

MAX 相陶瓷增强金属基复合材料: 制备、性能与仿生设计

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摘要: 由于原子间存在共价键、金属键与离子键的混合键合状态, MAX 相陶瓷兼具金属和陶瓷材料的性能特点, 并且常与金属之间表现出良好的润湿性, 有助于形成强界面结合, 独特的层状原子结构使 MAX 相陶瓷表现出良好的断裂韧性、阻尼与自润滑性能。因此, 作为金属基复合材料的增强相, MAX 相陶瓷具有显著优势, 本文着重介绍相关研究进展。目前, MAX 相陶瓷增强金属基复合材料主要通过搅拌铸造、粉末冶金和熔体浸渗等途径制备, 得到的复合材料表现出优于金属基体的强度、硬度与模量, 同时还具备良好的耐磨、导电、抗电弧侵蚀等性能。此外, 借助真空抽滤、冰模板等工艺可实现超细片状 MAX 相陶瓷粉体的择优定向排列, 然后利用金属熔体浸渗多孔陶瓷骨架, 可获得具有类贝壳结构的 MAX 相陶瓷增强金属基仿生复合材料, 进一步提升材料的强韧性能。MAX 相陶瓷增强金属基复合材料在承载、电接触等应用领域具有显著优势和广阔前景。

关键词: MAX 相陶瓷; 金属基复合材料; 仿生设计; 力学性能; 熔体浸渗; 专题评述

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Metal Matrix Composites Reinforced by MAX Phase Ceramics: Fabrication, Properties and Bioinspired Designs

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Abstract: MAX phase ceramics, with their mixed covalent-metallic-ionic atomic bonds, can uniquely combine the advantages of both metals and ceramics, offering a series of distinctive characteristics. The particular layered atomic structure further endows them with decent fracture toughness, good damping capacity, and self-lubricating property. As such, MAX phase ceramics are more appealing to serve as reinforcements for metal matrix composites (MMCs) than conventional ceramic materials. Here, we focused on the development. To date, fabrication of MMCs reinforced by MAX phase ceramics still involves the use of stir casting, powder metallurgy, and melt infiltration techniques. The obtained composites made by different methods may display distinct differences in their structural characteristics, show notable enhancement in strength, hardness, and stiffness as compared to their metal matrices, and exhibit good wear resistance, high electrical conductivity and remarkable arc erosion resistance. Moreover, ultrafine MAX phase

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platelets can be preferentially oriented and aligned, *e.g.*, by using vacuum filtration or ice templating techniques. By infiltrating metal melt into partially sintered porous ceramic scaffolds, bioinspired composites with nacre-like architectures can be obtained, thereby affording further improvement in strength and fracture toughness. Sufficient combinations of mechanical and functional properties enable the MMCs reinforced by MAX phase ceramics promising for a variety of applications, such as load-bearing structures, electrical contact materials. These composites can offer enhanced strength, stiffness, and wear resistance, making them ideal candidates for these applications.

Key words: MAX phase ceramics; metal matrix composite; bioinspired design; mechanical property; melt infiltration; perspective

金属基复合材料以其比强度和比模量高、热膨胀系数低等优异性能,作为结构材料被广泛应用于航空、航天以及汽车等领域^[1-4]。陶瓷具有高模量、高硬度以及良好的热稳定性和化学稳定性,是金属基复合材料常用的增强相组元。其中,MAX 相陶瓷是一类具有 $M_{n+1}AX_n$ (M 为前过渡金属; A 为主族元素; X 为碳和/或氮元素; $n = 1, 2$ 或 3) 组成的层状化合物,其原子间具有独特的共价键、金属键与离子键的混合键合状态,从而兼具金属与陶瓷材料的性能特点,并表现出良好的断裂韧性、阻尼、自润滑等性质^[5-8]。同时,MAX 相陶瓷与金属间通常具有良好的润湿性,有利于形成强界面结合。因此,与传统陶瓷材料相比,MAX 相陶瓷有望用作金属基复合材料更具吸引力的理想增强相。本文首先总结了 MAX 相陶瓷作为增强相在金属基复合材料制备中的诸多优势,然后介绍了利用不同制备技术制备的 MAX 相陶瓷增强金属基复合材料,重点关注其微观结构、力学性能与功能特性,并详细阐述了利用仿生结构设计与构筑策略提升复合材料强韧性能的最新研究进展。在此基础上,本文探讨了 MAX 相陶瓷增强金属基复合材料研究中的关键问题以及未来可行的发展方向,旨在为高性能 MAX 相陶瓷增强金属基复合材料的开发与应用提供参考。

1 MAX 相陶瓷作为增强相的优势

MAX 相陶瓷独特的混合键合状态与层状原子结构使其作为金属基复合材料的增强相组分,具备以下显著优势(图 1):

- 1) 高模量、大硬度、良好的热稳定性、抗腐蚀和抗氧化性能,这些特点得益于强共价键与离子键;
- 2) 具备一定的导电和导热性能,且易于加工,这些特性来源于金属键;
- 3) 优良的断裂韧性与阻尼性能,这些特点归功于层状原子结构;

