专家论坛

镀铬/高能束复合表面处理研究进展

李怀学[1,2]，张 坤[1]，陈光南[1]，罗耕星[1]，叶智军[1]
(1. 中国科学院力学研究所表面改性实验室，北京 100080；2. 中国科学院研究生院，北京 100049)

[摘 要] 根据高能束对镀铬涂层及其界面强化机制的不同，镀铬/高能束表面复合技术可分为两类：高能束强化镀铬涂层复合技术和高能束预处理基体/镀铬复合技术。前者典型代表有激光表面强化或等离子体氮化/镀铬涂层；后者主要代表是激光预淬火基体/镀铬复合表面处理。综合阐述了上述3种典型的复合处理技术的原理、目的及实际综合效果；通过试验初步探讨了激光预淬火基体/镀铬复合技术延长镀铬身管寿命的主要机理。

[关键词] 高能束复合处理；镀铬；界面强化
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0 前 言

利用激光束、电子束、离子束等高能束对材料表面进行改性或合金化的技术是近30年来迅速发展起来的材料表面处理新技术，其原理就是利用激光束、电子束和等离子体束作为热源，通过改变材料表面的局部成分或结构，实现对材料的局部表面改性[1,2]。高能束表面处理技术主要包括两个方面[3,4]：一是利用激光器和电子发生器可获得极高的加热和冷却速度，可制成微晶、非晶及其他一些奇特的亚稳态合金，从而赋予材料表面以特殊的性能；二是利用离子注入或等离子体氮碳化技术将异类原子直接引入表面层中进行表面合金化，以改善材料表面的耐磨性及耐蚀性。另外，高能束表面处理技术具有能量密度高、热影响区域小、加工位置可控性好，易于实现自动化、智能化等优点。

镀铬层因具有耐磨性好、硬度高、摩擦系数低、熔点高等优异的性能，被广泛应用于机械零部件以及武器身管内膛等表面，钢基体镀铬在高温、强腐蚀、高磨损等极端工况下应用非常普遍。然而，由于镀铬层含有许多显微裂纹而且质地硬且脆，其在高温、强腐蚀、高磨损等极端的工况下开裂和局部剥落往往导致高温强腐蚀的气体烧蚀基体[5~7]。随着各种机械及武器等零部件对表面性能和可靠性的要求越来越高，镀铬工件的工况更加苛刻，镀铬层的过早开裂和局部剥落失效问题日益突出，传统的单一表面镀铬处理技术已无法满足实际需求。

众所周知，基体与镀铬界面处的组织结构及性能是影响镀铬层开裂和局部剥落的关键因素之一。为缓解和抑制镀铬层过早的开裂和局部剥落，国内外许多学者提出将传统的镀铬技术与先进的高能束表面处理技术相结合即镀铬/高能束表面复合处理技术，通过它们之间的协同作用，发挥各自优势，从而改善镀铬层与基体界面处的组织结构以及镀铬层的组织结构，增强镀铬层的抗腐蚀性和抗剥落能力[8~23]。

为促进高能束表面处理技术与镀铬技术复合的应用和研究，本工作首先根据高能束/镀铬复合技术的研究现状对其加以分类，随后对典型的复合处理工艺进行了阐述，并对激光预处理基体镀铬复合工艺进行试验分析，初步探讨了镀铬层寿命延长的主要机理。

