

# 铝表面聚苯胺的电化学合成与性能研究

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**摘要:** 目的 提高铝在含氯离子介质中的耐腐蚀性能。方法 在含有 0.4 mol/L 苯胺的 1 mol/L 硫酸中, 采用恒电位法和循环伏安法在铝表面电化学合成聚苯胺, 用红外光谱、紫外光谱和扫描电镜对聚苯胺的结构和形貌进行表征。通过动电位极化曲线和电化学交流阻抗测试, 研究聚苯胺在 0.6 mol/L NaCl、0.6 mol/L HCl、0.3 mol/L H<sub>2</sub>SO<sub>4</sub> 和 0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl 几种腐蚀介质中对铝的防护性能。结果 红外光谱表明, 合成的是硫酸掺杂质聚苯胺。紫外-可见光谱表明, 不同电化学方法合成的聚苯胺吸收峰位置相近。扫描电镜观察显示, 恒电位法制备的聚苯胺为纳米短棒状结构, 而循环伏安法制备的聚苯胺呈现出颗粒状结构。聚苯胺涂层铝在各种腐蚀溶液中的自腐蚀电位都比铝正移, 在 0.3 mol/L H<sub>2</sub>SO<sub>4</sub> 中, 恒电位法和循环伏安法制备的试样自腐蚀电位分别提高了 769、894 mV。相比于恒电位法, 循环伏安法制备的聚苯胺涂层具有更好的防腐蚀性能, 在 0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl 中的保护效率高达 91.69%, 在 0.6 mol/L HCl 和 0.6 mol/L NaCl 溶液中的保护效率分别为 80.40% 和 6.54%。结论 聚苯胺涂层在酸性溶液中比在中性溶液中具有更明显的腐蚀防护效果, 在 0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl 强腐蚀性溶液中能对铝基体起到良好的防腐蚀作用。

**关键词:** 聚苯胺; 电化学合成; 恒电位; 循环伏安; 铝; 腐蚀

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## Electrochemical Synthesis and Properties of Polyaniline on Aluminum Surface

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**ABSTRACT:** **Objective** To improve the anti-corrosion performance of aluminum in medium containing chloride ion. **Methods** Polyaniline was electrochemically synthesized on aluminum in the solution of 1 mol/L sulfuric acid containing 0.4 mol/L aniline by potentiostatic method and cyclic voltammograms. The structure and morphology of polyaniline were characterized by the infrared spectrum (IR), UV-VIS spectra and scanning electron microscope (SEM). The anticorrosion performance was tested by the polarization curves and electrochemical impedance in four kinds of corrosive medium including 0.6 mol/L NaCl, 0.6 mol/L HCl, 0.3 mol/L H<sub>2</sub>SO<sub>4</sub> and 0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl. **Results** IR and UV-Vis spectra showed that polyaniline synthesized by dif-

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**Biography:** WANG Hua (1974—), Female, Ph. D., Associate professor, Research focus: metal corrosion and protection.

ferent electrochemical methods was  $\text{SO}_4^{2-}$ -doped structure and had similar absorption peak position. SEM indicated polyaniline had a structure of granular, nano-short rod. Polyaniline coatings shifted the corrosion potential to more positive values in all kinds of corrosive medium, the corrosion potentials of polyaniline prepared by potentiostatic method and cyclic voltammograms in 0.3 mol/L  $\text{H}_2\text{SO}_4$  were positively shifted by 769 mV and 894 mV, respectively. Compared to the potentiostatic potential method, the polyaniline coating prepared by cyclic voltammetry had better anticorrosion performance, its protection efficiency was as high as 91.69% in 0.3 mol/L  $\text{H}_2\text{SO}_4 + 0.6 \text{ mol/L NaCl}$ , and its protection efficiencies in 0.6 mol/L HCl and in 0.6 mol/L NaCl solution were 80.40% and 6.54%, respectively. **Conclusion** The corrosion protection effect of polyaniline for Al in the acidic solution was more obvious than in the neutral solution; the polyaniline coating on aluminum could play a good corrosion protection role in the aggressive environment of 0.3 mol/L  $\text{H}_2\text{SO}_4 + 0.6 \text{ mol/L NaCl}$ .

