

环氧纳米粉末涂层现场应用评价与 缓蚀剂技术研究

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摘要: **目的** 研究一种环氧纳米粉末涂层在新疆某油田的适用性与缓蚀剂技术。**方法** 利用高温高压釜模拟涂层在新疆某油田三种典型工况条件, 结合腐蚀失重法以及结合力测试、阴极剥离、扫描电子显微镜、交流阻抗等手段, 分析环氧纳米粉末涂层腐蚀后的形态及其与基体的结合度, 对其服役寿命进行预测, 并研究缓蚀剂添加对未涂覆、破损和完整三种涂层状态下试样腐蚀行为的影响。**结果** 环氧纳米粉末涂层在三种典型环境中未出现鼓泡和开裂现象, 且与基体结合较好。环氧纳米粉末涂层的阴极剥离半径小于 5 mm, 由阴极剥离半径和阻抗值所预测的寿命分别为 883 d 和 740 d。破损涂层的均匀腐蚀和点蚀速率分别为 0.6172 mm/a 和 1.5720 mm/a, 而完整涂层的腐蚀速率仅为 0.0029 mm/a, 破损涂层阻抗值与完整涂层的阻抗值相差 10^3 倍。微量的缓蚀剂添加可降低无涂层和破损涂层试样的腐蚀速率 1 个数量级, 其缓蚀效率分别高达 91.05% 和 92.75%。**结论** 环氧纳米粉末涂层在三种典型腐蚀环境中具有好的耐蚀性能, 抗剥离能力也较好, 阴极剥离半径与阻抗值两种方法所预测的寿命基本一致。然而涂层一旦破损, 腐蚀较为严重, 尤其是点蚀, 微量缓蚀剂的添加可实现不同防护技术间的优势互补。

关键词: 涂层; 环氧纳米粉末; 寿命预测; 缓蚀剂; EIS

中图分类号: TG172.8 **文献标识码:** A **文章编号:** 1001-3660(2019)12-0312-08

DOI: 10.16490/j.cnki.issn.1001-3660.2019.12.038

Field Application of Epoxy Nano Powder Coating and Corrosion Inhibitor Technology

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收稿日期: 2019-09-26; 修订日期: 2019-10-16

Received: 2019-09-26; Revised: 2019-10-16

基金项目: 国家自然科学基金 (51974245, 21808182); 西安石油大学研究生创新与实践能力的培养项目 (YCS18212052)

Fund: Supported by the National Natural Science Foundation of China (51974245, 21808182), and Postgraduate Innovation and Practice Ability Training Program of Xi'an Shiyou University (YCS18212052)

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ABSTRACT: The work aims to study the applicability of an epoxy Nano powder coating in an oilfield of Xinjiang and corrosion inhibitor technology. Three typical working conditions of coating in an oil field in Xinjiang were simulated by high temperature and high pressure autoclave, along with corrosion weight loss method, bonding force test, cathode stripping, scanning electron microscope, and AC impedance, etc. The morphology of epoxy nano-powder coating and its binding degree to the matrix were analyzed. The service life of the coating was predicted; and the effects of corrosion inhibitor on corrosion behavior of samples with uncoated, damaged and intact coatings were studied. The results showed that the bubbling and cracking did not present on the epoxy nano powder coating after corroded in three typical environments; and the coating was well combined with the substrate. The cathode stripping radius of epoxy nano powder coating was less than 5 mm; and the lifetime predicted by the cathode stripping radius and impedance values was 883 d and 740 d, respectively. The uniform corrosion and pitting rates of the damaged coating was 0.6172 mm/a and 1.5720 mm/a, respectively; while the corrosion rate of the complete coating was only 0.0029 mm/a; and the impedance value of the damaged coating was 10^3 times than that of the intact coating. And the corrosion rate of uncoated and damaged coating samples were reduced by one order of magnitude owing to the addition of the small amount of corrosion inhibitor; and their corrosion inhibition efficiency was 91.05% and 92.75%, respectively. Therefore, this epoxy nano powder coating has good corrosion resistance in three typical corrosion environments. Its anti - stripping ability is also good. The prediction life of stripping radius and impedance is basically consistent; while once the coating is damaged, the corrosion is more serious, especially pitting corrosion. The complementary advantages of different protective technologies can be achieved owing to the addition of small amount of corrosion inhibitor.