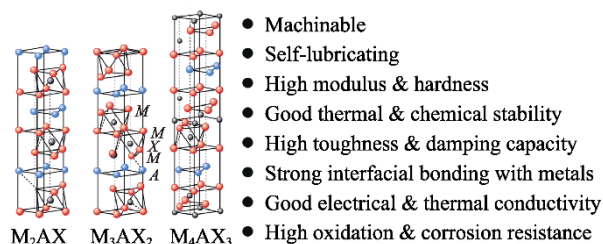


图 1 MAX 相陶瓷的原子结构与性能优势^[6]

Fig. 1 Atomic structures and advantageous properties of MAX phase ceramics^[6]

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4) 自润滑性能,有助于提高耐磨性和减磨性,这是因为原子层间结合力较弱,容易发生滑动;

5) 易与金属形成强界面结合,这得益于金属界面的润湿性和界面反应可控性。

2 MAX 相陶瓷增强金属基复合材料的制备、结构和性能

迄今,文献已报道了多种 MAX 相陶瓷增强金属基复合材料,涉及的 MAX 相陶瓷种类包括 Ti-Al-C、Ti-Si-C、Cr-Al-C、Ta-Al-C、Ti-Al-N、V-Al-C 等体系,金属基体包括铝、镁、铜、镍、银、钛、铁等^[9-12]。这些复合材料主要通过搅拌铸造、粉末冶金和熔体浸渗三种工艺制备。不同的制备工艺往往会引起显著不同的结构特点,从而在很大程度上决定复合材料的性能。因此,本文将基于制备工艺介绍现有 MAX 相陶瓷增强金属基复合材料的结构与性能。

2.1 搅拌铸造

搅拌铸造常用于制备金属基体熔点较低的 MAX 相陶瓷增强铝基和镁基复合材料。该工艺通过搅拌将 MAX 相陶瓷颗粒分散于金属熔体中,经铸造后凝固得到复合材料。为了确保熔体流动性,利用搅拌铸造制备的金属基复合材料中 MAX 相陶瓷含量

通常较低(一般不超过体积分数 20%), 且呈现弥散结构, 即陶瓷颗粒均匀分散在金属基体中^[11,13]。

以搅拌铸造的 Ti_2AlC 颗粒增强 AZ91D 镁合金复合材料为例^[13-16](图 2(a~c))。该复合材料的金属相与陶瓷相之间不发生化学反应, 并呈现强界面结合。在外加载荷下, 裂纹优先产生于陶瓷相, 并沿着金属基体中的晶界扩展, 直至不同陶瓷颗粒中的裂纹相互连接, 导致材料最终断裂。该损伤与断裂模式明显不同于以 SiC 、 TiC 等其他陶瓷增强的镁基复合材料, 即在外加载荷下, 复合材料倾向于发生两相界面脱黏, 进而造成材料提早失效。因此, 加入 Ti_2AlC 陶瓷可以有效提高材料的抗拉与抗压强度及耐磨性, 同时获得良好的阻尼性能, 而当陶瓷添加量过高(体积分数>10%)时, 陶瓷颗粒易发生团聚, 导致材料脆性增大, 从而降低抗拉强度。

此外, 热挤压等后续热变形处理可以促使 MAX 相陶瓷在金属基体中择优定向排布^[11, 14-15](图 2(d)), 获得各向异性的力学性能。对于以 Ti_2AlC 颗粒增强的镁基复合材料, 其沿平行于挤压方向的抗拉强度高于垂直方向的抗拉强度, 同时当摩擦方向与挤压方

向平行时, 耐磨性也明显更优^[15](图 2(e, f))。

2.2 粉末冶金

粉末冶金工艺是将 MAX 相陶瓷粉末与金属粉体混合后烧结进行材料成型, 已被用于制备铝、镍、钛、铜、银等多种体系的 MAX 相陶瓷增强金属基复合材料, 可获得优异的力学性能^[9,17-19]。以 Ti_3AlC_2 颗粒增强的 2009 铝合金复合材料为例^[17](图 3), 铝合金与陶瓷复合粉体经球磨混合、冷压成坯、热压烧结及热挤压变形后得到的复合材料, 其中 Ti_3AlC_2 颗粒倾向沿平行于挤压方向分布, 并与铝合金基体之间呈强界面结合, 这有利于促进载荷在两相之间传递, 提高增强相的强化效率。在材料变形破坏过程中, Ti_3AlC_2 颗粒可通过与基体脱黏、拔出、分层等微观机制有效消耗机械能, 在提高强度的同时, 保证材料良好的塑性, 使该材料比 SiC 、 TiO_2 、 TiB_2 等其他陶瓷增强 2 系铝合金复合材料表现出更为优异的强塑性匹配。