**KEY WORDS:** polyaniline; electrochemical synthesis; potentiostatic method; cyclic voltammograms; aluminum; corrosion

铝及其合金凭借优异的性能,在各个领域都有着非常广泛的应用,在国民经济建设中发挥着重要的作用。在大气中,铝表面可以形成一层厚约 0.520 nm 的致密自然氧化膜,这层氧化膜能对基体起到一定的防护作用,从而减轻环境对基体的腐蚀。不过这层膜是不均匀的,通常存在着较多的缺陷,如划痕、位错及夹杂物等,难以抵抗恶劣环境(尤其是氯离子存在的环境)的侵蚀<sup>[1]</sup>。因此,需要采取一些措施对铝表面进行保护处理。

长期以来,铝及其合金最常用且有效的表面处理方法是铬化处理。通过铬酸盐处理可以得到耐蚀性良好的氧化膜,但是处理过程中存在具有致癌性的六价铬,不但对人体有较大危害,而且会造成环境污染<sup>[2]</sup>。发展无铬涂层获得了人们的高度关注,成为研究热点问题。目前常用的铝合金表面处理方法包括阳极氧化法、阴极刻蚀法、化学转化膜法、电沉积法和溶胶-凝胶法等<sup>[3-7]</sup>。电沉积法与其他表面处理方法相比,具有工艺简单、形成的膜与基体界面结合力强等优点,在 20 世纪 60 年代就已经开始了工业化生产。电沉积导电聚合物可以用作铝及其合金的防腐涂层<sup>[8-12]</sup>,如聚苯胺、聚吡咯等。它们覆盖在金属表面,促使金属表面钝化形成保护膜,起到防腐蚀的作用。其中,聚苯胺因具有原料便宜、合成条件易于控制、合成工艺简单、化学稳定性优良等优点,得到了广泛的关注和应用<sup>[13-16]</sup>。文中采用电化学方法在铝表面合成聚苯胺,研究在各种腐蚀介质中聚苯胺对铝的防护作用。

## 1 实验

### 1.1 铝片表面聚苯胺的制备

基材为 1060 铝,厚度为 0.5 mm。将裁剪成 1 cm ×

4 cm 的铝片试样依次用 500#、800#、1500#、2000#砂纸逐次进行打磨,再用去离子水冲洗干净,并用无水乙醇浸泡 2 min,晾干后,用胶带进行封装,在样品一侧留出 1 cm×1 cm 的有效工作面积,待用。

在 CS310 型电化学工作站上,通过循环伏安法和恒电位法制备聚苯胺。采用三电极体系:以经过预处理的铝片作为工作电极,饱和甘汞电极作为参比电极,铂片作为对电极。在含 0.4 mol/L 苯胺的 1 mol/L 硫酸溶液中制备聚苯胺。待实验结束后,取出工作电极,用去离子水反复冲洗,尽可能去除残余的反应物质,晾干。循环伏安法合成的试样记为 Al/PANI-CV,合成条件为:扫描电位区间 -0.2 ~ 1.3 V, 扫速 30 mV/s, 扫描圈数 15 圈。恒电位法合成的试样记为 Al/PANI-E, 合成条件为:电位 1.3 V, 时间 10 min。

### 1.2 聚苯胺的表征

1) 使用 Nicolet 6700 型红外光谱仪研究聚苯胺分子链中的基本结构信息,选择扫描范围为 500 ~ 4000  $\text{cm}^{-1}$ 。

2) 采用 Lambda 750S 型紫外可见分光光度计探究不同聚合方法对合成聚苯胺结构的影响。将聚苯胺粉末溶于 N-甲基吡咯烷酮,放入比色皿中,以 N-甲基吡咯烷酮作为参比,选择扫描范围为 200 ~ 900 nm。

3) 采用 JSM-6360LV 型扫描电子显微镜观察聚苯胺膜的形态,工作电压为 20 kV, 放大倍数分别为 5000 倍和 10 000 倍。

4) 在 CS310 型电化学工作站上,采用三电极体系进行极化曲线和电化学阻抗谱(EIS)测试。极化曲线采用动电位扫描方式进行测量,扫描速率为 1 mV/s, 测试电位范围为 -0.25 ~ +0.25 V (vs. OCP)。EIS 测试在腐蚀电位下进行,激励信号振幅为 10 mV, 扫描频率范围为 0.1 Hz ~ 100 kHz。

