

# Studying metals and alloys with long term stable TGA system

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## Abstract

To study the corrosion resistance of metals and alloys, an analysis system needs to be able to simulate the harsh environments to which these materials are subjected. The system must also collect data from metal and alloy samples at elevated temperatures over long periods of time (typically days). Thermogravimetric analyzers (TGAs) are well accepted systems for both corrosion studies and long-term elevated temperature studies. However, until now, TGAs could conduct either corrosion studies or long-term studies, but not both. This paper presents a TGA system specifically designed to satisfy the requirements of long-term corrosion resistance studies of metals and alloys.

## Introduction

Metals and alloys are used widely in a variety of industries for a spectrum of applications. Their properties are studied by many different kinds of instrumentation to ensure the materials can withstand the conditions they are subjected to during manufacture and while in use. Among many of their properties, one of the most important ones is the corrosion resistance to harsh environment.<sup>1,2,3,4</sup> To study the corrosion resistance, the best technique is to measure a sample's property in the desirable environment. Therefore, there are two basic requirements for the instrument.

- The ability to simulate the desired harsh environment while operating properly.
- The ability to obtain sample information such as heating or isotherm during testing.

Since such tests normally take well over 24 hours AND are conducted under elevated temperatures, the analysis instrument needs to be designed and built specifically to meet these rigorous testing conditions.



Figure 1. Picture of TGA VERSATHERM HM system.

In most cases, corrosion is accompanied by either sample weight gain or loss. From that point of view, a thermogravimetric analyzer (TGA) is the best solution for these studies. A TGA can heat the sample to the desired temperature and obtain weight information during the entire process. The question lies in the fact that the system has to satisfy the above requirements.

In this paper, such a specially engineered TGA system is presented to demonstrate its capabilities to meet those stringent requirements.

## Experimental Set-Up

A Thermo Scientific TGA VERSATHERM HM system from Thermo Fisher Scientific was used for the studies, Figure 1. The TGA system features 100 g capacity, 35 mL sample volume, and 1  $\mu$ g sensitivity. The instrument has built-in reaction gas flow controlling and mixing capabilities.

Since previous papers and presentations had covered the corrosion resistant part of the Thermo Scientific's TGA systems,<sup>5,6</sup> this paper's focus is on the capability of these systems to operate for long periods of time.

Three different sets of metal samples with different corrosion resistance ratings (metal #1, #2, and an alloy

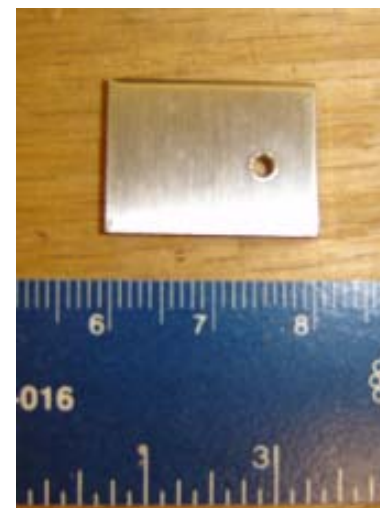


Figure 2. Typical sample piece.

sample) were analyzed with the use of the system. For each set, sample pieces were cut into the same size coupon, and a hole was drilled in each so that it could be hung directly within the system, Figure 2. Hanging the sample in this way provided the greatest exposure of the sample's surface to the reaction environment.

The samples were heated from room temperature to the desired isothermal temperature and then held at a constant temperature for approximately 24 hours. To simulate typical corrosion (oxidation), dry air with a flow rate of 80 mL/min was used as the reaction gas.

Throughout this article, only original data points are reported and presented.

## Results and discussion

In order to make sure that the system was working properly, two runs were performed. One was performed with a 3 g alumina sample tested at under 800 °C for 1000 minutes, the other was performed with a 4.3 g of CHROMEL® sample tested at under 800 °C in air for 40 hours, Figures 3 and 4.

Figure 3 illustrates that the system showed a very long-term stable (flat) baseline, even under high temperature. This is a critical requirement for such studies, since this is the zero line for the samples tested under isothermal conditions. Figure 4 confirms that the system performed as expected since it shows a typical weight gain curve for these types of samples.