除界面润湿现象外, MAX 相陶瓷还可与金属基体之间发生可控的界面反应, 促进界面结合。以粉末冶金工艺制备的 Ti_3AlC_2 颗粒增强铜基复合材料

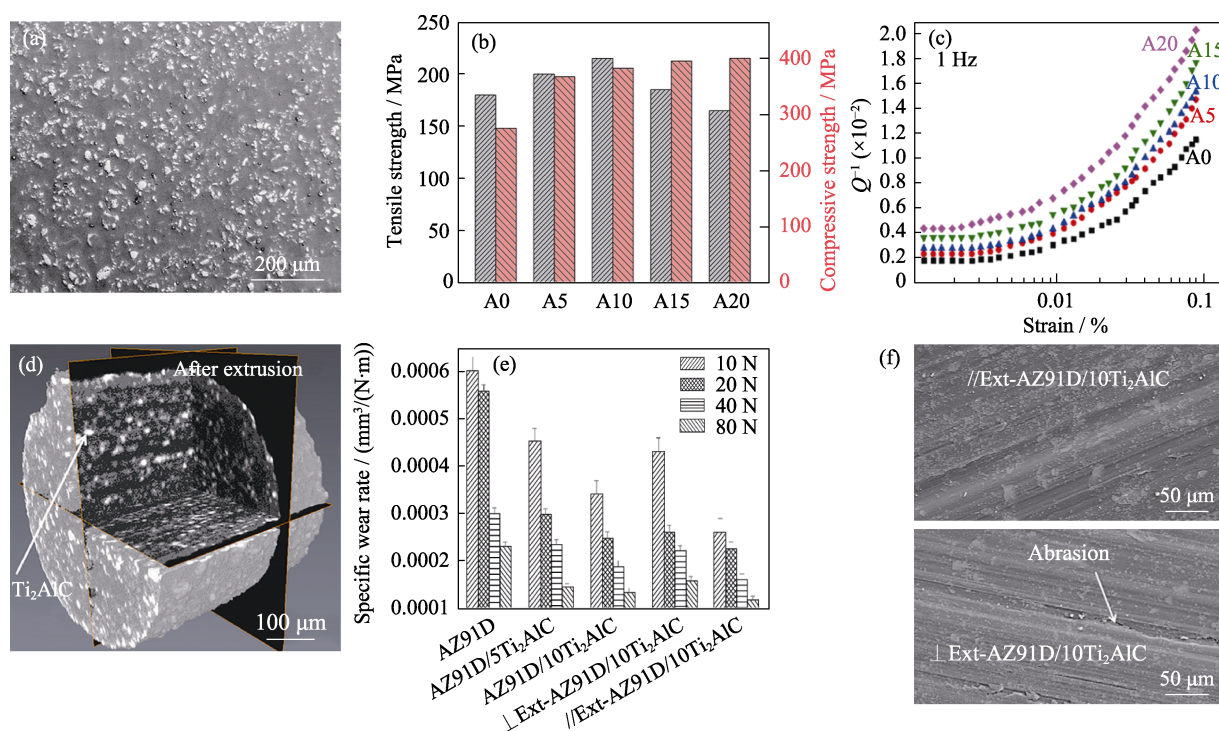


图2 搅拌铸造工艺制备的 AZ91D- Ti_2AlC 复合材料的微观结构与力学、阻尼及磨损性能

Fig. 2 Structure and mechanical, damping and friction properties of AZ91D- Ti_2AlC composites fabricated by stir casting technique (a) Representative structure of as-cast AZ91D- Ti_2AlC composite with white regions showing Ti_2AlC phase^[13]; (b) Tensile and compressive strengths of the as-cast composites with different Ti_2AlC contents (A0–A20 denoting the volume fraction of Ti_2AlC from 0 to 20%)^[13]; (c) Variations in the internal friction (Q^{-1}) with strain amplitude^[16]; (d) 3D structure of AZ91D- Ti_2AlC composite after hot extrusion^[14]; (e) Specific wear rates of as-cast and extruded composites compared to the AZ91D matrix under different applied loads^[15]; (f) Wear morphologies of extruded composite after sliding along parallel (//) and perpendicular (⊥) directions with respect to the extrusion axis^[15]

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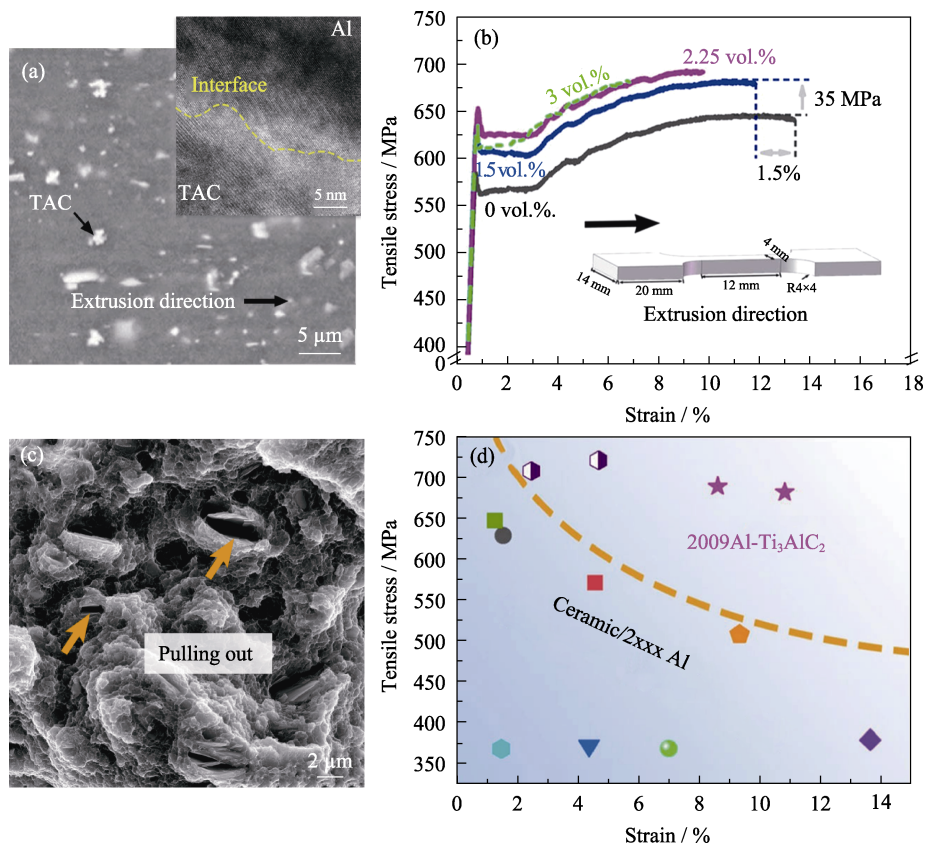