## 2 结果与讨论

### 2.1 聚苯胺结构与形貌分析

图1 为恒电位和循环伏安条件下制备的聚苯胺

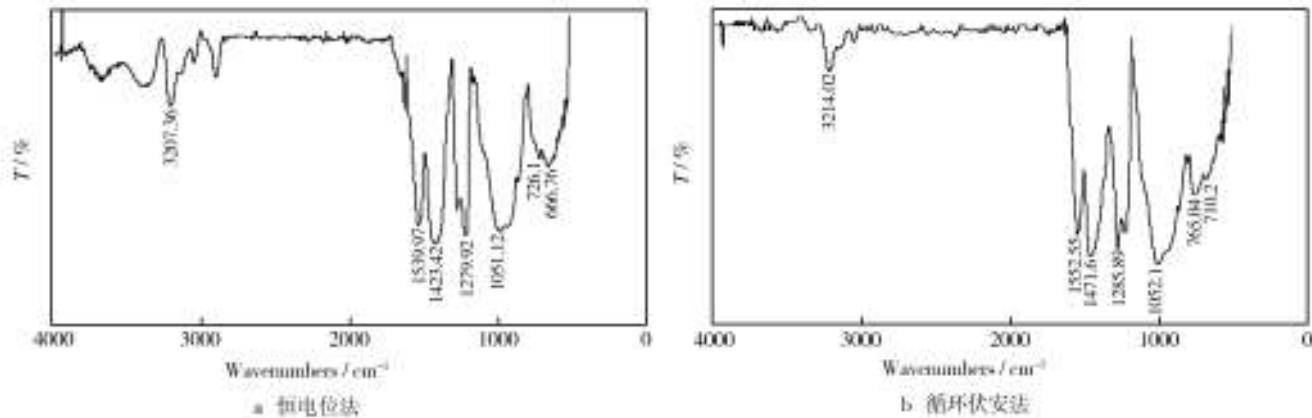


图1 聚苯胺的红外光谱图

Fig. 1 Infrared spectra of polyaniline: a) potentiostatic method, b) cyclic voltammetry

表1 聚苯胺的特征吸收峰

Tab. 1 Characteristic absorption peaks of polyaniline

| 制备工艺  | 波数/ $\text{cm}^{-1}$ |               |               |             |              |                    |
|-------|----------------------|---------------|---------------|-------------|--------------|--------------------|
|       | N—H伸缩振动              | =C—H伸缩振动      | 苯环结构上的C—N伸缩振动 | 醌式结构C=C伸缩振动 | 苯式结构中C=C伸缩振动 | $\text{SO}_4^{2-}$ |
| 恒电位法  | 3207.36              | 666.76、726.16 | 1279.92       | 1539.37     | 1423.42      | 1051.12            |
| 循环伏安法 | 3214.02              | 710.20、765.04 | 1285.89       | 1552.55     | 1471.60      | 1052.10            |

图2是聚苯胺的紫外-可见吸收光谱图。可以看出,两种方法制备的聚苯胺吸收峰位置相近,位于330 nm附近的吸收峰被认为是苯环上 $\pi-\pi^*$ 电子的跃迁,位于640 nm附近的宽吸收峰则对应于分子链内由苯环向醌环的电子跃迁。

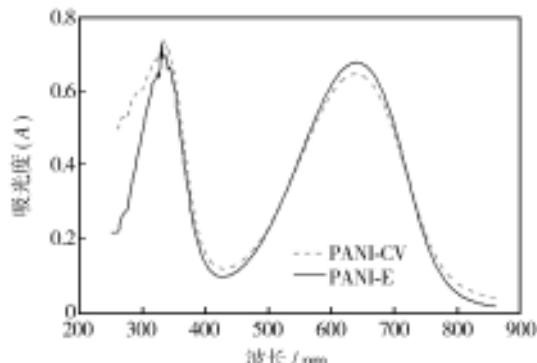


图2 聚苯胺的紫外-可见吸收光谱

Fig. 2 UV-VIS spectrum of polyaniline

的红外光谱图,各特征吸收峰列于表1。 $\text{SO}_4^{2-}$ 的振动吸收峰出现,表明合成的是硫酸掺杂质聚苯胺。由表1可见,循环伏安法制备的聚苯胺各特征峰波数值大于恒电位法制备的聚苯胺,表明循环伏安法的掺杂效应小于恒电位法。