Figures 5 and 6 show overlaid curves for the two metal samples, #1 and #2, which were held at different temperatures for 24 hours. For each sample, it's apparent that the higher the temperature, the greater the weight gained over time. This is especially clear in Figure 6, where the very small difference between 600 °C and 850 °C runs is quite obvious. It can also be seen from Figures 5 and 6 that metal samples #1 showed much faster "corrosion" under the same temperature. Under 1100 °C for 24 hours, the amount of weight gain for metal sample #2 was only about 0.074%, while metal sample #1 showed a gain about 1.972%. To demonstrate what the TGA system can do perform over time peri-

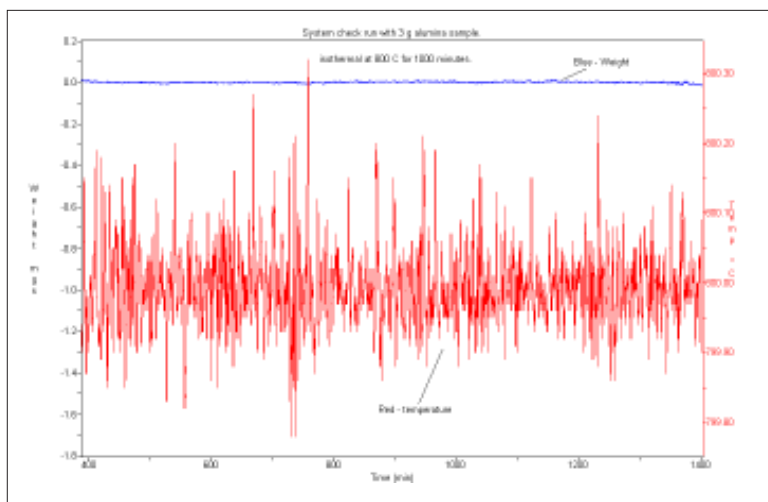


Figure 3. System check run with 3 g alumina sample.

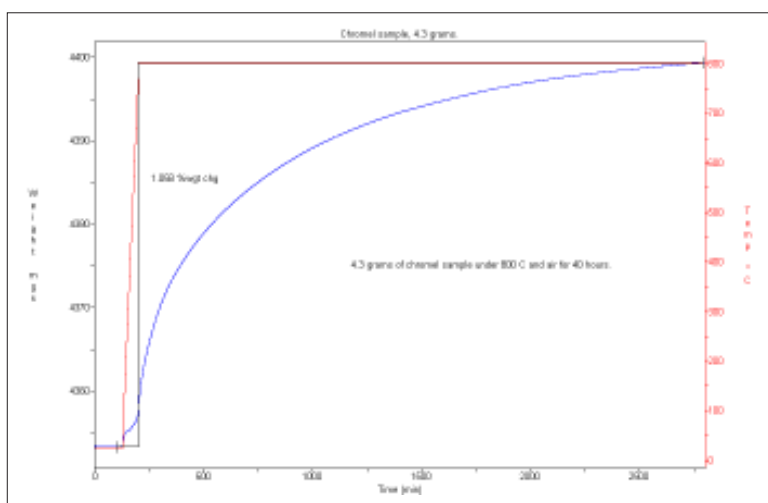


Figure 4. CHROMEL® sample run curve under 800 °C for 40 hours.

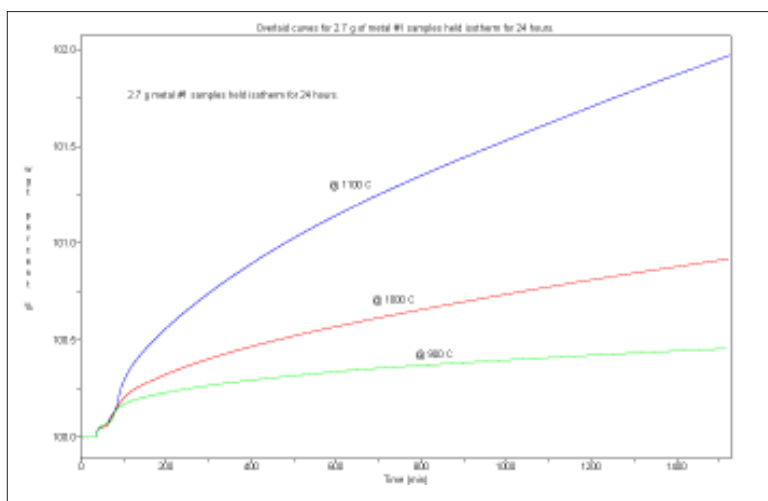


Figure 5. Overlaid curves for metal sample #1.

