

TC4 合金表面熔覆石墨烯增强 钛基复合涂层的组织及性能

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摘要: **目的** 通过氩弧熔覆技术在 TC4 合金表面制备石墨烯增强钛基复合涂层, 以改善其耐磨性能。**方法** 将钛粉和石墨烯在球磨机中充分混合。将混合后的粉末涂覆于 TC4 合金表面, 采用氩弧熔覆技术将预涂覆粉末熔化, 制备出陶瓷颗粒增强钛基熔覆层。采用 X 射线衍射分析仪分析涂层的物相, 利用光学显微镜、扫描电子显微镜分析熔覆层中颗粒相的组成及分布。采用显微维氏硬度仪和摩擦磨损试验机, 测试熔覆层的显微硬度和磨损性能。**结果** 熔覆层厚度可达 1 mm, 且表面及横截面没有气孔、裂纹等缺陷产生, 物相主要包括 TiC 和 α -Ti。熔覆层中不同区域的组织存在差别, 涂层的中上部组织主要为树枝晶, 底部组织中树枝晶逐渐减少。熔覆层与基体呈冶金结合, 组织致密。增强相 TiC 以颗粒状和花瓣状形式存在。石墨烯增强钛基复合涂层的显微硬度高达 845.4HV。在相同磨损条件下, TC4 合金基体与熔覆层的磨损量分别是 0.153 g 和 0.0123 g, 熔覆层的磨损量明显降低。涂层的磨损机制主要是磨粒磨损。**结论** 与 TC4 合金基体对比, 熔覆层的显微硬度提高约 2.5 倍, 耐磨性提高 12 倍。氩弧熔覆原位自生 TiC 陶瓷颗粒增强钛基熔覆层可显著提高 TC4 合金表面的耐磨性。

关键词: TC4 合金; 氩弧熔覆; 石墨烯; 显微硬度; 耐磨性

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Structure and Properties of Graphene Reinforced Ti-based Composite Coatings on TC4 Alloy

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ABSTRACT: The work aims to study the graphene reinforced titanium-based composite coating is prepared on the surface of TC4 alloy by argon arc cladding technology to improve its wear resistance. In this work, the Graphene power and Ti powder are ball-milled and mixed. The mixed powder was coated on the surface of TC4 alloy, and the ceramic particle-reinforced titanium-based cladding coating is prepared by alloy melting the precoated powder by argon arc cladding technology. The phase of the cladding coating was analyzed by X-ray diffraction analyzer. The composition and distribution of ceramic particles in cladding coating was analyzed by optical microscope and scanning electron microscope and optical microscope. Microhardness and wear properties of the cladding coating was measured by vickers hardness tester and universal friction and wear tester. The thickness of cladding coating of this paper is 1.0 mm, without obvious defects like pores, cracks on the surface and cross section. The phases of the cladding coating mainly include α -Ti and TiC. However, the results show that the microstructure of different areas in the coating is different. The dendrite structure is mainly distributed in the middle and upper part of the coating, while the dendrites in the bottom part decrease gradually. The cladding coating and the substrate are metallurgically bonded, and the structure is dense. The enhanced phase is granular and petal. The microhardness of the coating is as high as 845.4HV, the wear loss of the pure copper matrix and the cladding layer is 0.153 g and 0.0123 g under the same wear conditions, respectively, and the wear amount of the cladding layer is significantly reduced; there is no adhesion mark on the wear of the cladding coating, and the wear mechanism is abrasive wear. Compared with the pure copper matrix, the microhardness of the cladding layer is increased by about 2.5 times, and the wear resistance increased by 12 times. The TiC ceramic particle cladding coating can significantly improve the wear resistance of the TC4 alloy surface.

