Application Note AN-COR-016

ASTM G61:循环电位极化的标准测试方法

ASTM-compliant methods from Metrohm Autolab

ASTM G61 is a standardized method to test susceptibility of various alloys of iron, nickel, and cobalt to localized corrosion within a chloride environment [1]. The potential at which the anodic current increases rapidly is an indication of pitting. The higher the potential, for the same scan rates, indicates better protection against pitting corrosion.

By using a Metrohm Autolab instrument and our ASTM-compliant corrosion cells, it is possible to fully meet the requirements of this ASTM standard. The following Application Note describes an example measurement that was made using VIONIC powered by INTELLO according to the guidelines of ASTM G61.

SAMPLE PREPARATION

It is essential that the surface of the sample is free from contamination. Therefore just prior to immersion in the corrosive medium, the sample (a 1 cm^2 disk of Type 430 stainless steel) was cleaned by a combination of mechanical polishing with sandpaper and alternatively rinsing with ultrapure water and isopropyl alcohol.

EXPERIMENTAL

The test consists of polarizing the system toward potentials that are more positive than the open-circuit potential (OCP) until the current reaches a predetermined value (usually 5 mA). Then the scan is reversed, forming a hysteresis loop on the voltammogram. The higher the potential at which the hysteresis loop is closed, the less prone the alloy is to pitting corrosion.

For this experiment, the sample (430 SS) was used as a working electrode (WE). Two Metrohm platinum sheet electrodes were used as the counter electrode. As a reference electrode, an Ag/AgCl 3 mol/L KCl electrode was chosen. The cell used in this study was the ASTM-compliant Metrohm Autolab 1 L corrosion cell. The electrolyte was a 3.5% NaCl solution (artificial seawater).

Nitrogen gas was bubbled into the solution for one hour during the preparation step to remove any oxygen dissolved in the electrolyte. After one hour, the sample was immersed in the electrolyte, and the degassing continued for another hour.

Then 10 minutes before the start of the polarization (i.e., 50 minutes after insertion), the OCP of the counter electrode was recorded using the S2 connection of VIONIC. The OCP of the sample (WE) was measured, and the scan started from 0 V vs OCP. The scan rate used was 167 µV/s with a step potential of 150 µV.

The anodic scan continues until a current cut-off of 5 mA is reached, at which point the scan direction is reversed. The measurement stops when either the corrosion potential (E_{corr}) is reached, or the hysteresis is closed (noted by manual observation).

RESULTS AND DISCUSSION

The OCP (platinum potential) of the counter electrode was recorded as $E_{C\text{-}OCP}$ = 0.24 V. The corrosion potential of the working electrode was recorded as E_{corr} = -0.28 V vs Ag/AgCl.

In **Figure 1**, the resulting voltammogram (I vs E) is shown.

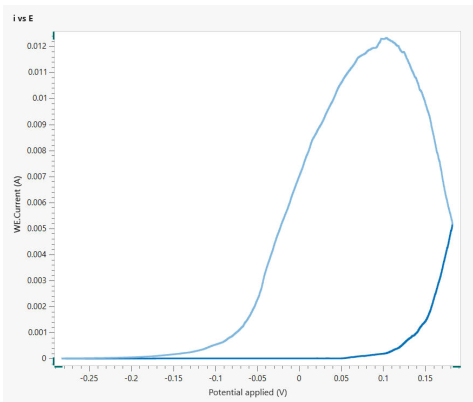

Figure 1. Voltammogram (I vs E) of thev Type 430 stainless steel sample under investigation.

RESULTS AND DISCUSSION

The data is transformed according to ASTM standard G3 [2] in **Figure 2**, where a plot of the potential (E) vs the log of the current density (j) is shown.

In this case, this sample exhibited an open hysteresis, so the measurement ended once the corrosion potential was reached again. E_{pitt} is the pitting potential and corresponds to the potential at which the pitting (localized) corrosion begins. Between the initial E_{corr} and E_{pitt} is a current density passivity range where new pits are unable to form, but existing ones can propagate [**3**]. The high hysteresis indicates that the sample has undergone pitting corrosion.

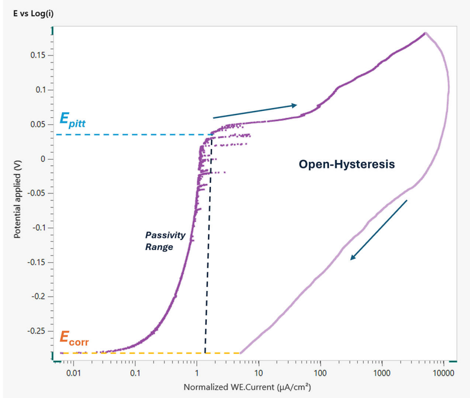

Figure 2. E vs log(j) plot of the sample under investigation. The corrosion potential (Ecorr), pitting potential (Epitt) and the passivity range are all indicated on the plot. The arrows indicate the scan direction.

REFERENCES

1. G61 Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys. https://www.astm.org/standards/g61 (accessed 2024-05-24).
2. Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing. https://www.astm.org/g0003-14r19.html (accessed 2024-03-08).
3. Bellezze, T.; Viceré, A.; Giuliani, G.; et al. Study of Localized Corrosion of AISI 430 and AISI 304 Batches Having Different Roughness. Metals **2018**, 8 (4), 244. DOI:10.3390/met8040244

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CONFIGURATION

该电极由铂金薄片嵌入玻璃杆中组成。此电极可作为相对电极用于多数电化学测量。其表面积约为 1 cm2。

KCl Ag/AgCl 12.5 cm

带双结系统的银/氯化银参比电极,安装长度 10 cm。由于配备标准磨口 14/15,故此可以方便的进行安装,并且可以随时更换对污染不敏感的柔性磨光隔膜。参比和外参比液电解质可根据用途自由选择,并且易于更换。该传感器预填充 c(KCl) = 3 mol/L 作为参比和中间电解质。

1 L

Autolab 1 L 腐蚀池适用于按照 ASTM 标准进行的腐蚀测量。该腐蚀池设有一个用于温度控制的恒温夹套和一些用于对电极、pH 传感器、温度计、Luggin-Haber 毛细管和气体排气的开孔。

该 1 L 的腐蚀池设计用于测量直径为 14.7 至 16 mm 厚度为 0.5 至 4 mm 的样品腐蚀情况。裸露的表面积为 1 cm2。用天然橡胶密封。

VIONIC

VIONIC 是我们的新一代恒电位仪/恒电流仪,由 Autolab 的新 INTELLO 软件提供动力。

相比目前市场上任何单一仪器,VIONIC 提供了**全面**的组合规格。

- 顺从电压:± 50 V
- 标准电流 ± 6 A
- EIS 频率:达 10 MHz
- 采样间隔:低至 1µs

VIONIC 的价格中通常还包括大多数其他仪器产生的额外费用,例如:

- 电化学阻抗谱 (EIS)
- 可选浮动配件
- 第二测量电极 (S2)
- 模拟扫描