图 3 粉末冶金工艺制备的 2009Al-Ti₃AlC₂ 复合材料的微观结构、力学性能与断后形貌^[17]
Fig. 3 Structure, mechanical properties, and fracture morphologies of 2009Al-Ti₃AlC₂ composites made by powder metallurgy technique^[17]

(a) Structure of 2009Al-Ti₃AlC₂ composite after extrusion and inverse fast Fourier transformation image of interface between Ti₃AlC₂ particle and Al alloy matrix. TAC: Ti₃AlC₂; (b) Tensile stress-strain curves of the composites with different Ti₃AlC₂ contents; (c) Fracture morphologies of composite after tensile fracture; (d) Comparison of tensile strength and ductility of 2009Al-Ti₃AlC₂ composites with other 2xxx Al alloy composites reinforced by ceramics. Adapted with permission from Ref. [17] Copyright 2023, Springer

为例^[20-22](图 4), Ti₃AlC₂ 中的 Al 在高温下扩散至铜基体中, 并在 Ti₃AlC₂ 与铜之间形成由 Cu(Al)固溶体和 TiC_x 组成的过渡层, 促进界面处的载荷传递, 从而提高抗弯强度。当陶瓷含量较高时(体积分数>10%), 陶瓷相的团聚导致复合材料的抗拉强度随陶瓷含量的增高而下降。复合材料的电导率和耐磨性均与 Ti₃AlC₂ 含量有关, 随着 Ti₃AlC₂ 含量增加, 复合材料电导率逐渐降低, 而磨损率则表现为先降后升的趋势, 当 Ti₃AlC₂ 体积分数为 40%时, 磨损率达到最小值。此外, 该复合材料还表现出良好的抗电弧侵蚀性能, 有望用作电接触材料。MAX 相陶瓷与金属基体之间的界面反应还可用于原位生成其他种类的增强相。例如, 在 Ti₂AlC 与 TiAl 组成的复合体系中, 两相可原位反应生成硬度更高的 TiAl₃ 等金属间化合物, 从而获得更为优异的强化和硬化效果^[23-24]。

2.3 熔体浸渗

在无压或真空/压力辅助条件下将金属熔体渗入预成型的多孔 MAX 相陶瓷骨架中, 可制备得到具有微观三维互穿结构的 MAX 相陶瓷增强金属基

复合材料, 即复合材料中各组分在三维空间中均保持连续并且相互穿插。该结构有利于保留各组元的性能优势, 并通过促进组元间的高效载荷传导与机械互锁, 提高材料的损伤容限^[25-26], 从而有望同步获得优异的力学性能与功能性质, 实现结构-功能一体化。

熔体浸渗工艺可用于开发 MAX 相陶瓷增强的铜基和银基电接触材料, 实现材料的性能提升和功能优化。材料中连续的陶瓷相起强化作用, 而连续的金属基体为材料提供高导电和导热性能。例如, 利用银熔体无压浸渗预烧结的 Ti₃SiC₂ 多孔骨架, 可获得高陶瓷含量(体积分数>50%)的三维互穿结构 Ag-Ti₃SiC₂ 复合材料^[27](图 5)。银和陶瓷两相三维互穿可促进应力传导并限制各相内部及两相界面处的损伤演化, 同时 Ti₃SiC₂ 陶瓷的自润滑性质还可有效减轻磨损。与粉末冶金工艺制备的 Ag-Ti₃SiC₂ 复合材料相比, 三维互穿 Ag-Ti₃SiC₂ 复合材料的硬度、抗弯强度、断裂韧性与耐磨性能明显更高, 同时兼具良好的导电性能。并且, 随着三维互穿结构从微