KEY WORDS: coating; epoxy nanometer powder; life expectancy; corrosion inhibitor; EIS

随着对石油、天然气等能源需求的日益增长，其开采环境越发复杂和苛刻^[1-2]。国内主体油气藏的产出流体通常具有侵蚀性粒子（如 H₂S、Cl⁻、CO₂）浓度高和矿化度高等特点^[3]，导致管道和管柱腐蚀穿孔事故频发^[4-5]。如塔河油田油气集输管道的腐蚀特征以内腐蚀为主，且点蚀严重^[6]，其点蚀速率甚至高达 8.00 mm/a^[7]。这不仅直接影响和威胁着油气的安全生产，且造成抢救维修与治理费用的大幅增加。因此，选用合理的防护技术已成为延长金属管道使用寿命、管控安全事故和环境污染风险的重要措施^[8-9]。

管道内涂层因其低成本、易涂覆等特点成为目前最有效和实用的防护手段之一^[10]。能抑制腐蚀介质往钢铁基材渗入的有机涂层可极大延长管道的服役寿命^[11-12]。由大量活性、极性基团组成的环氧树脂分子能与多种固化剂进行交联固化，可作为油气输送管道防腐的主要底漆和面漆^[13]。然而，在较为苛刻的工况条件以及复杂的服役环境下，涂层失效问题不断^[14-15]。如塔河油田部分内涂层因其服役年限的增加，出现老化、开裂、剥落等情况，致使金属管道部分裸露，造成严重的腐蚀穿孔^[16]。因此，如何提高环氧树脂防腐性能成为国内外学者和科技工作者关注的焦点。据国内外文献报道，化学改性和利用纳米无机填料是其主要两种途径^[13]。对于纳米颗粒，不仅其小的尺寸对应力传递有利，而且极强的活性对其与环氧树脂进行交联反应有极大地促进作用，可提高分子间的结合力，并增强涂层的致密性^[17]。

涂层的防护性能与其自身的组成和结构密切相关^[18]，而其寿命受其服役环境与工况条件的制约。油田为有效地治理注采管柱严重腐蚀的问题，通常定期

或连续地往井筒中添加缓蚀剂^[19]。然而，若直接将缓蚀剂添加到涂层中，可能会发生与树脂有机官能团反应甚至不相容的现象，导致涂层缺陷增多，防腐性能降低^[20-21]。因此，需要结合工况条件和服役环境，深入研究环氧纳米粉末涂层在油气田现场的适用性，并通过在模拟腐蚀介质中添加一定剂量的缓蚀剂评估其对涂层保护效果的影响。

1 试验

1.1 腐蚀模拟试验

针对某油田三个不同特征的区块，将环氧纳米粉末涂覆于 J55 碳钢表面，对其在 H₂S、CO₂、Cl⁻环境中的适用耐蚀性能进行研究。三种模拟环境下的高温高压耐蚀模拟试验条件见表 1，试验时间为 21 d。

表 1 内涂层耐蚀性试验条件
Tab.1 Experimental conditions for corrosion resistance of internal coating

No.	Environment	Pressure /MPa	Temperature /℃	c_{H_2S} /(mg·m ⁻³)	w_{CO_2} /%
1	1 [#] block	1	65	306	3.5
2	2 [#] block	1	65	17190	6.1
3	3 [#] block	2	75	15	2.55

为更好地分析缓蚀剂对涂层防护效果的影响，向 3.5% NaCl 溶液中添加 80 mg/L 某水溶性抗 H₂S/CO₂ 缓蚀剂，在 75 ℃、0.5 m/s、H₂S 分压为 0.1 MPa、CO₂ 分压为 0.5 MPa 的条件下，对三种状态（无涂层、