KEY WORDS: TC4 alloy; argon arc cladding; graphene; microhardness; wear resistance

TC4 合金材料由于具有优良的机械性能而广泛应用于航空航天、医疗和军事工业等关键零部件,如采用钛合金构件代替传统的钢构件,可以极大地降低飞机的质量,从而提升飞机的性能。然而,TC4 合金的应用和发展因其显微硬度低和耐磨性差而受到限制^[1-4]。近几十年来,表面改性技术成为解决这一问题的有效方式,在 TC4 合金表面添加或原位反应生成陶瓷颗粒可以极大地提高耐磨性能,如气相沉积^[5-6]、等离子熔覆^[7]、微弧氧化^[8]、激光熔覆^[9]和氩弧熔覆^[10-12]等。目前已经利用熔覆技术修复燃气涡轮发动机零件等,并获得了性能优异的涂层,提高了零件抗微动磨损的能力。在这些钛合金表面改性技术中,氩弧熔覆技术由于具有操作简单、能量稳定、绿色安全、无氧化等优点^[13-14],已成为钛合金表面高质量熔覆层制备的重要方式之一。

石墨烯(Graphene, Gr)具有优异的性能(如导电性、力学性能等),在材料科学、机械、能源和生物医学等领域有着广泛的应用^[15-16]。石墨烯由于其独特的二维层状结构,比石墨、碳纳米管等传统材料具有更大的比表面积,与基体材料具有更高的接触面积,从而提高结合强度。近年来,石墨烯已应用在金属基复合材料涂层中,使涂层具有优良的导电性和导热性、高强度和韧性、较好的耐磨性和耐腐蚀性等,石墨烯已被认为是金属基复合材料涂层最有前途的增强体^[17-18]。研究表明,石墨烯可以显著改善纯钛复合材料的强度和耐磨性^[19],如 Zhang 等人^[20]利用激光熔覆技术制备了石墨烯增强 Ti6Al4V 复合涂层,石墨烯与钛原位合成羽毛状的 TiC。通过上述分析可知,石墨烯作为一种新的复合材料,与熔覆技术结合起来

制备涂层,具有广阔的应用前景,但目前仅限于激光熔覆技术,使得制备成本提高。为了进一步降低涂层制备的成本及拓宽石墨烯在熔覆技术中的应用,文中采用氩弧熔覆技术制备出石墨烯增强钛基复合涂层,探讨了石墨烯的加入对涂层的组织及力学性能的影响,本研究对石墨烯在工程领域的应用具有一定的指导意义。

1 试验

1.1 混合粉末及涂层制备

选用 TC4 合金为基体材料,其化学成分(以质量分数计)为: 6.05%Al, 3.72%V, 0.10%C, 0.30%Fe, Ti 余量。将其加工成尺寸为 800 mm×20 mm×10 mm 的长方体,采用水磨砂纸打磨表面,利用无水乙醇进行超声波清洗。选用钛粉和石墨烯粉作为熔覆材料,其中钛粉纯度为 99.5%,粒径为 10~20 μm ;石墨烯粉纯度为 99.5%,如图 1 所示。钛粉和石墨烯粉的总质量为 20 g,石墨烯粉的质量占总质量的 0.5%。采用 BS-224S 分析天平称量粉体,在氩气(99.99%Ar)气氛保护作用下,在行星式球磨机中混合 240 min,球磨后的粉体 SEM 照片及 XRD 图谱如图 2 所示。利用胶水作为黏结剂将混合后的粉末调匀,涂覆于经处理后的 TC4 合金表面,厚度控制在 1.0 mm。利用 RX841Y 型电热鼓风干燥箱,将涂覆后的试样在 120 $^{\circ}\text{C}$ 下烘烤 120 min。熔覆设备选用国产 DXT-400 型逆变直流氩弧焊机,具体熔覆工艺参数如表 1 所示。

图 3 为熔覆涂层的 X 射线衍射图谱。分析表明, 熔覆涂层中主要由 TiC 和 α -Ti 组成, 说明石墨烯与钛发生了原位反应。氩弧熔覆涂层的横截面形貌 OM 图像如图 4 所示。熔覆层由表面至界面的深度约为 1 mm, 截面较平滑, 由于具有较好的保护, 使得涂层中未见气孔缺陷。熔覆层的形状取决于涂层对基体的润湿性, 较好的润湿性使表面呈凹陷状^[21]。基体与涂层之间存在明显的白亮带, 说明熔覆涂层和基体形成良好的冶金结合^[11]。同时, 在熔覆层中形成了非常细小的枝晶结构。

