ABSTRACT

The two hydrofluoric acid (HF) storage tanks used for holding 70% technical grade HF product at the HF plant at Necsa started leaking in March 2012. An evaluation of the failure was conducted in the form of a corrosion failure analysis. It was confirmed that a higher than usual nitric acid (HNO_3) content in the technical grade HF stream changed the corrosion mechanisms typically experienced within the HF storage vessels, which then caused the tanks to fail.

Immersion type corrosion experiments were done to safely simulate the corrosive environment experienced by the mild steel, stainless steel and nickel alloys used on site, and to predict the change in corrosion rates and characteristics associated with the HNO_3 contamination in the HF production plant circuit. Since the corrosion resistance of mild steel in HF is heavily dependent on the thickness of the protective scale on the steel, a series of planned interval corrosion tests (PICTs) was done to reproduce and then examine the oxide-fluoride barrier on mild steel coupons in pure 70% HF prior to corrosion tests. These shorter PICTs were also done on the stainless steel and nickel alloys and showed that the pre-passivation step had a surface cleaning effect when exposed for only 24 h.

Eleven day corrosion tests were conducted to establish the effect of HNO_3 concentration and temperature on mild steel corrosion in 70% HF, and to determine the change in corrosion rates and mechanisms associated with HNO_3 contamination (0.1-1% HNO_3) of the downstream HF products. The corrosion was characterized by analysing the corroded coupons for mass loss, apparent corrosion rates, acid consumptions, visual observations of scale formation and pits, as well as depth profiles from scanning electron microscopy and energy dispersive spectroscopy analyses. Linear relationships were frequently observed when analysing mass losses for the coupons over time, making it possible to define corrosion rates in terms of first order reaction kinetics. The harshest corrosive condition for mild steel in HF was determined to be 1% HNO_3 in 70% HF at a constant temperature of 25ºC.

The corrosion characteristics of alloys used in the HF plant, as affected by HNO_3 impurities (in the range 50–10000 ppm) in the final HF acid product (70% Technical grade) were successfully established. Normalized SA516 Grade 70 mild steel and Monel 400 were found not adequate for use as construction materials in a plant where HNO_3 contamination was >100 ppm. However, the corrosion resistance of SS 904 L was suitable under these

conditions and was recommended for applications in HF solutions where the presence of an oxygen-containing acid (e.g. HNO_3) is consistent.

It was recommended that Alloy 31, Alloy 33 or Nirosta 4565S, with higher chromium content (>20 wt% Cr), should be considered for construction material of the HF plant when HNO_3 contamination becomes unavoidable. However, if the continued use of mild steel at the plant cannot be avoided, other inhibition strategies tailored to the selective consumption of HNO_3 in the HF product stream need to be investigated.