涂层破损、涂层完整)的试样进行 21 d 的腐蚀模拟试验。

试验结束后,试样腐蚀速率 R_{corr} 按式(1)计算:

$$R_{\text{corr}} = \frac{8.76 \times 10^4 \times (m_0 - m_1)}{S \cdot t \cdot \rho} \quad (1)$$

式中: R_{corr} 为腐蚀速率, mm/a; m_0 、 m_1 分别为实验前后试样的质量, g; S 为试样总腐蚀表面积, cm^2 ; ρ 为试样材料密度, g/cm^3 ; t 为实验时间, h。

根据 GB/T 18590—2001《金属和合金的腐蚀点蚀评定方法》,用点蚀测深仪测量最深的点蚀深度。利用电子扫描显微镜将表面形貌放大 20 倍,测量点蚀深度。

点蚀速率 v_t 按照式(2)计算。

$$v_t = \frac{8.76 \times 10^3 \times h_t}{t} \quad (2)$$

式中: v_t 为点蚀速率, mm/a; h_t 为点蚀坑最深深度, mm; t 为实验时间, h。

1.2 性能检测与形貌表征

按照 ISO 4624—2016《色漆和清漆 拉开法附着力试验》相关步骤和要求,对涂层进行干态、湿态附着力检测。

按照 GB/T 23257—2009《埋地钢质管道聚乙烯防腐层》和 ASTM G8—2010《管道防腐层阴极剥离标准试验方法》进行阴极剥离测试,获得该试样的阴

极剥离半径 x ,再根据腐蚀试验时间 y ,利用最小二乘法原理外推预测涂层寿命。

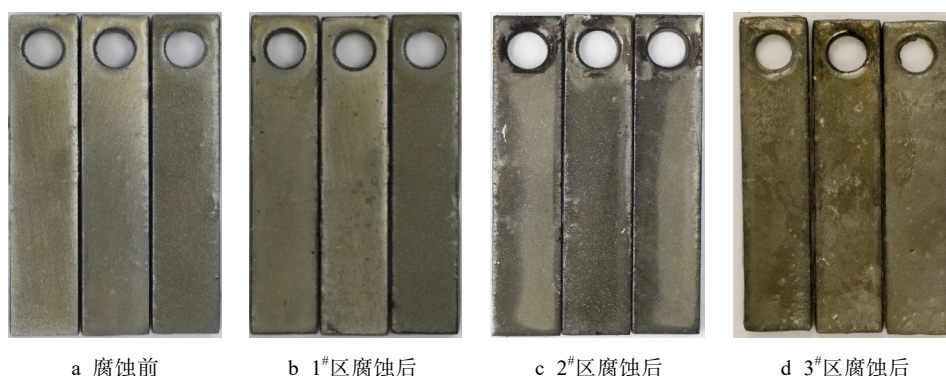
采用普林斯顿 2273 经典三电极电化学检测系统进行电化学测试,辅助电极为铂片,参比电极为 Ag/AgCl,工作电极为三种涂层状态的试样片,试验采用频率为 10 mHz~100 kHz、振幅为 10 mV 的正弦波,并利用电化学软件拟合,获取寿命预测所需交流阻抗值。另外,为进一步评估缓蚀剂的添加效果及其影响,向 3%NaCl 溶液中添加 80 mg/L 某水溶性抗 $\text{H}_2\text{S}/\text{CO}_2$ 缓蚀剂,在开路电位下分别对其电化学交流阻抗进行测量。

模拟腐蚀试验后,依次用去离子水、无水酒精清洗试样,冷风风干后,用数码相机对涂层表面进行拍照。用扫描电子显微镜(SEM)观察其微观形貌,并对涂层横截面进行表征。

2 结果与分析

2.1 不同区块腐蚀后形貌

涂层在 1[#]区、2[#]区和 3[#]区均表面完好,未见鼓泡和开裂现象,其宏观形貌如图 1 所示。将腐蚀后的涂层试样剖开,其横截面微观形貌如图 2 所示。可见,金属基体未被腐蚀,涂层与基体紧密结合,涂层内部也未见明显缺陷。