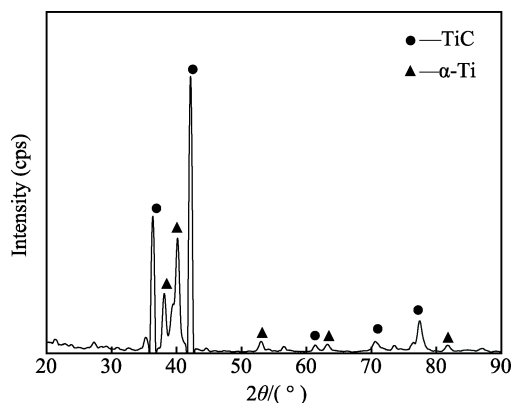


图3 熔覆涂层 X 射线衍射图谱

Fig.3 X-ray diffraction patterns of the cladding coating

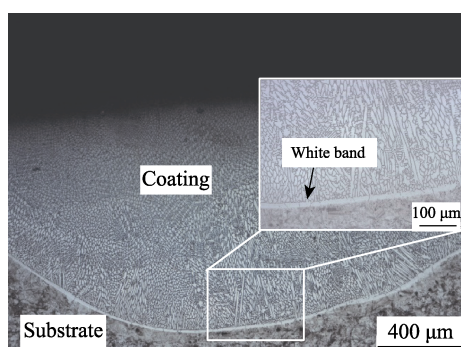


图4 氩弧熔覆涂层横截面的 OM 图像

Fig.4 OM image showing cross-section morphology of argon arc cladding coating

2.2 涂层显微组织

图5为熔覆层横截面不同区域的 SEM 图像, 区域分别为熔覆层的表面、中部(深度 400 μm)和底部(深度 900 μm)。通过对比发现, 熔覆层不同部位的微观结构存在差别。从图 5a 可以看出, 细小的颗粒相组成了枝晶组织, 也有一些颗粒分布在枝晶周围, 原位形成的颗粒尺寸比较小。对于深度为 400 μm 的涂层(图 5b), 熔覆层的显微组织主要以细枝晶、颗粒等形式存在, 且直径减小。从图 5c 可以看出, 在接近基体界面时, 树枝晶逐渐减少, 主要以颗粒状分布在黑色的基体中, 且形成的颗粒相比顶部和中部区域小得多, 分布比较均匀, 在界面处形成了良好的冶金结合。很明显, 熔覆层中枝晶的大小随深度的增加而减小。

图6为熔覆层典型的组织形貌。可以发现, 熔覆层中树枝晶主要由棒状结构及周围的细小颗粒状相构成(图 6a)。如图 6b 所示, 显微组织主要由花瓣状结构构成。对不同部位进行 EDS 分析, 结果见表 2。从表 2 中的元素及其含量, 可以分析出颗粒状相、鱼骨状组织和花瓣状组织的元素主要是 Ti 和 C, 结合 XRD 分析可以确定是 TiC 相。位置 2 主要含有 Ti、Al 和 V, 因此可以推断是基体 α-Ti 相。熔覆层中 TiC 以颗粒相结构为主, 且颗粒相尺寸十分细小, 为 0.3~1.0 μm, 枝晶中的鱼骨状组织尺寸约为 4 μm×0.5 μm。石墨烯

