂技术存在金刚石颗粒破损问题。虽然冷喷涂技术中低成形温度有效避免金刚石高温损伤,但是也存在技术难题。因为冷喷涂过程中高速

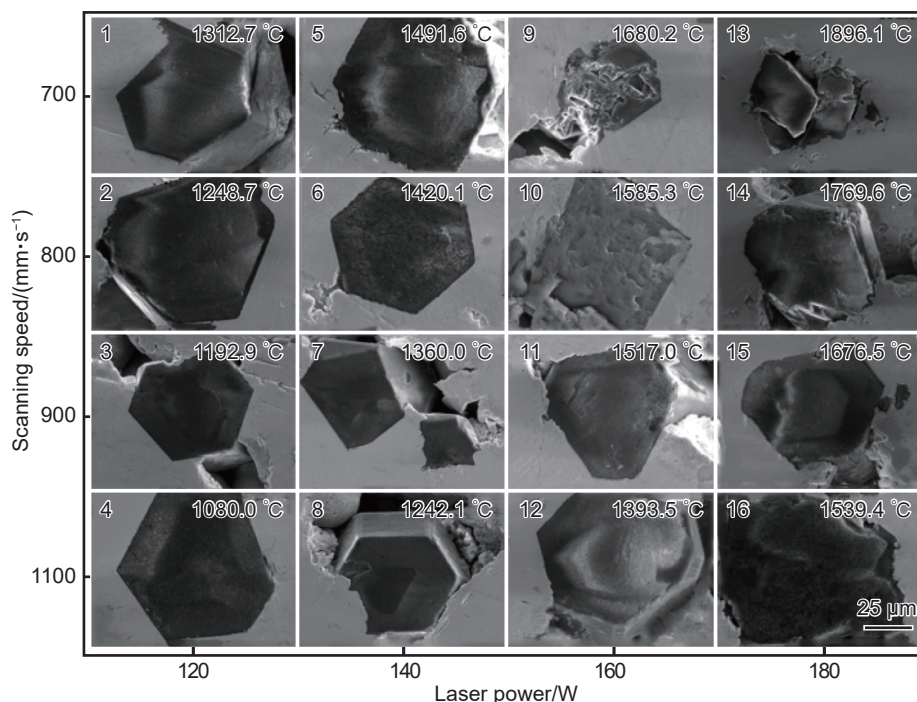


图 5 在不同成形工艺条件下金刚石颗粒的扫描电子显微镜图像^[46]

Fig. 5 Scanning electron microscopic images of the microstructures of diamond particles with different process parameters^[46]

粒子直接冲击基体,对于脆性颗粒来说,在高速冲击过程中极易出现金刚石破碎。Yin等^[48]通过原位观测方法研究在冷喷涂过程中,金刚石反弹和破损情况,将金刚石破损时冲击速度定义为破损速度。通过分析发现,沉积样品内部破损金刚石颗粒的含量与冲击速度和破损速度的比值有关,同时以Ni为过渡层,在金刚石表面包覆一层Cu作为缓冲层,可有效缓解金刚石冲击损伤^[34]。

4 主要应用领域

金刚石因其独特的物理化学特性,如极高的硬度、优异的耐磨性、低热膨胀系数等,广泛应用于硬质材料加工的砂轮、钻头、磨盘等工具。用于表面精磨的多孔金刚石砂轮是金属/金刚石复合材料的重要应用^[49],内部孔隙在金刚石砂轮中起着重要作用,可以用来存储精磨过程中的冷却液,防止磨削过程中温度上升引起的工件损伤,同时协调磨损产生的磨屑。随着工业产品对磨削质量的要求越来越高,金刚石砂轮的结构越来越复杂,同时对内部孔隙的均匀性和可控性要求越来越高。增材制造技术为复杂结构金刚石砂轮的加工,尤其是内部

