Autolab Application Note COR06

Corrosion
Part 6 – Critical Pitting Temperature Measurements with pX1000

Keywords

Corrosion; Pitting; Temperature control; pX1000 module

Summary

Critical pitting temperature (CPT) is the lowest temperature on the test surface at which stable propagating pitting occurs under specified test conditions indicated by a rapid increase beyond a set limit of the measured anodic current density of the specimen.

The method is suitable for characterizing the pitting corrosion resistance of stainless steel and related alloys. This measurement technique can be used in research and development studies, production test and acceptance, materials control.

The Critical pitting corrosion consists in a Chrono amperometric experiment combined with a temperature ramp. The temperature is controlled through NOVA software or adjusted manually (ramp of 1ºC/min) using a thermostatic bath and a temperature sensor placed in the measurement cell. The test solution used to run all experiments was 1M NaCl and a Metrohm PT1000 (6.1110.100) connected to the pX1000 module (Figure 1).

Figure 1 – Metrohm PT1000 sensor

The cell used for the experiment is shown in Figure 2. The counter electrode is a glassy carbon rod Metrohm (6.1248.040), the reference electrode is a Ag/AgCl 3M KCl Metrohm (6.0726.100). The stirrer has been use during the stabilization temperature before and after all the experiments.

The solutions were not deareated.

There are two different ways to measure the CPT:

1. Apply a temperature ramp from 25ºC to 60ºC and monitor the current variation at fixed potential
2. Apply 5ºC temperature steps from 25ºC to 60ºC and monitor the current for 60 seconds at fixed potential

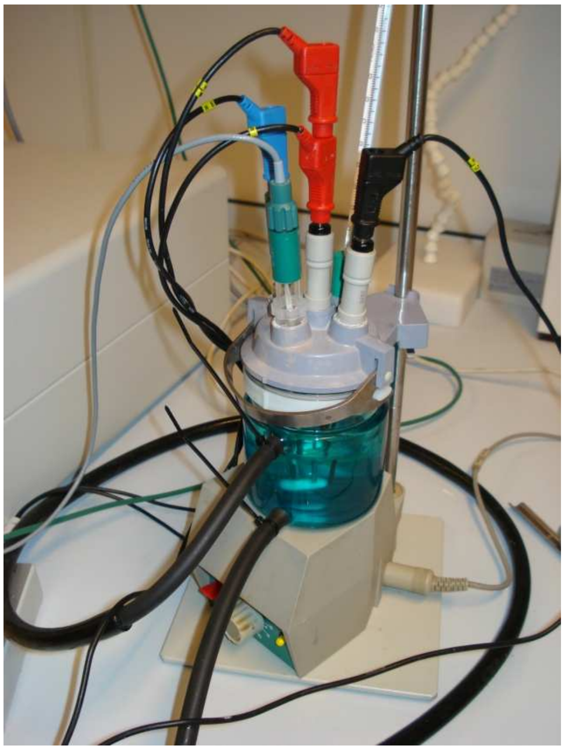

Figure 2 – Cell setup used in this application note

In both cases a sudden variation of the current will indicate the CPT.

Preliminary measurements

Polarization resistance measurement

To arrange proper measurement condition for CPT determination, we measured the linear polarization curves at different temperatures in order to determine a suitable temperature range.

Figure 3 shows typical linear polarization curves for 304 steel coupons at 25ºC (blue line) and at 60ºC (red line).

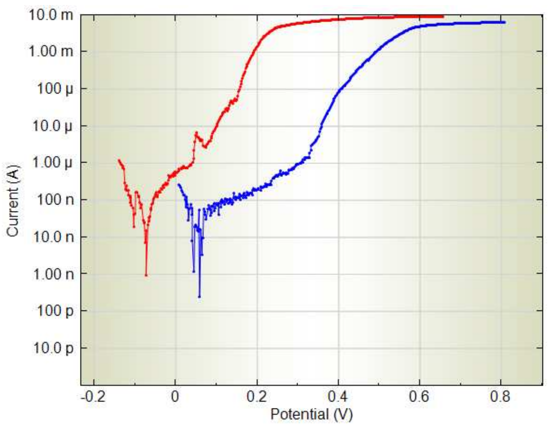

Figure 3 – Polarization curves for 304 steel recorded at 25℃ (blue line) and 60℃ (red line)

From the above graph we can see the change of the corrosion potential with increasing temperature and a rapid increase of the current on the anodic side of the curve even though the final current value reached is similar.

From the Figure 3 it is possible to recognize the change in potential where the corrosion occurs.

Multipotential step chronoamperometry

A second useful test consists of a multi potential step from 100mV to 700mV Chrono amperometry experiment at different temperature.

Figure 4 shows a 60 s measurement with potential steps from 200 mV above the OCP to 700 mV at 25ºC and 60ºC.