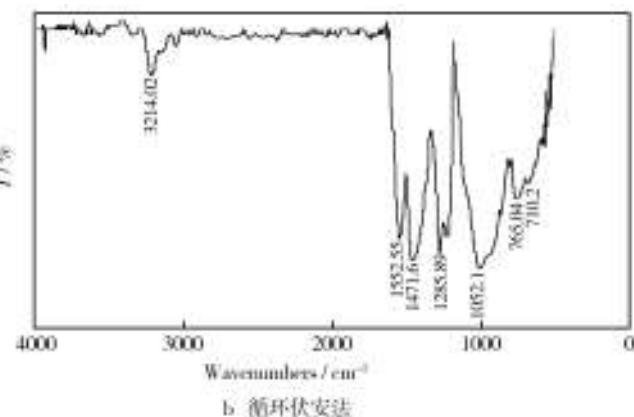


图3 聚苯胺的电镜照片

图3为恒电位条件下聚合得到的聚苯胺的电镜照片。由图3a可以看出,聚苯胺在铝表面聚集成网状,分布比较均匀。由图3b可以看出,在更高放大倍数下,硫酸掺杂质聚苯胺呈现出长为1  $\mu\text{m}$ 、直径为100 nm的棒状结构。

图4是循环伏安法制备的聚苯胺的电镜照片。由图4a可以看出,聚苯胺颗粒发生团聚,形成了致密的簇状结构。由图4b可以看出,在更高放大倍数下,硫酸掺杂质聚苯胺呈现出致密的颗粒状结构。

### 2.2 聚苯胺的耐蚀性能

#### 2.2.1 极化曲线

图5为空白铝片和聚苯胺涂层铝在不同腐蚀溶液中的动电位扫描极化曲线。将获得的数据用系统自带软件进行拟合,拟合结果见表2。其中, $E_{\text{corr}}$ 是自腐蚀电位, $J_{\text{corr}}$ 是腐蚀电流密度, $b_a$ 是阳极塔菲尔斜率,

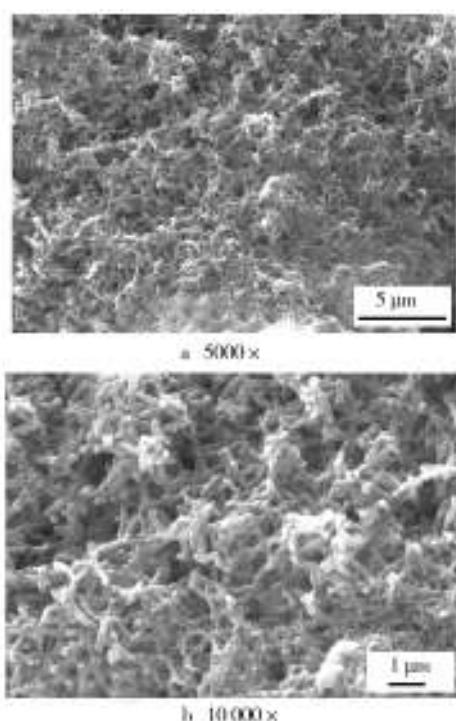


图3 恒电位法制备的聚苯胺扫描电镜形貌  
Fig. 3 SEM of polyaniline synthesized by potentiostatic method

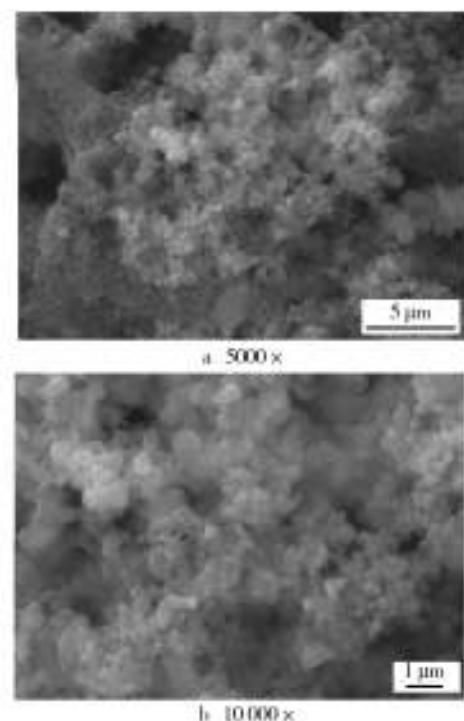


图4 循环伏安法制备的聚苯胺扫描电镜形貌  
Fig. 4 SEM of polyaniline synthesized by Cyclic voltammetry