1 镀铬高能束表面复合处理技术分类

目前，高能束表面处理技术对镀铬层的强化处理主要有激光束/镀铬的复合处理、等离子体氮(碳)化/

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镀铬的复合处理以及离子注入/镀铬的复合处理等[8~23]。利用高能束强化镀铬层的界面可概括为两类:一是利用高能束对镀铬层及其界面同时强化处理;二是利用高能束预处理基体,通过改变基体表层的组织结构及性能实现调整镀铬层界面处组织结构及性能。根据高能束对镀铬层及其界面强化机制的不同,镀铬/高能束复合处理技术可分为两类:高能束强化镀铬涂层复合技术和高能束预处理基体/镀铬复合技术。前者主要设计思路:在镀铬涂层制备后利用高能束表面处理技术对镀铬层及其界面同时强化,以减少镀铬层中的微裂纹,通过界面冶金或界面扩散实现镀铬层与基体间的冶金结合。激光表面强化镀铬层和等离子体氮(碳)化镀铬层是其典型的复合技术[8~18]。后者从镀铬层制备工艺的角度出发,基于镀铬层/基体间界面的组织结构及性能主要由基体的表面状态和电沉积方法及工艺参数所决定,在优化的电镀工艺条件下,通过高能束表面处理改善基体表层的组织结构及性能,从而改善镀铬层/基体界面的组织结构,提高镀铬层的抗开裂及局部剥落等能力。激光预处理基体/镀铬表面复合处理是其典型的复合技术[9~23]。

2 激光表面强化镀铬层复合处理

20世纪70年代末,美国的R. S. Montgomery[8]首先对激光束表面处理镀铬层进行了研究,其目的是通过实现镀铬与基体间冶金结合以提高镀铬层结合力,其结果显示在实现镀铬层与基体间冶金结合的条件下,由于镀铬层和钢基体的熔点差别较大,在激光束高温作用下,镀铬层发生再结晶,而界面处的钢基体重熔凝固。由于界面处的钢基体重熔再结晶过程中伴随着铬原子的扩散,在界面处形成一层脆硬而耐腐蚀的Fe-Cr合金扩散层,扩散层下的钢基体形成高硬度的未回火马氏体。虽然镀铬层的再结晶使原镀铬层中的许多微裂纹消失,但是镀铬层发生很大的软化,而且更易于破碎。耐磨性试验证实激光处理过的镀铬层耐磨性比原始镀铬层更差。20世纪80~90年代,安世民等[9~14]对激光处理镀铬的组织与性能变化进行了研究,结果显示激光处理过的镀铬层中的组织由柱状晶向等轴晶转变,缺陷较少,致密度提高,硬度下降,在适当的工艺条件下,在镀铬层未熔融而紧靠铬层的钢基体熔化状态下,在固-液两相界面附件形成Fe-Cr合金的扩散层,其耐腐蚀性极高,同时基体被部分淬火,硬度升高;这些试验结果与R. S. Montgomery的研究结果相吻合。总之,先镀铬后激光束表面复合处理使镀铬层的硬度大幅度降低,即发生软化,而且更易于破碎,耐磨性更差,因此该复合工艺未取得良好的综合效果,其实际应用未见报道。

3 等离子体氮(碳)化镀铬层复合处理

由于镀铬层通常含有许多微裂纹,腐蚀性介质往往通过这些微裂纹腐蚀铬层下的基体,因此这些微裂纹降低了镀铬层的耐腐蚀性;另外镀铬层的硬度随着温度的上升而下降,在高温条件下导致镀铬层的耐磨性降低。为了克服这些缺点,不少学者利用离子注入或等离子体氮(碳)化处理镀铬层,这样镀铬层的表面形成一层氮化铬[15~18]。等离子体氮(碳)化处理镀铬层不仅可以消除镀铬层表层的微裂纹,而且在镀铬表层形成一层高硬度的CrN,提高了镀铬层的耐磨性和耐蚀性。同时由于长时间的高温作用,镀铬层与基体界面形成一扩散层,提高了镀铬层的界面结合强度[18]。然而,等离子体氮化效率较低,成本高,高温条件对基体性能影响较大,特别是当零件形状复杂时,等离子体氮(碳)化难以实现。