a 腐蚀前

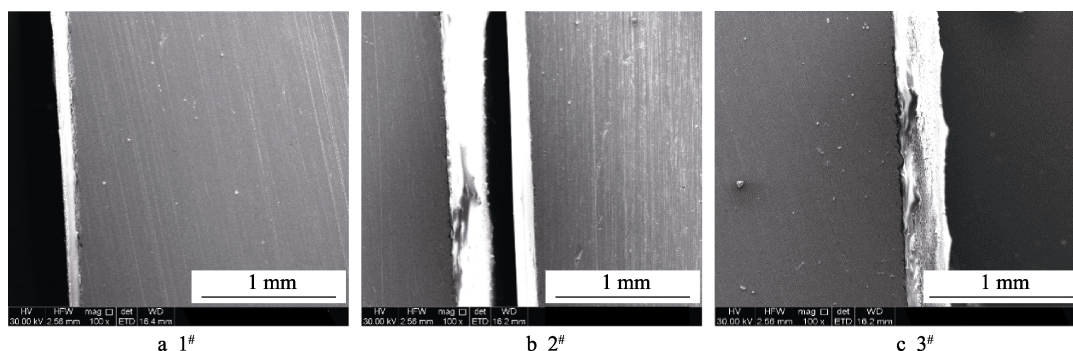
b 1[#]区腐蚀后

c 2[#]区腐蚀后

d 3[#]区腐蚀后

图 1 环氧纳米粉末涂层宏观形貌

Fig.1 Macro-morphology of epoxy nano powder coating: a) before corrosion; b) 1[#] block after corrosion; c) 2[#] block after corrosion; d) 3[#] block after corrosion



a 1[#]

b 2[#]

c 3[#]

图 2 环氧纳米粉末涂层腐蚀后截面微观形貌 (100×)

Fig.2 Micro-morphology of cross-section of epoxy nano powder coating after corrosion (100×)

总体而言,在三种典型工况条件下,该环氧纳米粉末涂层腐蚀后的宏观和微观形貌基本类似,均未观察到涂层与基体存在间隙,也未发现界面处有腐蚀产物存在,表现出与金属基体紧密结合的特性。其在干法和湿法条件下的附着力分别为 8.85、8.45 MPa,也符合技术指标要求的 8 MPa。表明该环氧纳米粉末涂层在该油田环境中的耐蚀性能较好。

与常规尺寸材料相比,纳米材料具有迥异的性能^[22],如小的尺寸、大的表面积以及宏观量子隧道效应等^[23-25]。因此,向具有三维网状结构的环氧树脂中添加纳米粉末,不仅可提高基料与填料间的相容性、紧密性,还可有效填充环氧树脂中的孔隙,显著提高涂层的屏蔽性能^[26-30],减少或阻碍侵蚀性离子到达金属基体表面,进而减缓基体表面的电化学反应,最终实现不同组分间的优势互补,达到更好的防腐效果^[31]。

2.2 内涂层服役寿命预测

首先进行油相和水相模拟环境中的高温高压

腐蚀试验,条件见表 2,试验周期分别为 30、60、90 d。

表 2 寿命预测腐蚀试验条件
Tab.2 Corrosion test conditions for life prediction

Temperature /℃	H ₂ S partial pressure/MPa	CO ₂ partial pressure/MPa	Flow velocity /(m·s ⁻¹)
75	0.1	0.5	0.5

环氧纳米粉末涂层试样在水相和油相环境中腐蚀不同时间后的宏观形貌如图 3 所示。由图 3 可见,在水相环境中,试样经过 30 d 腐蚀后已失效,这与涂层失效机制一致。尽管垢物在流动状态难以在涂层表面沉积^[32],然而水相中由于水分子和侵蚀性离子的迁移而易进入涂层内部^[14],并经孔隙到达金属的表面,进而诱发电化学腐蚀反应^[31],生成如 FeCO₃ 或 FeS 等,阻塞了孔隙的通道。然而该类腐蚀产物不仅具有离子选择透过性(半渗透性)^[33],而且还具有足够的机械强度以抵抗渗透压,涂层发生失效—鼓泡的条件随之形成^[34]。