孔隙的控制提供了一种高效低成本的解决方案。Tian等^[50]采用激光选区熔化技术制备具有多种胞状结构的多孔铝合金/金刚石复合材料,孔隙率在30%~70%范围内可调,性能优于传统电镀法加工的金刚石砂轮。在钻探工具应用上,Wu等^[51]加工了含有网格状结构的CoCrMo黏结金刚石钻头,利用增材制造技术的优势,优化切削面结构,该钻头可用于破碎坚硬的岩层,提高硬岩钻井效率。Yang等^[52]采用增材制造技术加工金属/金刚石磨盘,实现了磨盘结构的设计,解决了金刚石颗粒的可控分布问题。张绍和等^[53]提出栅格状金刚石钻头、超薄网状锯片、蜘蛛网状金刚石砂轮等异型金刚石工具(如图6所示)的增材制造生产工艺。表1^[32,47,50,54]列举了几种增材制造金属及金属/金刚石耐磨材料的摩擦性能。

由于金刚石是自然界导热性能最好的材料之一,利用金刚石颗粒增强较高热导率的金属基体(如Al、Cu和Ag)可得到兼具极高导热性能和较低热膨胀系数的复合材料。增材制造技术可实现金刚石颗粒的界面控制,进而增强复合材料的整体导热性能,因此增材制造金刚石/金属复合材料在微电子散热材料领域也受到极大关注。

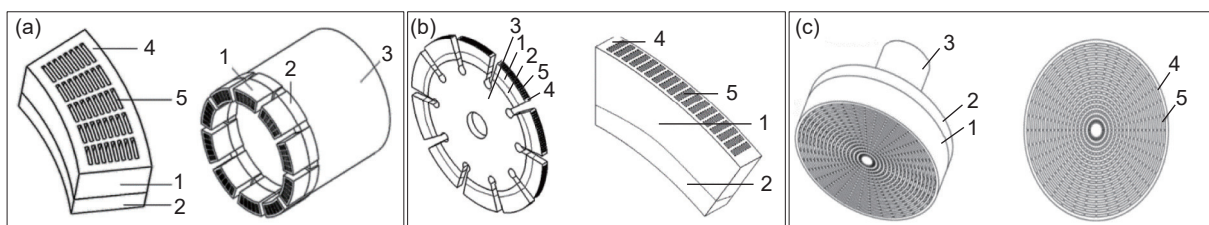


图6 增材制造异型金刚石工具示意图^[53] (a)栅格状金刚石钻头;(b)网格状金刚石锯片;(c)蜘蛛网状金刚石砂轮

Fig. 6 Schematic diagrams of some additive manufacturing special-shaped diamond tools^[53] (a) grid shaped diamond drill bit; (b) grid shaped diamond saw blade; (c) spider web diamond grinding wheel

表1 增材制造金属及金属/金刚石耐磨材料的摩擦性能

Table 1 Frictional properties of additive manufacturing metals and metal/diamond wearable materials

Preparation technology	Composition	Friction and wear coefficient	Mass loss/ ($\text{g} \cdot \text{min}^{-1}$)	Wear amount of grinding disc/%
Selective laser melting	Ti/TiC	<0.2 ^[54]		
Selective laser melting	AlSi10Mg/diamond			17.2 ± 2.4 ^[50]
Selective laser melting	CuSn10/diamond	0.55, 0.62 ^[47]		
Selective laser melting	Cu-Sn-Ti/diamond		0.0125 ^[32]	

目前增材制造金属/金刚石复合材料的应用主要在于金刚石的耐磨和导热性能,所以基本用于硬质材料,虽然目前在航空航天领域还没有明确报道,但是凭借其优良的导热性能在航空航天和微电子领域具有较大的应用潜力。

5 结束语

相较于传统粉末冶金制备技术,金属/金刚石复合材料增材制造技术成本略高。同时,金刚石颗粒的存在导致金属熔池稳定性较差、金刚石颗粒与

高温熔池接触时易发生损伤、成形质量和精度的控制难度较大等问题。迄今为止,国内外关于金刚石颗粒增强金属基复合材料的研究还较少,仍处于基础研究阶段,成形样品的性能和稳定性与传统方法制备的金属/金刚石复合材料仍有一定差距。同时,复合材料致密化与缺陷形成机制及高温熔池作用下金刚石石墨化机理尚不明确。因此,在复合材料的结构与成分设计、增材制造工艺优化等方面仍然需要大量研究工作,通过系统实验建立工艺参数-成型质量-金刚石完整度-性能之间的关系,指导金属/金刚石复合材料构件的成形工艺。其中,重点需要解决的问题是优化成形工艺,实现复合材料致密性、界面结合、金刚石防护等方面协同控制。

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