In this case it is possible to recognize an increase of the current at 60ºC already at 300 mV above the OCP, this current increase is similar to the increment at 700 mV at 25ºC.

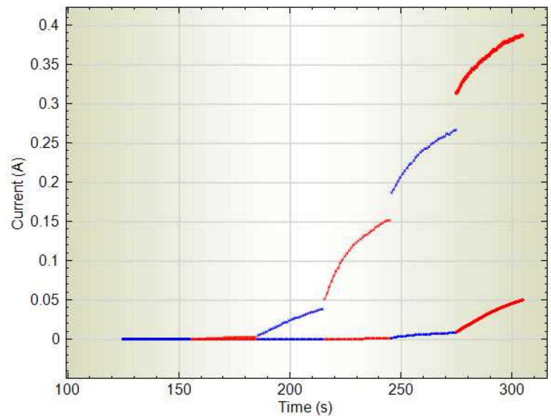

Figure 4 – Multipotential steps measurements at different temperature

Critical pitting temperature experiments

To test the CPT we apply a continuous ramp of temperature from 25ºC to 60ºC while monitoring the temperature and the current at the same time while keeping the potential fixed.

Figure 5 shows two experiments: (1) at 100 mV above the OCP (blue line) and (2) at 50mV above the OCP (green line) the temperatures where the CPT takes place are very different: one is 49.5ºC and the other 55.4ºC.

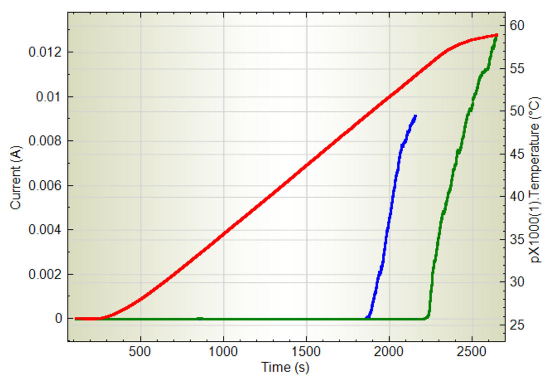

Figure 5 – CPT measurements at different potentials

Figure 6 shows two different kinds of steel samples at 100mV above OCP same temperature ramp same conditions but two different CPT.

A second type of CPT can be performed with temperature steps instead a temperature ramp protocol. In Figure 7 temperature steps of 5ºC from 30ºC to 60ºC were applied and the current was measured for 60 seconds until there is a measurable variation.

In red temperature steps every 5ºC, then in green the increment of the temperature between the steps. The other colored lines are the current vs time graphs.

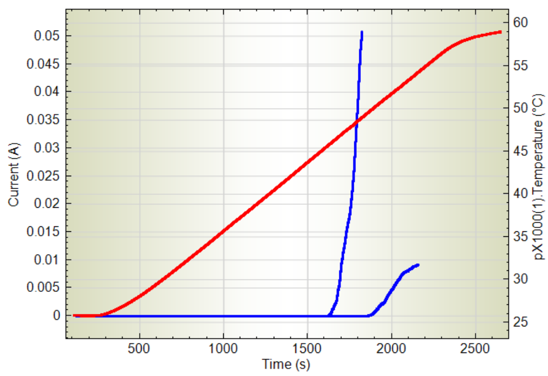

Figure 6 – CPT measurements for different steel samples

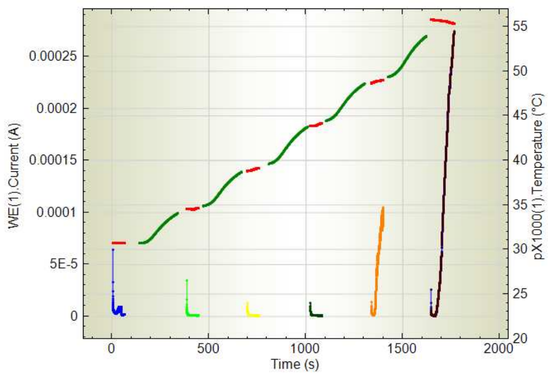

Figure 7 – Composite analysis graph temperature steps

From these graphs it is possible to see that the current increment at the last two temperature steps is increasing dramatically since the temperatures were higher than the Critical pitting temperature of the material tested.

Conclusions

This application note has illustrated the use of the pX1000 module for temperature monitoring and setting in linear polarization and pitting corrosion.

All experiments are easy to manage with Autolab PGSTAT equipments and NOVA software.

RS232 control of the water bath directly through the software allows automatic programming of temperature steps or ramps.

More information about CPT measurements can be found in ASTM G150-99

References

- ASTM G150-99, Standard Test Method for Electrochemical Critical Pitting Temperature Testing of Stainless Steels

Date

1 July 2011