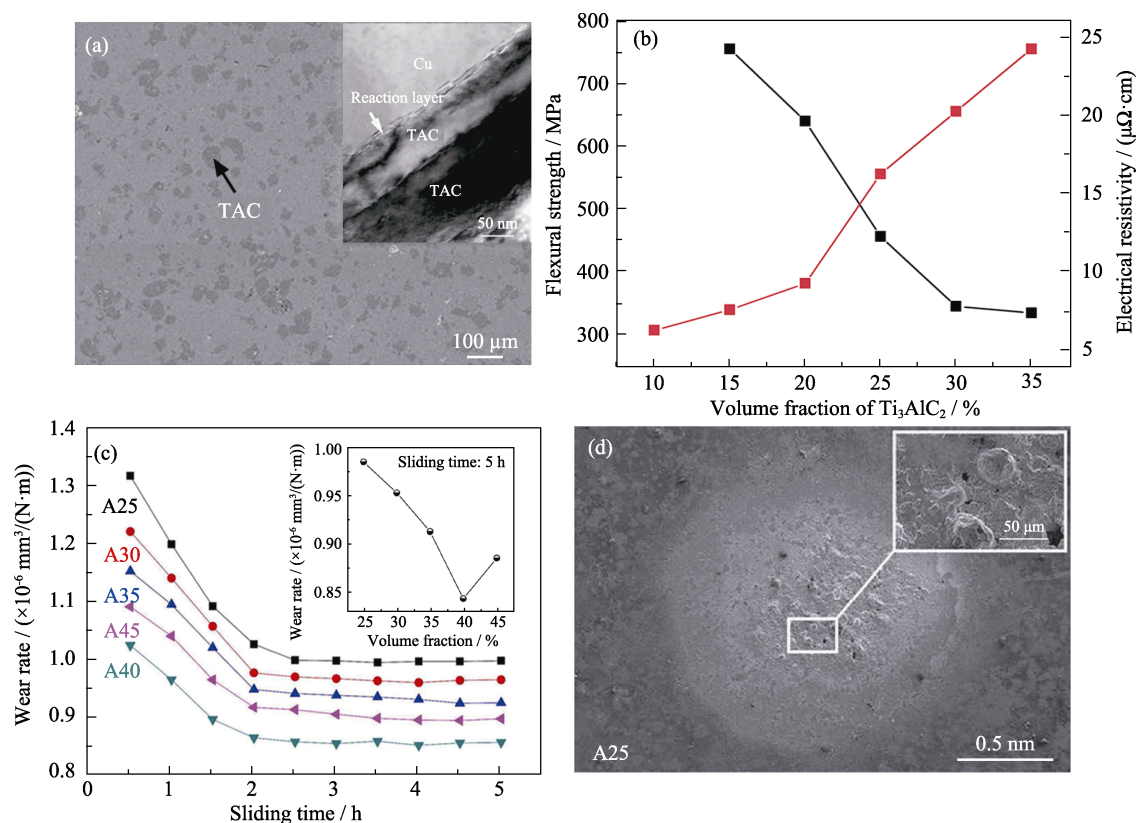


图4 粉末冶金工艺制备的 Cu-Ti₃AlC₂ 复合材料的微观结构、力学性能与功能性质

Fig. 4 Structural, mechanical and functional characteristics of Cu-Ti₃AlC₂ composites fabricated by powder metallurgy technique (a) Structure of Cu-Ti₃AlC₂ composite and its reaction layer between Ti₃AlC₂ (TAC) and Cu phases^[20]; (b) Variations in the flexural strength and electrical resistivity of the composites as a function of the volume fraction of Ti₃AlC₂ phase^[20]; (c) Wear rate of the composites against increasing sliding time with inset showing the dependence of wear rate on the volume fraction of Ti₃AlC₂ phase at a sliding time of 5 h^[22]; (d) Surface morphology of Cu-25% Ti₃AlC₂ composite after arc erosion^[20]. (a, b, d) Adapted with permission from Ref. [20] Copyright 2017, Taylor & Francis. The inset in (a) is adapted with permission from Ref. [21] Copyright 2007, Elsevier. (c) Adapted with permission from Ref. [22] Copyright 2019, Springer