图 3 环氧纳米粉末涂层试样在不同环境中腐蚀后的宏观形貌

Fig.3 Macro-morphologies of epoxy nano powder coating samples after corrosion in different environment: a) water phase; b) oil phase

刘野等人^[35]认为,涂层失效形式主要有三种:涂层与基体间的粘着力不够,涂层因此而易脱落;涂层自身耐酸、碱、盐等介质化学侵蚀性能不好,导致其对基体的保护减弱;涂层因缺陷或孔隙而阻挡 Na⁺、Cl⁻等小离子渗透性差,导致涂层电阻下降,其中涂层下电化学腐蚀在油气田现场最为典型。无论哪种失效形式或失效机制^[36],涂层一旦失效,其在管道表面的完整性将遭受破坏,形成“大阴极-小阳极”的腐蚀状态,管道将会在极短的时间内发生腐蚀穿孔。因此,就需要额外的防护技术(如缓蚀剂)进行替代或弥补。

阴极剥离和电化学阻抗测试试验选择在油相环境腐蚀后的涂层试样,获得涂层试样不同腐蚀时间的阴极剥离半径和阻抗值。经 1.5 V 电火花检查未发现漏点,阴极剥离半径均小于 5 mm,阻抗值高,其结果分别见图 4 和表 3。通过最小二乘法原理对腐蚀后涂层试样的阴极剥离半径和电化学阻抗值数据进行拟合,得到阴极剥离半径和阻抗值与腐蚀时间的拟合曲线和拟合函数。

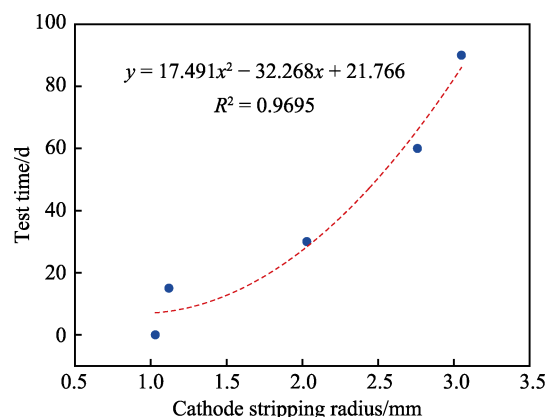


图 4 涂层阴极剥离半径与腐蚀时间及其拟合曲线
Fig.4 Relationship between cathode stripping radius of coating and corrosion time and their fitting curve

设定涂层阴极剥离半径大于 8 mm 或者阻抗值低于 $1 \times 10^6 \Omega \cdot \text{cm}^2$ 时即涂层失效^[37-38],利用上述拟合曲线的二次多项式预测该环氧纳米粉末涂层寿命分别

表3 油相环境腐蚀后的涂层试样电化学阻抗值
Tab.3 Electrochemical impedance value of coating samples after corrosion in oil phase environment

Test time/d	0	15	30	60	90
Impedance value/($\Omega \cdot \text{cm}^2$)	7.3×10^9	4.5×10^9	1.9×10^9	7.2×10^8	5.3×10^8
Fitting function and variance	$y=44.147x^2+20.066x+3.4928$, $R^2=0.972$				

为 883、740 d, 该预测的寿命远小于实际工况环境下涂层的服役时间。涂层失效不仅是一个自然老化过程, 而且还是受其服役诸多工况条件影响(如表面处理、涂层厚度、流体介质等)的复杂过程。因此, 若要准确地预测涂层服役寿命, 需考虑在现场放置涂层挂片, 定期取出, 进行相关测试, 得到涂层的某种性能数据。然后通过曲线拟合求得二次多项式, 从而进行寿命预测。或采用实验室高温高压环路模拟系统或类似装置模拟现场腐蚀环境, 进行相关测试, 以此对照, 将室内试验模拟的预测结果乘以相应的系数。

2.3 缓蚀剂模拟工况腐蚀试验

2.3.1 质量损失率

两种状态(裸钢、破损涂层)试样在(未)添加缓蚀剂条件下的质量损失率如图5所示。其试验条件见表2, 试验周期为7 d。可见, 未添加缓蚀剂时, 不仅裸钢试样的腐蚀速率较高, 破损涂层试样金属也遭受较严重腐蚀, 尤其是点蚀, 其点蚀速率为 1.572 mm/a 。