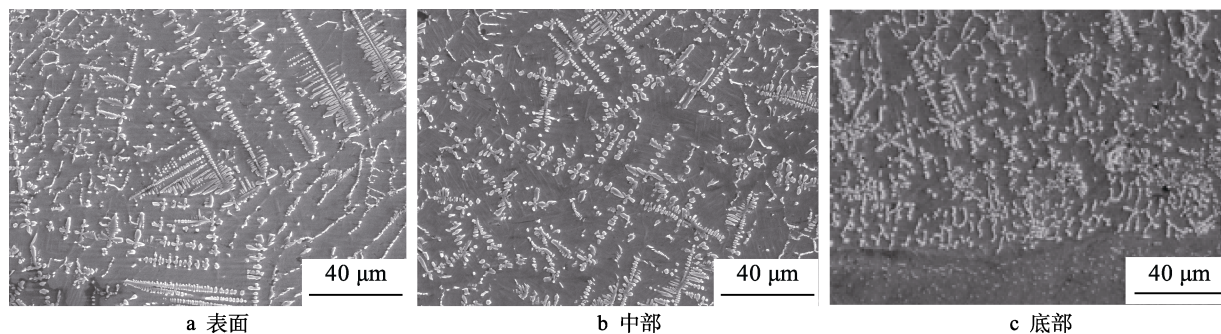


图5 氩弧熔覆涂层横截面不同区域 SEM 形貌

Fig.5 SEM morphology showing cross-section morphology of argon arc cladding coating: a) surface zone; b) central zone; c) bottom zone

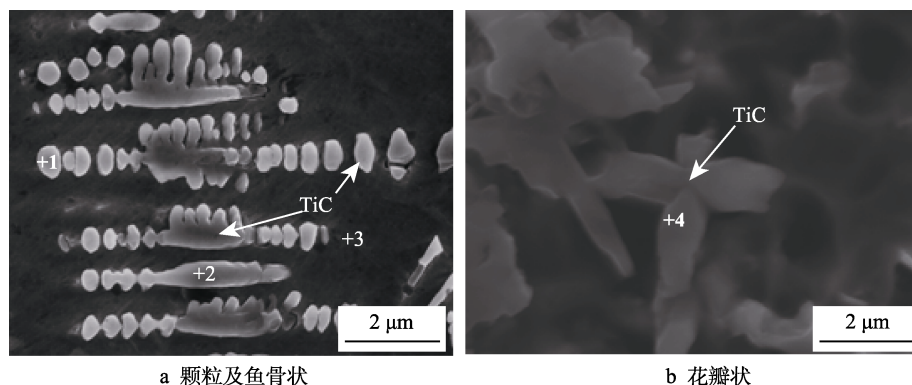


图6 熔覆涂层 SEM 高倍形貌及能谱分析

Fig.6 High power morphology of SEM and energy spectrum showing the microstructure of coatings: a) granular and fishbone; b) flower-like

表 2 图 6 不同位置的能谱分析
Tab.2 Energy dispersive spectrometer of different regions in Fig.6

Element Point	Al		Ti		C		V	
	wt%	at%	wt%	at%	wt%	at%	wt%	at%
1			87.63	55.46	12.37	44.54		
2			88.46	57.82	11.54	42.18		
3	17.56	28.78	78.93	47.94	1.54	18.63	1.97	4.65
4			88.65	58.67	11.35	41.33		

粉和钛粉在氩弧热作用下,原位反应生成了大量均匀分布且细小的 TiC 颗粒,这些尺寸细小的 TiC 颗粒将有助于提高 TC4 合金的力学性能。

2.3 熔覆层显微硬度及耐磨性

图 7 显示了熔覆层的显微硬度。从图 7 中可以看出,熔覆层具有较高的显微硬度。从显微硬度曲线的分布来看,存在 3 个区域:熔覆层区域、热影响区和基体。熔覆层的显微硬度由表面到基体逐渐减少,熔覆层的平均显微硬度可达 845.4HV,且硬度分布比较均匀,曲线波动较小,熔覆层的硬度约为基体硬度的 2.5 倍。这主要是由于在熔覆层中,石墨烯与钛原位反应生成了细小的第二相(TiC),且生成的第二相分布均匀,具有较高的硬度(TiC 硬度可达 3400HV);根据第二相强化理论,这些细小且在涂层中均匀分布的第二相会阻碍位错的运动,从而显著增强位错的稳定性,提高涂层的强度和硬度。