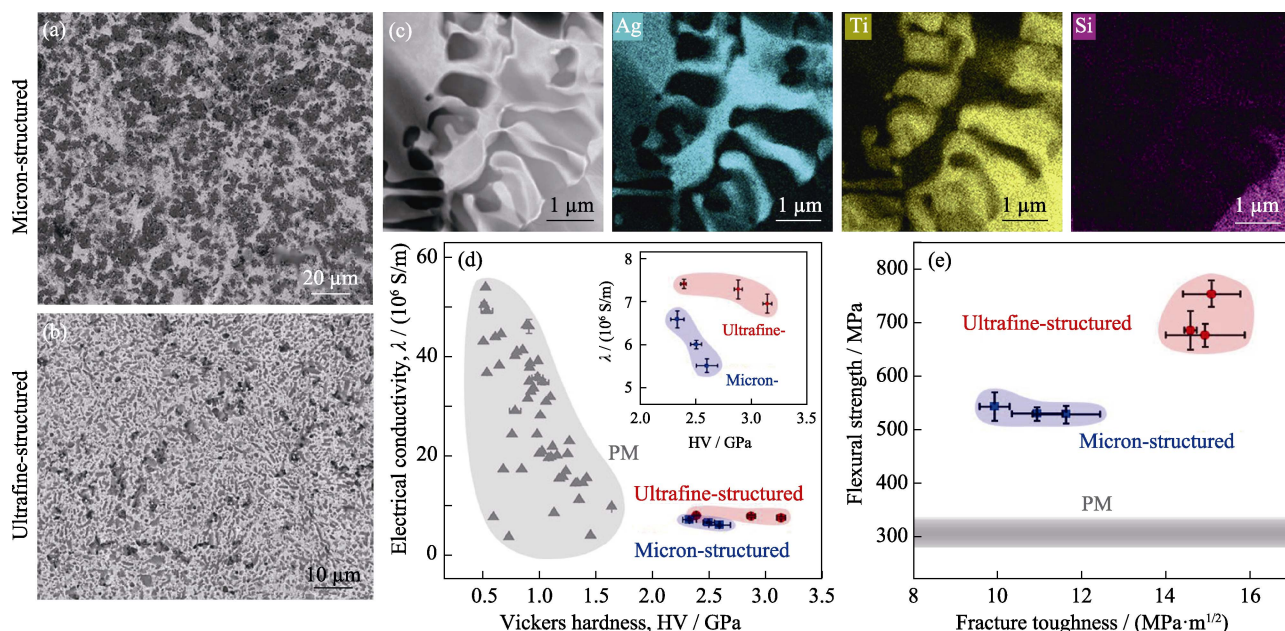


图5 熔体浸渗工艺制备的 Ag-Ti₃SiC₂ 复合材料的微观结构及力学与导电性能^[27]

Fig. 5 Structural, mechanical and electrical characteristics of Ag-Ti₃SiC₂ composites made by melt infiltration technique^[27] (a, b) Structures of melt infiltrated Ag-Ti₃SiC₂ composites with different structural dimensions of micron- (a) and ultrafine (b) length scales with dark regions showing Ti₃SiC₂ phase; (c) Chemical characteristics of the micron-structured Ag-Ti₃SiC₂ composite; (d, e) Electrical conductivity versus Vickers hardness (d) and flexural strength versus fracture toughness (e) of the melt infiltrated Ag-Ti₃SiC₂ composites with other Ag-MAX phase composites fabricated by powder metallurgy technique. Adapted with permission from Ref. [27] Copyright 2023, Tsinghua University Press

米细化到亚微米尺度,材料的硬度、强度、电导率和断裂韧性可同步提高。优异的综合性能与简便的制备工艺使该材料有望满足未来电接触领域的应用需求。

3 MAX 相陶瓷增强金属基复合材料仿生结构设计及强韧化

尽管由简单组元在相对温和的条件下自组装而成,天然生物材料凭借其巧妙的微观结构设计——软硬相复合并且形成跨越多尺度的复杂层级结构,表现出优异的力学性能和独特的功能特性^[28]。这种高效的性能优化策略可为研制高性能人造材料提供重要启示。

通过设计并构筑类似典型生物材料的结构,可获得 MAX 相陶瓷增强金属基仿生复合材料,在结合组元性能优势的同时进一步提高强韧性能。例如,利用真空抽滤可实现 Ti_3AlC_2 薄片的择优定向排列,

将得到的坯体部分烧结成多孔陶瓷骨架并利用 AZ91D 镁合金熔体进行浸渗,可获得具有类似天然贝壳结构的 Ti_3AlC_2 增强镁基仿生复合材料^[29](图 6)。金属与陶瓷两相均具有超细尺度并且在三维空间呈交替定向排列,各自保持连续且相互穿插,与天然贝壳珍珠层微观结构类似^[30]。该仿生结构在强化材料的同时,可通过 MAX 相薄片的桥接与拔出阻碍裂纹扩展,降低材料对拉应力的敏感性,使材料的抗压与抗弯强度同时超过 1 GPa,并具有良好的断裂韧性($>16 \text{ MPa}\cdot\text{m}^{1/2}$)。此外,高密度两相界面有助于提高材料的阻尼性能,使得仿生材料的阻尼系数高于基体镁合金。高强度与低密度赋予该仿生材料超高比强度(超过 $350 \text{ MPa}/(\text{g}\cdot\text{cm}^{-3})$,以密度归一化),高于绝大多数块状镁及镁合金、陶瓷以及其他金属-陶瓷复合材料。

此外,利用冰模板技术,可通过定向生长的冰晶将水基浆料中的 Ti_3AlC_2 薄片挤到相邻冰晶之间,实现粉体的定向自组装,并结合后续烧结与熔体浸渗工艺,进一步构筑多级类贝壳结构^[31](图 7),即微

