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金属腐蚀监测的光波导传感方法研究

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摘要: 本文提出一种用于金属腐蚀监测的光波导传感方案,用金属膜层局部取代光波导的介质包层,构成腐蚀敏感膜,从而获取金属腐蚀信息.依据波导理论选用721比色皿上的石英玻璃作为光波导材料,利用电化学方法在波导材料内表面上形成Fe-C合金腐蚀敏感膜,并用XRD、EDX等对膜层结构进行分析.在电化学腐蚀膜的同时,用光学方法记录光波导输出光功率的变化,实验结果表明电化学方法与光学方法获取的腐蚀信息同步,证实了所提传感方案可行.

关键词: 光波导;腐蚀监测;Fe-C合金

中图分类号: TG174.3

文献标识码: A

金属腐蚀造成的经济损失约占国民生产总值的4%,由它所引起的结构破坏方面的例子在实际工程中屡见不鲜^[1].应用在线监测对于跟踪金属腐蚀状态及其变化规律,确保化工设备及各种金属构件安全运行,确有十分重要的意义.由于金属构件所处环境复杂(如难以直接接触、取样困难、周围有强电磁场干扰等),应用传统的化学和电化学腐蚀监测方法十分困难^[2],而基于光学方法的光纤腐蚀传感器则具有径细、质轻、抗强电磁干扰、耐高温、集信息传输与传感于一体、易于集成以到达难以接近的待测区域等一系列优点,有望克服传统腐蚀监测方法的缺陷. K. D. Bennett 等人^[3]提出了一种基于“腐蚀保险丝”的光纤传感器,用于大型钢结构腐蚀监测. P. L. Fuhr 等人^[4]则根据铁锈颜色变化建立反射式光纤腐蚀传感方案,取得了一些初步研究结果.但是, K. D. Bennett 和 P. L. Fuhr 的方法只能用于判断腐蚀是否发生,不能在线监测腐蚀状态.本文提出一种采用腐蚀敏感膜传递腐蚀信息的光波导传感法,其原理是利用光纤纤芯上 Fe-C 合金敏感膜层受到腐蚀时能对光纤纤芯内传导的光信号产生影响,通过实时测量输出光信号的改变即可获取钢铁材料腐蚀信息^[5].由于所用光纤纤芯直径很小,仅在100 μm 左右,在纤芯上直接研究该腐蚀膜层性质较困难,根据光纤圆柱光波导与平板光波导传光性能相近的特点,先在平面石英玻璃上进行实验.其研究结果将对深入研究光纤腐传感器(FOCS)具有指导作用.

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1 实验

将 721 比色皿(30mm ×12mm ×40mm)的石英玻璃侧壁两端面抛光至光学镜面.石英玻璃内壁经丙酮除油、CrO₃ 酸洗后,用 SnF₂ 敏化、化学镀银、电镀 Fe-C 合金处理^[5],其敏化液组成及操作条件按表 1 条件进行.所用试剂为 A. R 级,溶液用去离子水配制.同时,将 Fe-C 合金沉积至 Ag 或 SiO₂(湿法生长在单晶硅片上)表面,按 GB223. 69-89 中燃烧气体容量法测定膜层中 C 含量,并用日本岛津 XD-3A 型 X-射线衍射仪和 KYKY-X 射线能谱分析系统测定 Fe-C 合金层的结构.

表 1 石英玻璃内壁处理的敏化液组成及操作条件

Tab. 1 Composition of sensitization solution and operation conditions

SnF ₂	HF(40%)	tin particles	temperature	time
2.0 g/L	10~15 ml/L	A few pieces	25 ± 1℃	5~6 min

采用图 1 所示装置研究 Fe-C 合金膜层的腐蚀传感性能,腐蚀试验条件如表 2 所示;在对 Fe-C 合金膜层进行电化学腐蚀的同时,从石英端面射入激光,并用光功率计及时间-函数记录仪记录光学信号的变化。

表 2 Fe-C 合金膜层的电化学腐蚀试验条件

Tab. 2 Conditions of electrochemical corrosion test for the Fe-C alloy layer

corrosion medium	potential range	scanning rate	CE	RE
5% NaCl	-0.69~0.71V	0.5mv/s	Pt	SCE
simulated concrete pore solution ^[6]	-0.30~0.71V	0.5mv/s	Pt	SCE

2 结果及讨论

2.1 Fe-C 合金腐蚀敏感膜层结构分析

用燃烧气体容量法对敏感膜层中的碳含量进行分析的结果表明:在含少量柠檬酸和抗坏血酸的硫酸亚铁溶液中电镀能够获得碳含量在 1%(wt)左右的 Fe-C 合金镀层.图 2 给出了银电极上 Fe-C 合金电镀层的 X-射线衍射谱图,图中衍射峰归属 -Fe^[7],但(110)、(200)峰的半高宽(FWHW)显著增加,且(211)峰观察到有“肩”的出现,其晶胞参数为 a = 0.2862nm, c = 0.2895nm, c/a 轴比为 1.011. Fe 的原有体心立方晶格因 C 的填充而出现点阵歪扭,并转变为具有体心四方结构的单晶马氏体,证明采用该工艺获得的镀层为 Fe-C 合金层.

