Metrohm Autolab B.V.

Autolab Application Note COR08

Stepwise dissolution measurement

Keywords

Corrosion, aluminum, coating, acceleration methods.

Introduction

The stepwise dissolution measurement (SDM) is an electrochemical technique used to test the corrosion properties of aluminum-brazing sheets. The SDM is a modification of the anodic accelerated dissolution technique (AAD), developed by Corus Aluminium Walzprodukte (formerly Kaiser Aluminum; Koblenz, Germany). Both techniques have been developed in order to have results in relatively short times, typically hours, compared to days needed to complete the classical salt spray tests. However, in AAD, high over potentials (50 to 60 mV) are used, resulting in a too aggressive corrosion acceleration and a poor comparison with the salt spray experiment [1].

Therefore, the SDM technique has been developed as improvement of the AAD. The scope is to have a non-aggressive technique in which selective dissolution can take place and it is possible to distinguish between good and bad corrosion behavior of different materials.

Experimental Setup

Three sheets of coated aluminum have been tested. All of them have a thin coating of aluminum oxide, Al_2O_3. Two of them present one further coating layer. The exact composition of the coatings was not made available. Therefore, further in the text, we will refer to them as “pink” and “gold” coatings. Each sheet was punched to obtain disks of 1.5 cm diameter, to fit the sample holder of the AUTOLAB 1 L corrosion cell, shown in Figure 1. One side of each sample was polished with sandpaper, to remove the coating layers and to assure good electrical contact with the sample holder. All measurement were carried out in artificial seawater, obtained by dissolving 33 g of NaCl in one liter of Millipore water. A stainless steel counter electrode and an Ag/AgCl 3M KCl reference electrode completed the three-electrode setup. In this note, all potentials are quoted with respect to this reference electrode. The electrodes were connected to an Autolab PGSTAT204, equipped with a FRA32M impedance module, shown in Figure 1.

Figure 1 - The 1 L corrosion cell and the PGSTAT204 with the FRA32M module

The procedure

The NOVA procedure has been written in accordance with the SDM technique, which consists of the following steps:

- Record the open circuit potential (OCP) for five minutes.
- Increase the potential 20 mV vs. OCP, and keep the system polarized for 30 minutes.
- Record the OCP for five minutes.

The two latter commands are repeated twelve times, for an overall duration of ~ 7 hours. A plot of OCP vs. cycle number has been built. The charge is also sampled, since the charge collected per step is an indication of the amount of aluminum facing the electrolyte, so being an indirect measure of the coating dissolved in that step. From the collected charge for each step, the cumulative charge is then calculated. The cumulative charge is the total charge accumulated up to each step. A plot of the cumulative charge vs. the cycle number has been built.

Results and discussion

In Figure 2, the OCP at the end of each cycle is plotted vs. the cycle number. The oxide-coated sample displays the most stable OCP during all the measurement, with also the highest mean OCP value, reported as - 0.73 V. The pink-coated sample has OCP values close to the oxide values, but less stable. After the first cycle, its value increases of ~ 40 mV and then it decreases of a similar amount (i.e., 35 mV) after the 4th cycle. Then, the OCP constantly increases after each cycle. The mean value of the OCP is - 0.74 V. The gold-coated sample has an average OCP of - 0.83 V. Besides, its

value increases after the first cycle, then it changes less dramatically than the pink-coated sample, remaining at ~ -0.83 V.

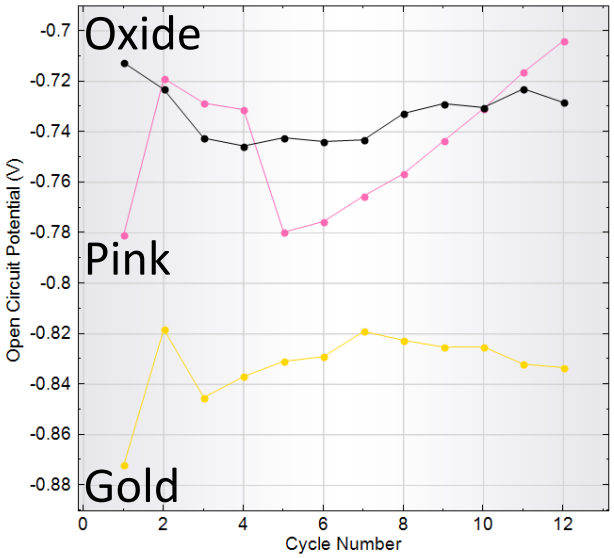

Figure 2 – Open circuit potential vs. cycle number, for the three samples

Usually, a material with a lower OCP has a weaker protection against corrosion. In this example, the gold-coated has the lowest OCP and, therefore, it should be the most prone to corrosion, with respect to the other two samples.

The plot in Figure 3 can be of further help. Here, the cumulative charge versus the cycle number has been plotted, for each samples.

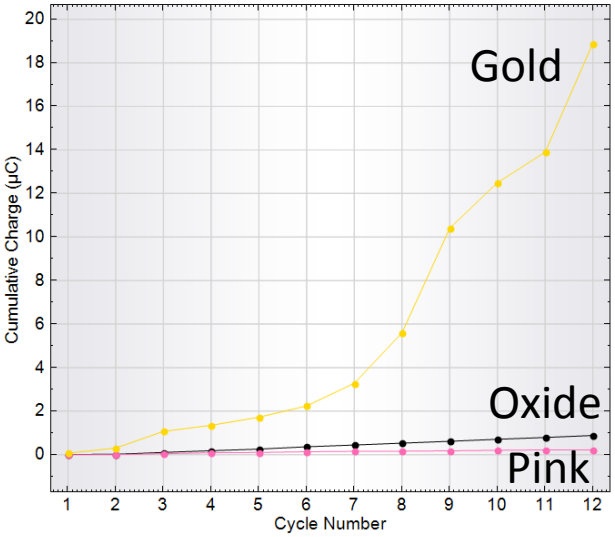

Figure 3 – Cumulative charge vs. cycle number for the three samples

As it can be seen, the highest amount of charge is collected from the gold sample. Also the cumulative charge increases dramatically after each step. On the other hand, the pink sample delivers the least amount of charge and the oxide-coated sample has close values to the pink-coated one. Both of them have a slight increase of cumulative charge, after each cycle.

The plot in Figure 3 indicates that during the stepwise dissolution measurement, the gold-coated sample corrodes more than the two other samples. In this respect, the pink-coated sample seems to give the best protection against corrosion.

Conclusions

The stepwise dissolution measurement is a relatively fast technique, firstly developed to test the aluminum-brazing sheet. In this application note, SDM is applied to aluminum samples coated with different materials, in order to have insights on the differences in terms of corrosion protection. The combination of Autolab PGSTAT204 with the 1 L Autolab corrosion cell and the NOVA software provides the suitable setup to perform SDM and other corrosion experiments.

References

[1] S.D. Meijers, “Corrosion of aluminium brazing sheet”, 2002 Proefschrift, Technische Universiteit Delft, NL; pp 156.

Date

20 January 2015