4 激光预处理基体/镀铬复合处理技术

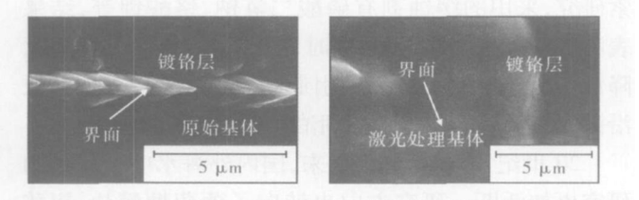
目前其典型应用是将激光处理的材料表面直接作为工作表面,获得良好的应用效果如激光毛化[24~25],激光表面淬火等。然而,激光预淬火基体/镀铬复合技术利用激光对基体材料的表面改性优势,通过基体/镀铬层间界面及镀铬层间接发挥作用。由于镀铬层的制备温度较低,一般在40~80 ℃范围内,该温度范围不会对激光预处理材料的表面结构及性能产生影响,同时除保持镀铬层的各种优异性能外,激光预处理基体的表层因具有较高的硬度,缓解了镀铬层/基体间的硬度梯度,提高了镀铬层的承载能力,从而减缓了镀铬层过早的开裂或失效。因此,激光预淬火基体后镀铬层的复合处理技术可把激光表面强化和镀铬层的优势结合起来。

激光预淬火基体后镀铬层的复合处理技术先后由中国科学院力学研究所和德国莱茵公司于1999年先后提出[19~20],而且成功地解决了我国某型号武器镀铬身管的寿命长期不达标的关键技术难题。另外,利用该复合技术制备的镀铬身管的寿命提高了50%以上,

初步试验结果表明,在高温、强腐蚀及复杂的机械载荷作用下,该复合工艺制备的镀铬层的抗剥落能力大幅度提高[21]。

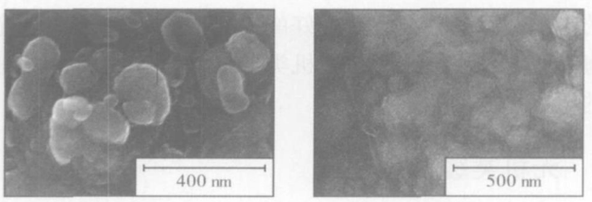
该复合处理工艺延长镀铬层的寿命初步机理分析:一般认为基体的淬火有利于提高基体对镀层的支撑作用,提高镀铬层的抗疲劳和抗剥落的能力,并未考虑基体表面预处理对镀层及其界面组织结构的影响[26]。在低温工况下,可以认为基体激光处理提高了基体对镀层的承载能力和抗疲劳能力,从而提高镀铬层的界面抗剥落能力。然而,在高温、强腐蚀及复杂载荷作用等极端工况下,高温作用很快导致基体软化,在这种条件下身管寿命仍然能够延长 50%以上。因此利用基体激光处理延长基体对镀层的支撑作用解释镀铬身管寿命提高证据并不充分,必有其他内在的本质原因,其中基体表面激光预处理对镀层及其界面的组织结构的影响是一个关键因素。

根据电镀理论[27],镀铬层与基体界面的组织微结构是影响镀铬层使用寿命的关键因素之一。利用离子刻蚀法和溶解基体法研究了镀铬层与基体界面微结构特征见图 1、图 2。从图 1中可以看出原始基体/镀铬层间界面存在一层大约 2 μm厚的“夹杂层”,而激光相变淬火基体/镀铬层界面处不存在上述所谓的“夹杂层”,且其界面非常平整。文献[19]也指出了上述“夹杂层”,但并未阐述其形成的原因。图 2显示了激光淬火基体对镀铬层组织结构及形貌的影响,基体激光淬火区上的镀铬层组织/晶粒细化,而且表面非常光滑平整。根据图 2可以清晰反映上述“夹杂层”,因为激光