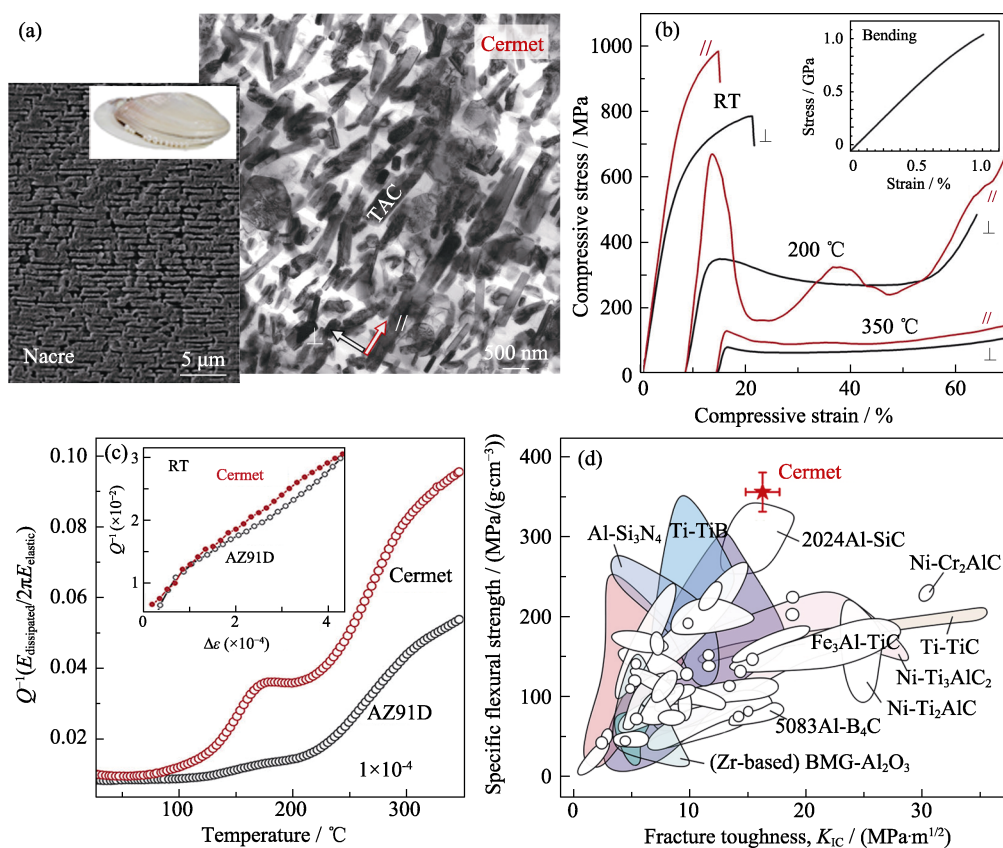


图 6 具有微观类贝壳结构的 Ti_3AlC_2 增强镁基仿生复合材料的微观结构及力学与阻尼性能^[29]

Fig. 6 Structure, mechanical properties, and damping characteristics of nacre-like Mg- Ti_3AlC_2 composites^[29]

(a) Structure of nacre-like composite compared to natural nacre (of *Sinanodonta woodiana* shell, TAC: Ti_3AlC_2); (b) Compressive stress-strain curves of the nacre-like composite at different temperatures when loading parallel and perpendicular to its layered structure with inset showing bending stress-strain curve of the composite at room temperature (RT); (c) Variations in the internal friction (Q^{-1}) with temperature and strain amplitude for the composite compared to the AZ91D alloy matrix; (d) Comparison of the specific flexural strength, i.e., flexural strength normalized by density, and fracture toughness of the composite with other materials. The structure of natural nacre in (a) is adapted with permission from Ref. [30] Copyright 2016, Elsevier. (a-d) Adapted with permission from Ref. [29] Copyright 2023, Elsevier