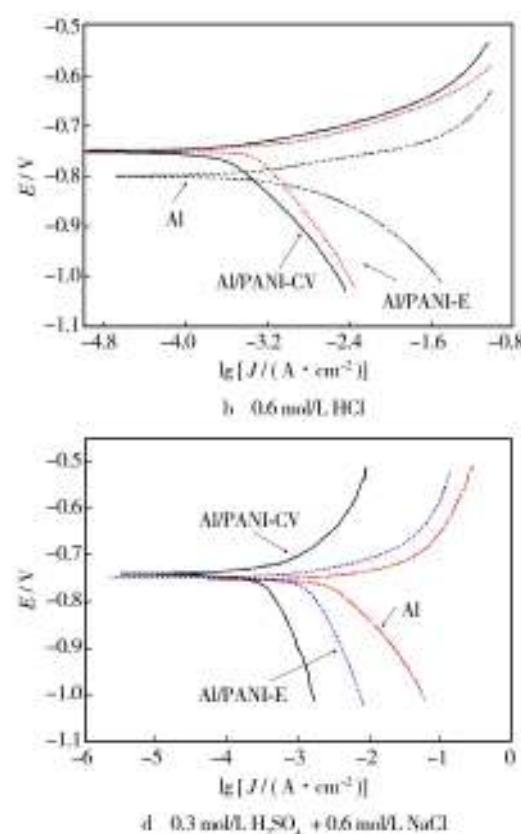
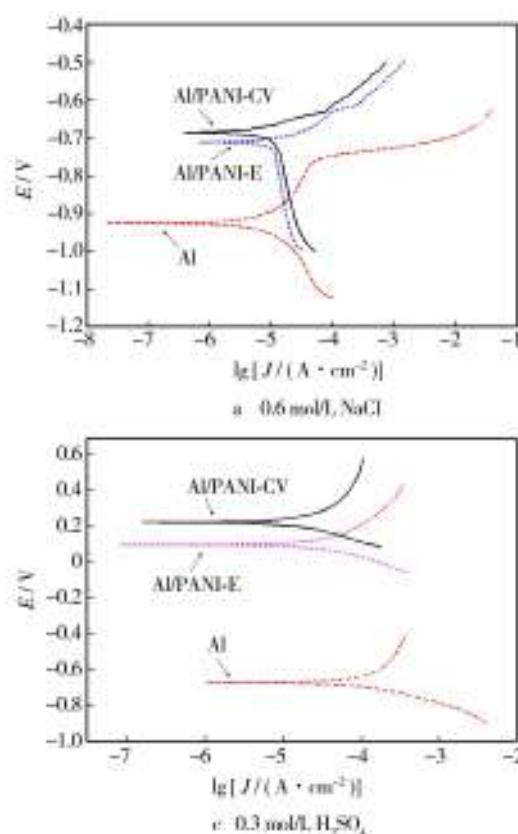


图5 铝及聚苯胺涂层铝在各种腐蚀溶液中的极化曲线  
Fig. 5 Polarization curves of aluminum and polyaniline coated aluminum in all kinds of corrosive medium

表2 空白铝及聚苯胺涂层铝在各腐蚀溶液中的腐蚀参数

Tab. 2 Corrosion parameters of aluminum and polyaniline coated aluminum in all kinds of corrosive medium