图 3 为 SiO₂ 表面上 Fe-C 合金膜层的 X 射线能谱(EDX)分析结果,图中有 Si、Fe 和 Ag 的特征 X 射线峰存在.随着电镀时间延长,镀层厚度增加,Si、Ag 特征峰逐渐减弱,而 Fe 特征峰

逐渐增加,表明 Fe-C 合金层的厚度在逐渐增加.

2.2 膜层的腐蚀传感

特性研究

按图 1 装置对 Fe-C 合金膜层在电化学腐蚀过程中极化电流和输出光功率的变化进行测定,结果如图 4、图 5 所示.从图中可以看出:不论是 5%NaCl 溶液还是模拟混凝土孔溶液,随着电化学腐蚀过程的进行,石英玻璃输出光功率总体呈下降趋势,且图中光功率迅速下降处的电位正好与扫描峰电位相一致,表明测量腐蚀过程中的光功率变化能够获取可靠的腐蚀信息.根据腐蚀理论,动电位扫描过程中峰电位处腐蚀反应最剧烈,此时表面膜将受到破坏,腐蚀介质很容易渗透进疏松多孔的表面膜层,并使膜层变得粗糙,对光吸收增加,输出光功率降低.

3 结 论

初步研究表明,石英玻璃上的腐蚀敏感膜能够传递金属腐蚀信息,改变光波导上的金属敏感膜材料,可适应对不同金属材料腐蚀的监测.实验证明电化学镀膜方法能够满足光波导上金属包层制备要求.若将该镀膜工艺移植到光纤上,则可形成一种新型光纤腐蚀传感技术,满足金属构件腐蚀监测的需要.

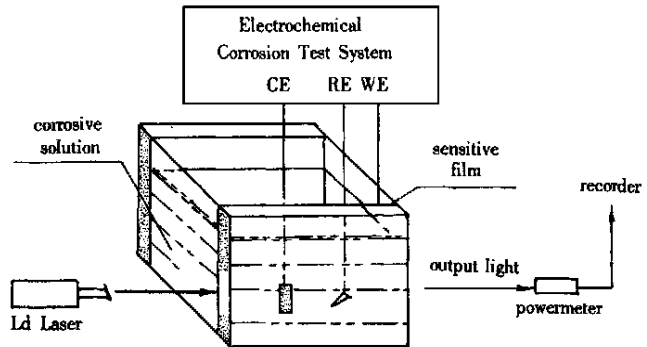


图 1 光学方法与电化学方法研究 Fe-C 合金膜层的腐蚀传感性能实验装置

Fig. 1 The Scheme of electrochemical corrosion and corrosion sensing test for the Fe-C alloy layer

Output of Ld laser is 0.4mW, angle of incidence is 30°

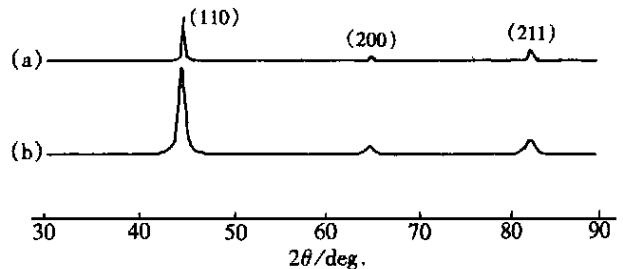


图 2 Ag 电极上 Fe-C 合金 XRD 谱图

Fig. 2 X-ray diffraction patterns of Fe-C alloy layer deposited on silver electrode