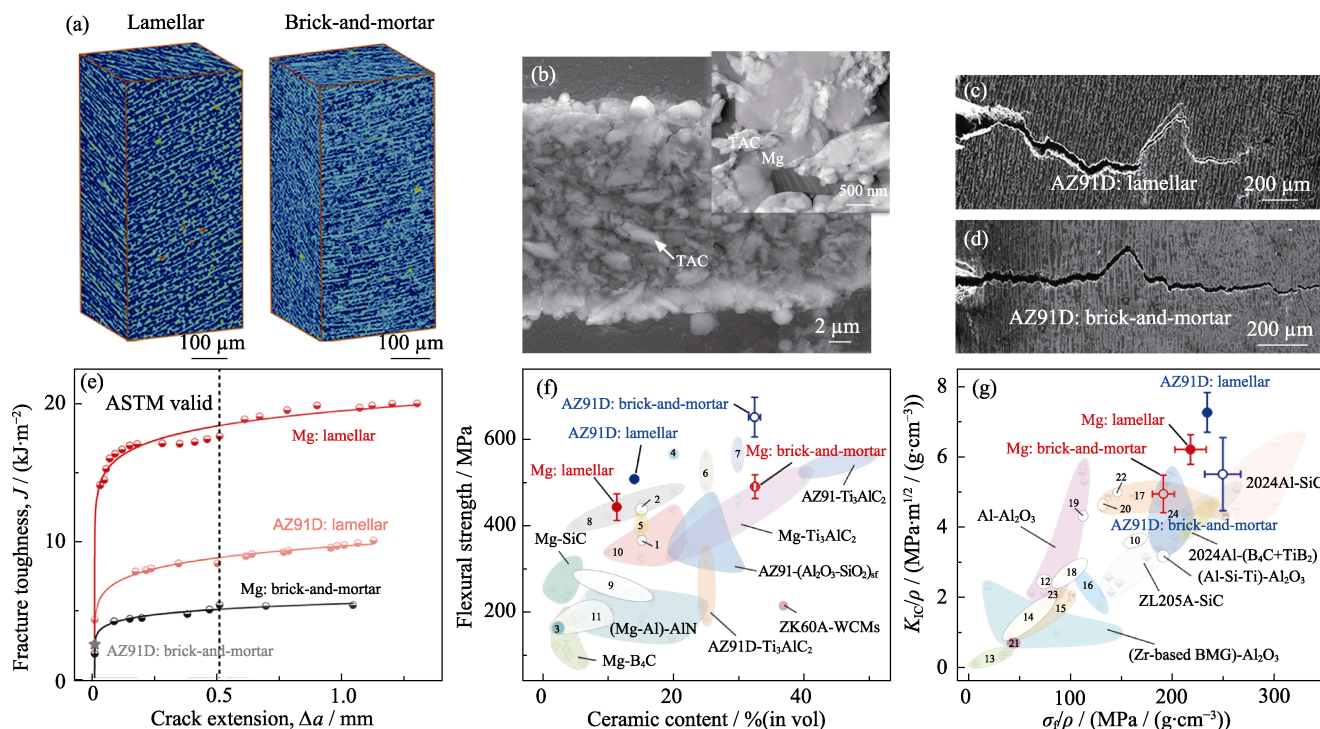


图7 利用冰模板技术结合熔体浸渗工艺制备的多级类贝壳结构 Ti_3AlC_2 增强镁基仿生复合材料的微观结构与力学性能^[31]

Fig. 7 Microstructure and mechanical properties of the hierarchical nacre-like $\text{Mg-Ti}_3\text{AlC}_2$ composites made by ice templating and melt infiltration techniques^[31]

(a) 3D structures of the composites with hierarchical nacre-like lamellar and brick-and-mortar architectures; (b) Ultrafine structure of the composite showing the preferential alignment of Ti_3AlC_2 platelets and full filling interspaces between platelets and metal phase in the ceramic-rich layer; (c, d) Deflected cracking paths in the composites with lamellar (c) and brick-and-mortar (d) architectures; (e) Rising R-curve behavior of the composites demonstrating stable crack propagation; (f) Variation in the flexural strength as a function of ceramic content in Mg and Mg-alloy composites reinforced with various kinds of ceramics; (g) Comparison of fracture toughness and flexural strength normalized by density for various metal-ceramic composites with nacre-like architectures. Adapted with permission from Ref. [31] Copyright 2023, Springer Nature

观金属与陶瓷两相在不同尺度上交替定向排列。该仿生结构能够在多尺度上诱导裂纹偏转和分叉,并促进微裂纹形成与裂纹尖端钝化,从而在保持较高抗弯强度的前提下,进一步提升材料的断裂韧性,获得上升的 R 曲线行为。多级类贝壳结构 Ti_3AlC_2 增强镁基仿生复合材料经密度归一化后的强韧性匹配优于多数以其他陶瓷增强的具有相似仿生结构的金属基复合材料。

4 总结与展望

MAX 相陶瓷性能优异且能与金属形成强界面结合,作为金属基复合材料的增强相具有诸多显著优势。MAX 相陶瓷增强金属基复合材料可利用搅拌铸造、粉末冶金和熔体浸渗等工艺制备,得到的复合材料不仅力学性能比金属基体大幅提升,而且能够获得独特的阻尼、导电、导热、耐磨、抗电弧侵蚀等性能,因此在诸多领域具有突出的应用潜力。

然而,相比于其他金属基复合材料,MAX 相陶瓷增强金属基复合材料相关研究开展时间较短,仍存在诸多局限,亟需进一步解决: 1) 尽管也有关于

Cr_2AlC 、 V_2AlC 等 MAX 相陶瓷增强金属基复合材料的报道^[32-33],目前用作金属基复合材料增强相的 MAX 相陶瓷主要有 Ti-Al-C 和 Ti-Si-C 体系,有待探索更广泛的增强相种类; 2) 现有 MAX 相陶瓷增强金属基复合材料主要表现为均匀结构或简单定向结构,缺乏梯度、非均质等更为精细、复杂的多级设计,特别是构筑的仿生结构以模仿贝壳为主,远未达到天然生物材料的复杂巧妙程度,存在很大的结构优化与性能提升空间; 3) 部分 MAX 相陶瓷与金属基体的组合存在润湿性差或者反应不可控的问题,导致材料界面弱化及成分改变明显,使性能优化效果与预期不符。

后续研究工作可重点关注以下几个方面:

1) 新材料体系开发。MAX 相陶瓷及其衍生物种类丰富且性能独特,可探索不同类型的增强相与金属基体的组合,开发新型高性能金属基复合材料。

2) 微观结构优化设计。MAX 相陶瓷增强金属基复合材料的性能依赖于两相含量、分布、取向、尺度等微观结构特征,优化设计并构筑微观结构,特别是设计更为巧妙复杂的仿生结构,有望进一步提升复合材料的力学性能与功能性质。

3)界面调控与反应控制。通过调整制备工艺参数、添加合金元素等方法可调控 MAX 相陶瓷与金属基体的界面性质,优化材料性能,甚至可以利用 MAX 相作为前驱体,通过原位界面反应获得具有新增强相组分的复合材料。例如,铜与 Ti_3AlC_2 陶瓷可原位反应获得 TiC_x 增强的复合材料。

4)使役性能与损伤机制研究。为推进材料走向实际应用,需针对目标应用需求,系统评价 MAX 相陶瓷增强金属基复合材料在模拟工况条件下的使役性能,揭示其损伤机制,并针对性地优化材料结构与使役性能。

开展上述相关工作有望促进 MAX 相陶瓷增强金属基复合材料的发展与应用。

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