| 腐蚀溶液   | 试样         | $b_a$<br>/mV | $b_c$<br>/mV | $E_{corr}$<br>/V | $J_{corr}/$<br>( $\mu A \cdot cm^{-2}$ ) | PE<br>/% |
|--|------------|--------------|--------------|------------------|--|----------|
| 0.6 mol/L NaCl 溶液  | Al         | 218.67       | -227.91      | -0.9171          | 9.7964                                   |          |
|  | Al/PANI-E  | 46.10        | -530.70      | -0.7130          | 9.3210                                   | 4.85     |
|  | Al/PANI-CV | 81.39        | -510.88      | -0.6824          | 9.1558                                   | 6.54     |
| 0.6 mol/L HCl 溶液   | Al         | 53.65        | -148.50      | -0.7913          | $1.5827 \times 10^3$                     |          |
|  | Al/PANI-E  | 52.40        | -253.83      | -0.7538          | $0.5025 \times 10^3$                     | 68.25    |
|  | Al/PANI-CV | 50.46        | -248.71      | -0.7511          | $0.3102 \times 10^3$                     | 80.40    |
| 0.3 mol/L H <sub>2</sub> SO <sub>4</sub> 溶液                  | Al         | 301.25       | -108.07      | -0.6722          | 94.7290                                  |          |
|  | Al/PANI-E  | 114.78       | -82.118      | 0.0975           | 15.6520                                  | 83.48    |
|  | Al/PANI-CV | 244.07       | -123.50      | 0.2222           | 13.2390                                  | 86.02    |
| 0.3 mol/L H <sub>2</sub> SO <sub>4</sub> + 0.6 mol/L NaCl 溶液 | Al         | 41.50        | -188.19      | -0.7531          | $3.4775 \times 10^3$                     |          |
|  | Al/PANI-E  | 42.21        | -296.07      | -0.7449          | $1.2253 \times 10^3$                     | 64.76    |
|  | Al/PANI-CV | 73.79        | -272.6       | -0.7396          | $0.2889 \times 10^3$                     | 91.69    |

$b_c$  是阴极塔菲尔斜率, PE 是保护效率(式(1)算得)。

$$PE = \frac{J_{corr} - J_{corr}^*}{J_{corr}} \times 100\% \quad (1)$$

式中: $J_{corr}$  为空白铝的腐蚀电流密度, $J_{corr}^*$  为聚苯胺涂层铝的腐蚀电流密度。

由图5可以看出,相比于空白铝,聚苯胺涂层铝在各种腐蚀溶液中的自腐蚀电位都有所提高,热力学稳定性增强,而腐蚀电流减小。结合表2,在0.6 mol/L HCl溶液和0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl溶液中,腐蚀电位正移幅度最小(小于50 mV),但是聚苯胺涂层对阴极和阳极反应都有明显的阻滞作用,如在0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl溶液中,在相同的极化电位下,阴极还原电流和阳极溶解电流都比铝基体减少了约2个数量级。在0.3 mol/L H<sub>2</sub>SO<sub>4</sub>溶液中,腐蚀电位正移最显著,恒电位法和循环伏安法制备的试样自腐蚀电位分别提高了769、894 mV;而在0.6 mol/L NaCl溶液中,恒电位法和循环伏安法制备的试样自腐蚀电位分别提高了204、235 mV。由此可见,聚苯胺促进了铝的钝化,在铝表面形成了具有保护作用的钝化膜,抑制了铝在腐蚀介质中的阴极和阳极反应。

从表2还可以发现,在不同的腐蚀液中,聚苯胺涂层均能降低金属铝的腐蚀电流密度,而且循环伏安法制备的聚苯胺比恒电位法制备的聚苯胺效果更加

明显。通过比较腐蚀电流密度可以看出,金属铝在0.6 mol/L NaCl溶液中腐蚀速度最小;而在相同氯离子含量的条件下,pH降低会使腐蚀速度显著增加,在0.6 mol/L HCl溶液和0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl溶液中,腐蚀速度分别增加了161倍和355倍。后两种溶液虽然具有相同的pH值和Cl<sup>-</sup>浓度,但是混合腐蚀液由于离子浓度较大,具有更高的电导率,加速了腐蚀的发生,因此腐蚀速度最大。循环伏安法制备的聚苯胺膜层在0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl溶液中的保护效率高达91.69%,在0.6 mol/L NaCl溶液中的保护效率只有6.54%,说明聚苯胺膜层在酸性溶液中比在中性溶液中具有更明显的腐蚀防护性能。

