

Adaptation of standard instruments to stand specific sample and process conditions – corrosive atmospheres

INTRODUCTION

It may occur that standard instruments are limited in terms of application because of temperature, pressure ranges available, because of the lack of chemical compatibility of some materials against the atmospheres to be tested, or because some functionalities aren't available.

Thermogravimetry is a common technique for the characterization of the thermal stability of materials, to understand their degradation process or to analyze their composition. Some materials like fluorides or oxyfluorides - which have interesting optical properties - release significant amounts of corrosive gases during their thermal stability testing. To fit to a customer request, a standard TGA was modified in order to stand the release of fluorine and hydrofluoric acid (up to 20% of initial sample mass). A further challenge was that the users wanted to test the material mass uptake under a flow of a gas containing up to 10% in mass of fluorine.

Key elements

Our skills in customized instrument design, our experience in materials compatibility at high temperature, and the organization of our R&D service being structured to manage specific projects have been applied for a long time in such cases.

The key point was the choice of the appropriate materials for all the TGA components that were supposed to be in contact with the fluorinated gases either at room or high temperature (up to 600°C)

Technical achievements

Gas flow / gas panel

All of the O-rings in the auxiliary gas panel and in the furnace potentially in contact with F₂ or HF were replaced by Kalrez ones. All of the aluminum based parts in contact with these gases were replaced by Hastelloy based equivalents.

The mass flowmeters were replaced by F₂ proofed ones. The whole gas circulation tubing was in stainless steel with silver brazing.

In order to avoid the condensation of HF (boiling point 19.5°C) all the potentially cold zones have been adapted to be heated up to 60°C with the help of a water circulation system.

Sample holder / Temperature measurement

A special type K, Inconel sleeved thermocouple was used for furnace temperature control.

The alumina sample holder was modified to replace platinum wire based handle and suspensions by Inconel based ones.

Going further

The TGA was meant to be coupled to a gas analyzer, which also had to be resistant to the mentioned gases. A corrosive atmosphere resistant mass spectrometer including a special vacuum pump and oil and a HF resistant gas cell was proposed. A study involving this setup was published in [1].

This type of customization of standard instrument for operations under corrosive atmosphere is increasingly popular. Do not hesitate to submit your project to us.

[1] *Journal of Fluorine Chemistry, Volume 134, February 2012, Pages 35–43*