(a) 原始基体与镀铬层界面　(b) 激光相变硬化基体与镀铬层界面

图 1　基体与镀铬层间界面微结构离子刻蚀 SEM 形貌图



(a) 无激光预处理　(b) 经激光预处理

图 2　去掉基体后靠近基体侧镀铬层表面 SEM 形貌图

淬火基体,不仅改变基体表层的微观组织结构,而且对初始镀铬层的组织结构及形貌产生很大的影响。这些基体/镀铬层界面微结构的差异可能就是激光预处理基体/镀铬复合工艺提高的镀铬层寿命主要原因。关于激光淬火基体对电沉积镀铬层及其界面的组织结构及力学性能影响机理今后将进一步研究。揭示基体激光表面改性对电沉积镀铬层的组织结构及力学性能影响机理不仅为解释激光预处理基体/镀铬复合技术提高镀铬层的抗剥落能力提供理论基础,将该界面强化机理应用到其他电沉积涂层提供理论基础,而且这可望为提高电沉积涂层界面的抗破坏能力提供一种新的途径和思路。

5 总　结

激光预处理基体/镀铬表面复合处理技术是一种新型的表面复合处理技术,其良好的综合效果已被实际应用所证实,但激光淬火基体与镀铬层界面微观结构及其力学性能等基础理论需要进一步解释清楚,为该复合技术及相类似的技术推广提供坚实的理论基础;高能束预处理基体/其他涂层技术复合可能产生良好的综合效果。

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由于带钢在运输和存放过程中可能发生氧化,在表面生成氧化铁皮,而预清洗不够彻底,使得基板表面残留有氧化铁。如果残留氧化铁皮面积较小,则 Al-Zn 液的表面张力足以令镀层连续覆盖,表观上不会出现露铁和漏镀现象,但镀层与基板间的结合力仍然会受到影响,镀层易脱落;若氧化铁皮面积较大,则会使镀层连续性受到破坏,产生露铁,从而导致镀层出现悬崖和缺失,形成凹陷,涂漆后凹陷中的气体烘干时受热膨胀,冲破面漆而成为漏涂缺陷,在生产中应注意避免。

3 结 论

综合几种缺陷的表面形貌和截面特征,可以得出以下结论:

(1)凹坑缺陷处面漆破损,底漆正常涂覆;

(2)除去涂层,镀铝锌钢板表面露铁,特征为离散分布;

(3)基板表面附着氧化铁皮,预清洗未能完全除净,在表面形成细微裂纹;

(4)基板表面存在非金属夹杂,影响了镀铝锌层的连续性和厚度。

针对彩涂镀铝锌板所出现的缺陷形貌及产生原因,在今后的生产中,需要在以下几个方面进行改进:

(1)加快锌花均匀性研究工作,减小锌花尺寸,降低三叉晶界处材质的孔洞与疏松;

(2)注意入口段清洗效果和炉内露点稳定性控制[3],防止由于带钢表面氧化铁皮的存在和炉内弱氧化产生露铁缺陷;

(3)尽量通过提高涂料覆盖能力来消除凹坑缺陷。

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MA Sheng-ge, KANG Guang-yu (National R&D center for Surface Engineering of China, Shenzhen 518029, China). *Cailiao Baohu* 2007, 40(04), 35～38(Ch). Multi-layer Ti-N-C black hard films were deposited making use of cylindrical cathode arc ion plating and medium frequency twin target unbalanced magnetron sputtering. The morphologies and mechanical properties of the resulting hard films were investigated using a scanning electron microscope, spectrophotometer, and hardness meter. It was found that both the techniques could be well used to deposit deep black Ti-N-C hard films. The deposition efficiency of the cylindrical cathode arc ion plating was higher than that of the medium frequency twin target unbalanced magnetron sputtering, and the Ti-N-C hard film prepared using the former technique had better mechanical properties. At the same time, the black hard film deposited by medium frequency twin target unbalanced magnetron sputtering was smoother and more black as compared to the film deposited by cylindrical cathode arc ion plating.