ods longer than 24 hours, an alloy sample was held isotherm for 5 days. Results are shown in Figure 7.

Under the two isothermal temperatures, 1100 °C and 850 °C, the sample showed a very smooth curve over 5 days, especially considering the very small weight change at 850 °C (about 0.049%). This result, in combination with the curve derived in

Figure 3, clearly demonstrates that the system is capable of performing long runs.

## Conclusions

The presented system, Thermo Scientific's TGA system, is capable of monitoring the metal sample's corrosion resistance. With the special designed hardware and especially the long term stable balance, the system can tell the small differences among the samples, over LONG time (more than 24 hours or longer).

Based upon the data obtained, further kinetics analysis can be obtained to understand more about the kinetics of the samples

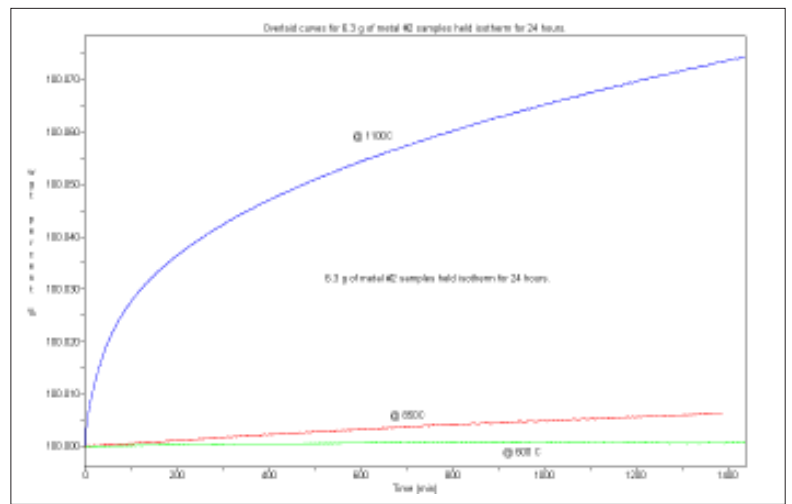


Figure 6. Overlaid curves for metal sample #2.

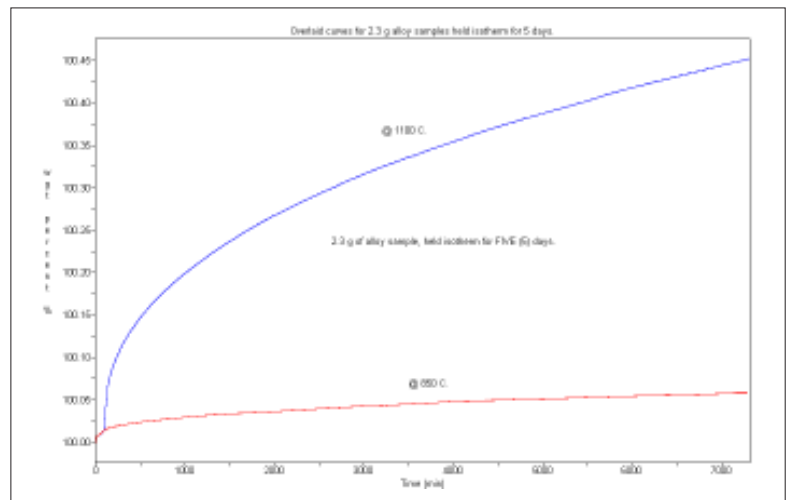


Figure 7. Overlaid curves for alloy sample held isotherm for 5 days.

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