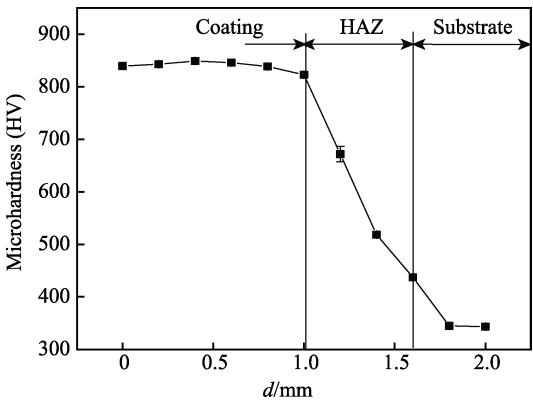


图 7 熔覆涂层截面的硬度分布
Fig.7 Hardness distribution of cladding coating

图 8 为熔覆层与 TC4 合金基体摩擦因数随时间的变化曲线。从图 8 中可以看出,在稳定阶段,TC4 合金基体表面的平均摩擦因数达到 0.69,而石墨烯增强钛基涂层的摩擦因数较基体合金要小得多,平均摩擦因数仅为 0.28,说明熔覆层的抗磨性能优于 TC4 合金。在复合材料/涂层中若生成的增强颗粒分布均匀且与基体有良好的结合时,可有效地降低复合材料/涂层的摩擦因数^[22]。通过氩弧熔覆制备的石墨烯增强钛基熔覆层,原位生成的第二相颗粒与基体具有良好

的结合,且弥散分布,因此熔覆层的摩擦因数大幅度降低。

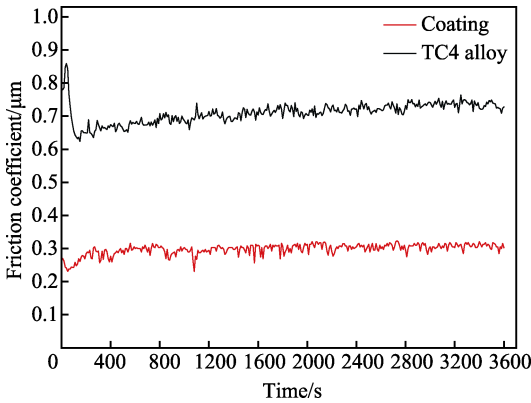


图 8 TC4 合金表面与熔覆层摩擦系数曲线
Fig.8 Friction coefficient of TC4 alloy and cladding coating

图 9 为熔覆层与 TC4 合金基体的磨损量及其耐磨性示意图。在相同磨损条件下,TC4 合金基体的磨损量为 0.153 g,熔覆层的磨损量仅为 0.0123 g,相对耐磨性表明熔覆层的耐磨性较基体提高了 12 倍,熔覆层的耐磨性得到了极大的提高,这与氩弧熔覆条件下形成的组织有关。由于石墨烯的加入,熔覆层组织中获得了大量的第二相,使得涂层具有较高的硬度,在磨损过程中,硬质颗粒相与 α-Ti 基体达到一种强韧性配合,进一步提高涂层的耐磨性。

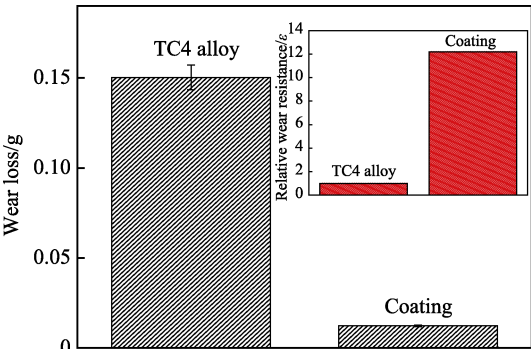


图 9 TC4 基体和熔覆层的磨损量
Fig.9 Wear loss of the TC4 subrbstrate and cladding coating

TC4 合金基体和熔覆层的磨损形貌如图 10 所示。图 10a 所示的 TC4 合金基体表面有相当多的磨屑堆积,在磨损表面同时存在犁槽,通过对区域 A 进行放大(图 10b 所示),发现在表面发生了撕脱现象,表明 TC4 合金基体受到严重的粘着磨损和磨粒磨损。熔覆层磨损表面比较光滑,通过对区域 B 进行放大(图 10d 所示),发现熔覆层磨损表面的犁沟深度较浅,只是轻微划伤,没有发现严重的粘着痕迹,说明熔覆层的磨损程度要低于 TC4 合金基体,熔覆层的磨损机理仅是磨粒磨损。这是由于在熔覆层内均匀分布的第二相可以分担载荷,使得摩擦副表面微凸体难以压入表面,减少粘着现象的发生,提高涂层的耐磨