Key words: black hard film; Ti-N-C; cylindrical cathode arc ion plating; medium frequency sputtering; unbalanced magnetron sputtering

Progress in Study of Duplex Surface Treatment of Chromium Electroplating and High Energy Beam

LI Huai-xue[1,2], ZHANG Kun[1], CHEN Guang-nan[1], LUO Geng-xing[1], YE Zhi-jun[1] (1. Institute of Mechanics, Chinese Academy of Sciences, Beijing 100080, China; 2. Graduate School, Chinese Academy of Sciences, Beijing 100039, China). *Cailiao Baohu* 2007, 40(04), 39～41(Ch). A review was given on the principles, purposes and practical effectiveness of chromium electroplating/ laser surface strengthening, plasma nitriding/chromium electroplating, and substrate laser pre-quenching/ chromium electroplating as the duplex surface treatment techniques. The mechanisms for the substrate laser pre-quenching plus chromium electroplating as a duplex surface treatment process to increase the service life of the chromium-plated gun tubes were discussed based on experimental exploration. It was pointed out that, according to the different interfacial strengthening mechanisms of high energy beam irradiation and chromium electroplating, the duplex surface treatment technique of chromium electroplating and high energy beam irradiation can be divided into two categories: high energy beam hardening of electroplated chromium coatings and the hybrid process of substrate high-energy-beam pre-treating plus chromium post-electroplating. The typical examples of the former include laser surface hardening (plasma nitriding) of electrodeposited chromium coatings, while the representative of the latter is the duplex treatment of substrate laser pre-quenching plus chromium post-electroplating.

Key words: high energy beam duplex surface treatment; chromium electroplating; interfacial strengthening

Progress in Study of Green Corrosion Inhibitor Used in Seawater

LIU Xin-hua[1], YU Jing-min[2], LIANG Ying-hua[1] (1. College of Chemical Engineering and Biotechnology, Hebei University of Technology, Tangshan 063000, China; 2. Teaching Group of Chemistry, Vocational Institute of Foreign Language, Qinhuangdao 066000, China). *Cailiao Baohu* 2007, 40(04), 42～46(Ch). The research progress in the study of green corrosion inhibitors for metals in seawater was reviewed. It was pointed out in future study on new environmentally friendly corrosion inhibitors focuses should be paid to the extraction, separation, and processing of effective ingredients from natural plants, the synthesis of low-toxic or non-toxic multifunctional macromolecules and inorganic type corrosion inhibitors and the matching among various corrosion inhibitors. Moreover, the optimal utilization of natural resources should also be emphasized.

Key words: green corrosion inhibitor; seawater; inorganic type; multifunctiond type; extraction from natural plants type

Characterization and Analysis of the Holiday Defect on the Surface of Color-Coated Galvanized Steel Sheet

JU Chen[1], CHU Shuang-jie[2], SUN Bao-de[1], WANG Jun[1], ZHANG Quan-cheng[2] (1. State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200030, China; 2. Baoshan Iron & Steel Co., Ltd, Shanghai 200941, China). *Cailiao Baohu* 2007, 40(04), 47～49(Ch). Surface holiday is one of the typical defects of color-coated galvanized steel sheet. Thus the causes leading to the holiday defects in the surface varnish of the color-coated galvanized steel sheet with well painted bottom varnish were investigated by means of sectional metallographic analysis, energy dispersive spectroscopic analysis, scanning electron microscopic observation, and electron probe microanalysis, in connection with consideration of the actual production conditions. It was primarily found that the inadequate pre-cleaning and Fe_3O_4 residue could mainly account for the surface holiday defects of the surface varnish of the steel sheet. In particular, Fe_3O_4 caused discontinuousness of the varnish, leaving absence of varnish at some locations, while the residue gas entrapped in the concave zones would expand and break through the surface of the vanish in post-painting heating.