缓蚀剂的添加使金属基体得到了有效的保护, 其均匀腐蚀速率降低1个数量级, 无涂层试样和破损涂层试样的缓蚀效率分别为91.05%和92.76%, 基本相同。对于完整涂层而言, 在未添加缓蚀剂的条件下, 其腐蚀速率为 0.0029 mm/a ; 添加了缓蚀剂后, 其腐蚀速率反而稍有增加, 为 0.0078 mm/a 。此与 Shen 等人^[20]的研究结果相一致。可见, 尽管缓蚀剂组分和环氧树脂有机官能团间存在一定的相互作用^[21], 但当涂层发生破损时, 添加的缓蚀剂可在第一时间达到金属基体表面, 对其进行保护。

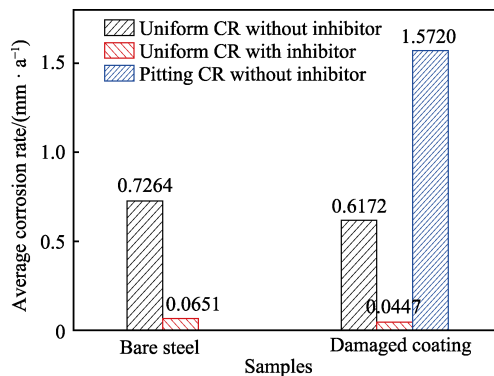


图5 裸钢与破损涂层试样在添加缓蚀剂前后的质量损失速率

Fig.5 Weight loss rate of bare steel and damaged coating sample before and after adding corrosion inhibitor

2.3.2 形貌特征

三种状态试样腐蚀前的形貌如图6所示, 其在未添加缓蚀剂与添加缓蚀剂溶液中腐蚀后的形貌如图7和图8所示。对比可见, 试验后试样的表面颜色均发生变化, 因硫化物的产生而呈黑色。清洗后, 未有涂层覆盖的试样在未添加缓蚀剂的溶液中腐蚀后, 表面点蚀坑明显, 这与上述点蚀质量损失率较高有很好的一致性; 而添加缓蚀剂后, 未有涂层覆盖的试样表面几乎还光亮如初, 更未见明显的点蚀坑, 表明该缓蚀剂的缓蚀效果较好。

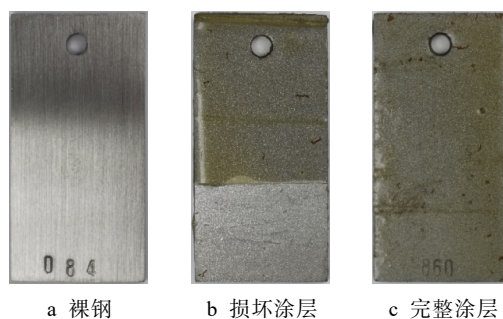


图6 未添加缓蚀剂试样腐蚀前宏观形貌
Fig.6 Macro-morphology of specimen before corrosion without inhibitor: a) bare steel; b) damaged coating; c) integral coating

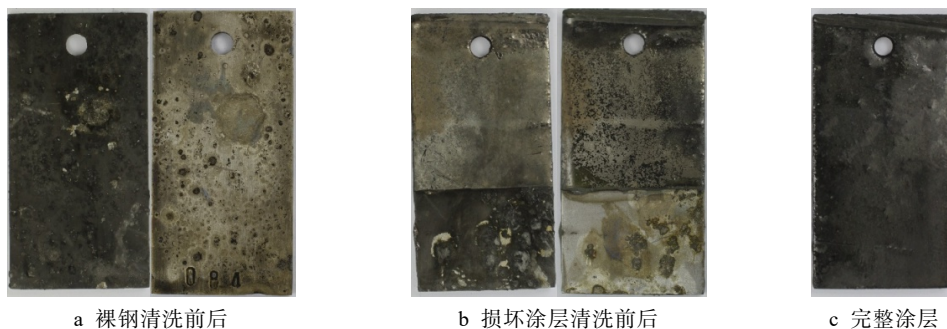


图7 未添加缓蚀剂条件下试样腐蚀宏观形貌

Fig.7 Macro-morphology of sample after corrosion without inhibitor: a) bare steel before and after cleaning; b) damaged coating before and after cleaning; c) integral coating