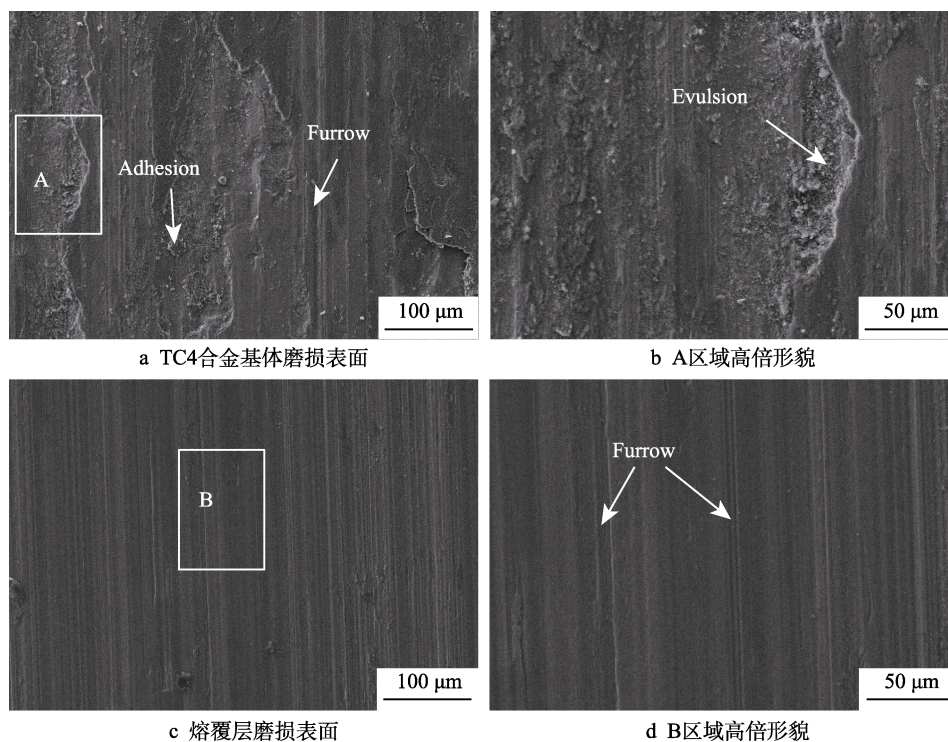


图 10 TC4 合金基体与熔覆层磨损形貌

Fig.10 Wear morphologies of TC4 alloy and cladding coating: a) TC4 alloy substrate; b) enlarged morphology of A; c) cladding coating; d) enlarged morphology of B

性。此外, 尚未发现由石墨烯在摩擦过程中产生的润滑膜, 这说明石墨烯在涂层中的添加量较低, 无法形成大面积的润滑膜。

3 结论

1) 采用氩弧熔覆技术, 以石墨烯粉和 Ti 粉为原料, 在氩气气氛下制备了石墨烯增强钛基熔覆层, 熔覆层与基体呈现冶金结合, 表面无飞溅, 内部无缺陷。

2) 熔覆层的物相主要由 TiC 和 α -Ti 组成。熔覆层中的组织主要以颗粒状和棒状构成的树枝晶以及花瓣状组成, 石墨烯的加入使得生成的第二相颗粒尺寸更为细小, 且弥散分布于熔覆层中。熔覆层不同区域的组织存在差别, 沿表面至涂层界面树枝晶逐渐减少, 颗粒相增多。

3) 石墨烯增强钛基熔覆层具有优异的摩擦磨损性能, 熔覆层的平均显微硬度可达 854.4HV, 摩擦因数 (~ 0.28) 低且波动小, 在高载荷长时间摩擦磨损作用下仍可以表现出较好的耐磨性能。

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