Key words: hot-dip galvanizing; color-coated steel sheet; holiday defect; ribbon steel; aluminizing and galvanizing coating

Adsorption of Ni^{2+} in Electroplating Wastewater by Polyquaternary Ammonium Salt Polyacrylamide

LUO Dao-cheng, LIU Jun-feng (College of Chemistry and Chemical Engineering, Hunan University of Science and Technology, Xiangtan 411201, China). *Cailiao Baohu* 2007, 40(04), 50～52(Ch). The adsorption of heavy metallic ion Ni^{2+} in Ni^{2+}-containing electroplating wastewater by polyquaternary ammonium salt polyacrylamide (PQAAM) was studied under static condition. Thus the effects of the PQAAM dosage, the pH value of the electroplating wastewater, and the adsorption time and temperature on the removal efficiency of the Ni^{2+} ions were investigated. The results showed that the Ni^{2+} in the electroplating wastewater containing 0～100 mg/L Ni^{2+} could be removed at a rate over 98%, when the wastewater with a mass ratio of Ni^{2+}/PQAAM = 1∶30 was treated at a wastewater pH value of 6.0～8.0 and temperature of 20 ℃ for an adsorption duration of 80 min. After being treated with the PQAAM, the content of Ni^{2+} in the electroplating wastewater was lower than the relevant national discharge standard.

Key words: polyquaternary ammonium salt polyacrylamide; wastewater treatment; adsorption; Ni^{2+}; electroplating wastewater

Application and Evaluation of Eight Kinds of Inside Anticorrosion Technique for Pipelines Used in Shengli Oilfield

LONG Yuan-yuan, SHI Ren-wei, LIU Yan-guo, SUI Guo-yong, LV De-dong (Research Institute for Corrosion and Protection, Technology Detection Center of Shengli Oil Field, Dongying 257000, China). *Cailiao Baohu* 2007, 40(04), 53～54(Ch). Eight kinds of inside anticorrosion technique for coated H87, HT515 and EP-67 tubes, glass fiber reinforced plastic tube, steel-frame composite tube, nylon-based composite tube, tube with polyethylene inner liner, and tube with glass enamel inner liner were applied and evaluated in field pilot test with respect to criterion 1 a, making use of macro-observation, alternating current impedance measurement, leaking test with electric spark, adhesion test, anti-impact test, test of resistance to chemical attack, and observation of micro-morphology. It was found that the coated EP-67 tube, the steel-frame composite tube and the nylon-based composite tube had better comprehensive properties among the eight kinds of tubes tested. This could make it feasible for them to be well used as the internal anticorrosion pipelines for the collection and transfer of acidic gases and fluids in oilfields. However, the tube made of the glass fiber reinforced plastic was unsuitable under a relatively high pressure. Moreover, the tube with polyethylene inner liner had good anticorrosion behavior, but it had poor adhesion between the inner liner and the tube body at a temperature of 50 ℃ and above. Besides, the tube with glass enamel inner liner and the coated HT515 and H87 tubes had poor anticorrosion ability and comprehensive properties as well, thus they were not suitable to be used in oilfields.

Key words: pipelines for collection and transfer of petroleum; inside anticorrosion technique for pipelines; field pilot application; dissection test; evaluation of performance

Improvement of Galvanizing Process for Welding Net of Low Carbon Steel

MIAO Li-xian[1], WANG Zheng-yin[2], HOU Zhi-jiao[1], CUI Huan-ming[1], MIAO Ying[3] (1. Tianjin Gongda Galvanizing Equipment Co., Ltd, Tianjin 300132, China; 2. Zhejiang Haili Group Co., Ltd, Quzhou 324000, China; 3. Henan College of Science and Technology, Xinxiang 453003, China). *Cailiao Baohu* 2007, 40(04), 55～57(Ch). The problems emerging in the hot-galvanizing process of low carbon steel welding net were analyzed based on the principles for galvanizing of low carbon steel. Thus the shortage of the process, the irrationality of the ingredients of the galvanizing liquid, and the quality