## 2.2.2 电化学阻抗

图6为空白铝片和聚苯胺涂层铝在不同腐蚀溶液中的电化学阻抗谱。可以看出,在各种腐蚀溶液中,聚苯胺涂层铝的阻值都明显大于空白铝,这表明聚苯胺涂层在不同的腐蚀溶液中都能对铝片起到腐蚀防护作用。循环伏安法制备的聚苯胺涂层铝在0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl溶液中的阻抗值提高最明显,为空白铝的8倍,在0.6 mol/L NaCl溶液中的阻抗值提高为空白铝的2倍。这表明聚苯胺在酸性溶液中具有更明显的防护作用,与极化曲线测试结果相一致。

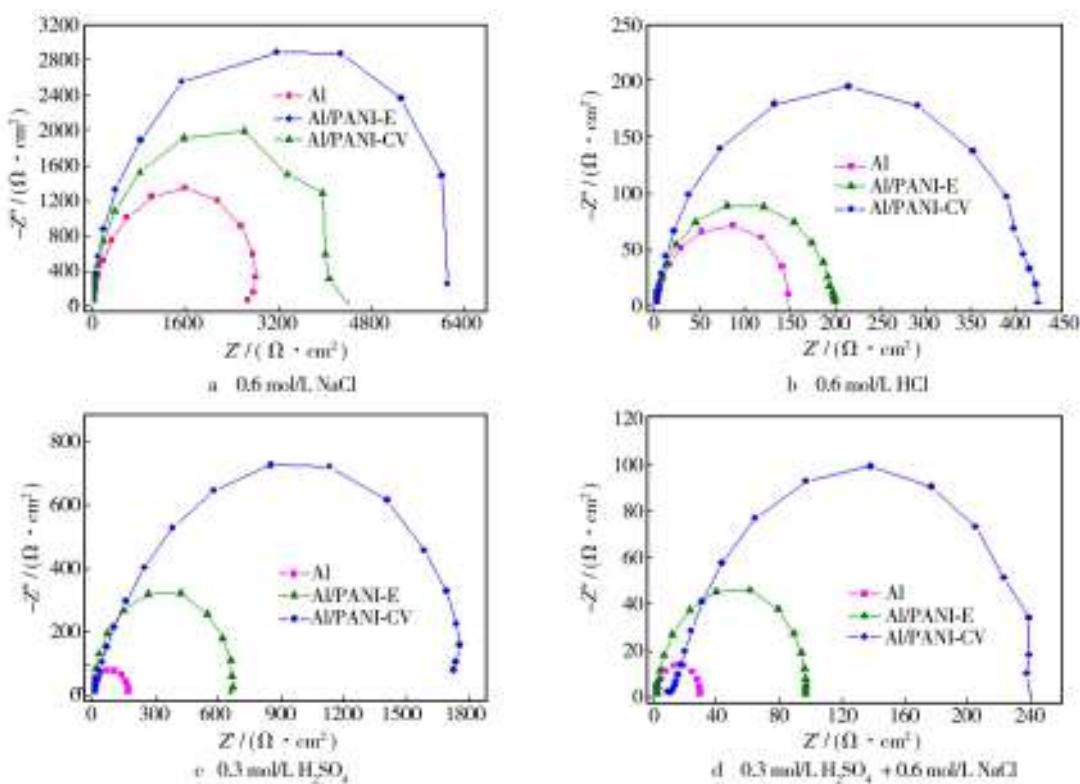


图6 铝及聚苯胺涂层铝在不同腐蚀溶液中的交流阻抗图谱

Fig. 6 EIS of aluminum and polyaniline coated aluminum in all kinds of corrosive medium

### 3 结论

1) 聚苯胺形貌受电化学合成条件的影响,可呈现出颗粒状、纳米短棒状等结构。  
2) 在 0.6 mol/L NaCl 溶液、0.6 mol/L HCl 溶液、0.3 mol/L H<sub>2</sub>SO<sub>4</sub> 溶液和 0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl 混合溶液中,聚苯胺涂层均能提高铝的自腐蚀电位,降低腐蚀电流密度,减小腐蚀速率,提高其使用寿命。

3) 循环伏安法制备的聚苯胺涂层比恒电位法制备的聚苯胺涂层具有更好的腐蚀防护性能,在 0.3 mol/L H<sub>2</sub>SO<sub>4</sub>+0.6 mol/L NaCl 溶液中的保护效率高达 91.69%,而在 0.6 mol/L NaCl 溶液中只有 6.54%。因此,聚苯胺膜层可以应用于较苛刻的酸性腐蚀环境中。

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