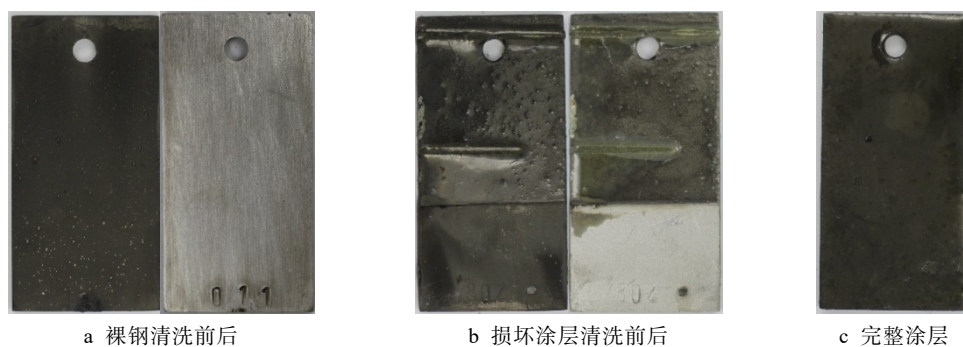


图 8 添加缓蚀剂条件下试样腐蚀宏观形貌

Fig.8 Macro-morphology of sample corroded after corrosion with inhibitor: a) bare steel before and after cleaning; b) damaged coating before and after cleaning; c) integral coating

2.4 缓蚀剂电化学特征

破损涂层与完整涂层以及缓蚀剂添加前后的交流阻抗图谱如图 9 所示。经拟合发现,破损涂层阻抗值与完整涂层相差 10^3 倍,说明涂层破损后金属基体会受到明显的腐蚀。添加缓蚀剂后,阻抗值明显增大,表明缓蚀剂的添加缓解或阻碍了腐蚀介质通过微孔进入涂层内部,进而减缓或抑制了在涂层/金属基体界面处所发生的电化学腐蚀反应^[39-40]。结合腐蚀质量损失试样的结果,说明当管道内涂层发生破损时,可以采用加注缓蚀剂方法对金属基体进行保护。

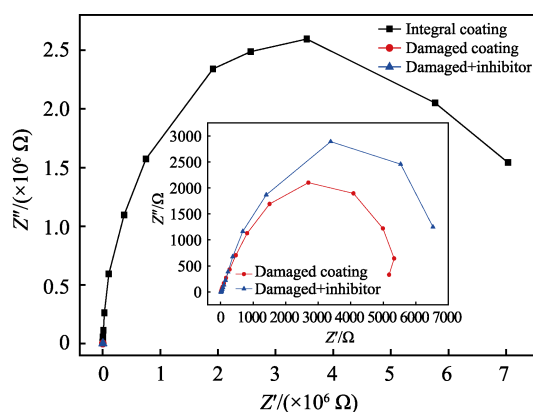


图 9 涂层+缓蚀剂电化学交流阻抗谱

Fig.9 EIS of the coating without and with corrosion inhibitor

3 结论

1) 环氧纳米粉末涂层在新疆某油田的三种典型环境中表现出较好的耐蚀性能。

2) 该涂层的阴极剥离半径小于 5 mm, 剥离半径与阻抗值两种方法的预测寿命基本一致且较长, 约 2~2.5 年。

3) 破损涂层遭受较为严重的腐蚀, 尤其是点蚀。微量缓蚀剂的添加将极大地降低管道腐蚀穿孔的风险, 可实现不同防护技术间的优势互补。

4) 考虑到注采系统中缓蚀剂添加的普遍性, 建

议在预测涂层服役寿命时以现场放置涂层挂片所采集的数据为参考, 或在室内预测结果基础上乘以相应系数。

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