(a) diffraction peak of α -Fe; (b) diffraction peak of Fe-C alloy layer

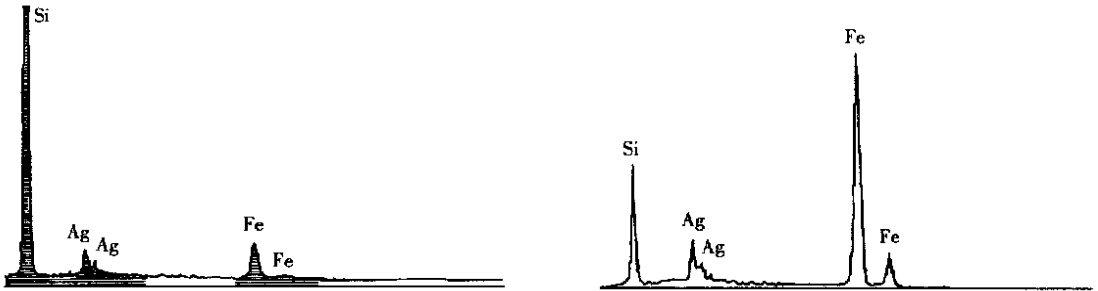


图 3 不同电沉积时间的 Fe-C 合金膜层的 EDX 谱图

Fig. 3 EDX patterns of Fe-C alloy layer deposited on SiO₂ in 5 min(left) and 20 min(right)

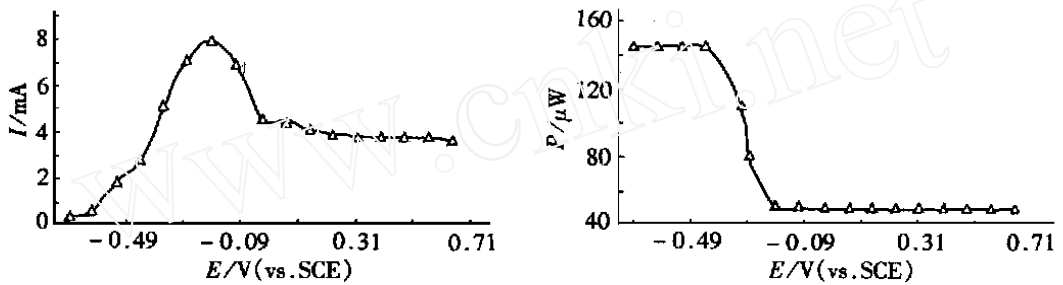


图 4 Fe-C 合金膜层在 5% NaCl 溶液中的腐蚀传感性能

Fig. 4 Polarization current and light power with respect to scanning potential for the Fe-C alloy layer in 5% NaCl solution

(Left): electrochemical signal; (right): optic signal

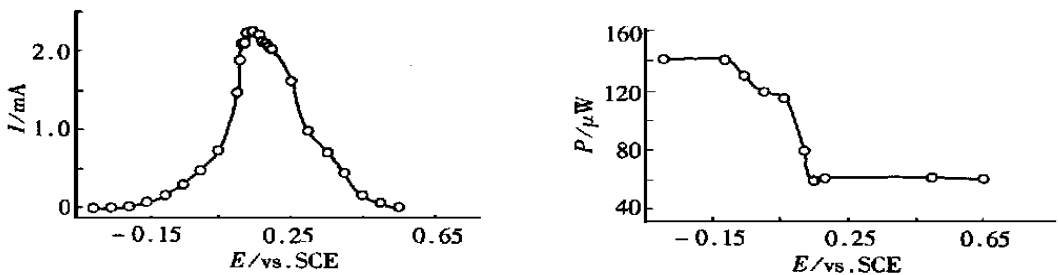


图 5 Fe-C 合金膜层在模拟混凝土孔溶液中的腐蚀传感性能

Fig. 5 Polarization current and light power with respect to scanning potential for the Fe-C alloy layer in simulated concrete pore solution

(Left): electrochemical signal; (right): optic signal

A Study of Monitoring Corrosion of Metal Via an Optic Waveguide Sensing Method

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Abstract: An optical waveguide sensing method was proposed for monitoring corrosion of metal in outdoors steel structures. Replacing the cladding of waveguide with metal film in a particular location, the method takes the metal film as the corrosion sensitive film. The experiment of waveguide sensor was carried out on a 721 colorimetric vessel, with which the solution is held in itself and the light is propagated in its sidewall. A Fe-C alloy film was electroplated onto the inner side of vessel to form the corrosion sensitive film. The structure of the Fe-C alloy film was studied by XRD, EDX and burning gas capacity method. The comparison test of optics and electrochemistry was used to evaluate the sensing ability of the film; it's shown that the light propagating in the optical waveguide is affected by the polarization current. The result demonstrated the feasibility of using optical fiber corrosion sensors for monitoring corrosion of outdoor steel structures.

Key words: Optic waveguide, Corrosion monitoring, Fe-C alloy

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