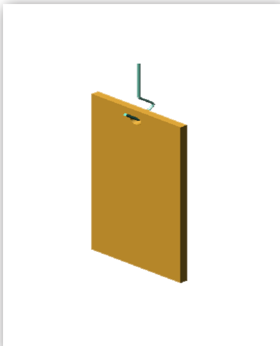
Cyclic oxidation of metallic alloys by symmetrical TGA

INTRODUCTION

Metallic alloys are frequently subjected to combined environmental attack and mechanical stresses during their actual service life. Their resistance to this complex loading relies partly on their ability to form a protective oxide scale, i.e. an oxide layer with low growth kinetics and high adherence to the alloy. The nature and kinetics of the growth of the oxide layer is , often studied using isothermal laboratory tests. When testing the resistance to oxidation of high-temperature materials, the cyclic-oxidation test is used as a reference because it integrates isothermal oxidation kinetics, oxide-scale adherence, mechanical stresses, metallic alloy and oxide creep and the evolution of these properties with time, for conditions close to the actual conditions of use.

Description

THEMYS DUO is a symmetrical analyzer with a top loading balance.
This configuration allows to hang the metallic sample (sheet, disk, ribbon…) to the balance without using a crucible in order to increase the contact surface for the gas interaction.
Different hanging systems are availbale according to the shape of the probe.



Furnace ***Pt hanging device***

EXPERIMENT

A NiAl single crystal was oxidized in a THEMYS DUO. The experiment consisted of 168 cycles including dwell times of 5 min at 1150°C and 30 min dwell times at 150°C. Heating was at a constant rate of 60°C/min and cooling was controlled at 20°C/min down to 150°C.
The experiment was conduced in flowing oxygen. The obtained Net Mass Gain recording, without any data processing, is given in Fig. 2. This curve includes 12,000 data points. The usual shape of cyclic oxidation data combining mass gain due to oxidation and mass loss due to spalling is obtained.

RESULTS AND CONCLUSION

The recorded signal is detailed in Fig. 3 which is an enlargement of Fig. 2 during two cycles (148th and 149th) selected at random for illustration. On this enlargement, it is seen that the mass gain during the high-temperature dwell is about 10 mg at 1150°C.

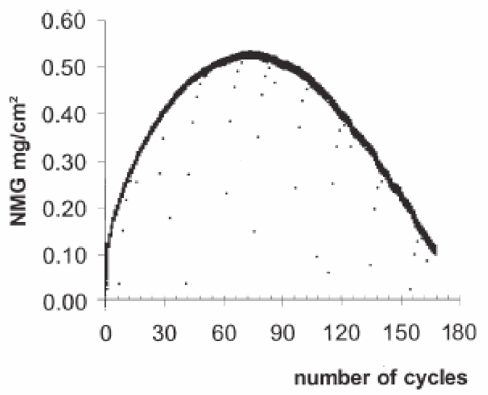


Fig. 2. Experimental Net Mass Gain of a NiAl single crystal (100) oxidized during 168 cycles of 45 min at 1150°C in flowing oxygen. Data set contains 12,000 points recorded with a SETARAM TAG24s thermobalance.

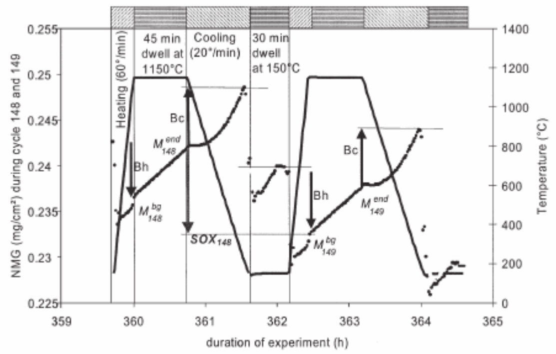
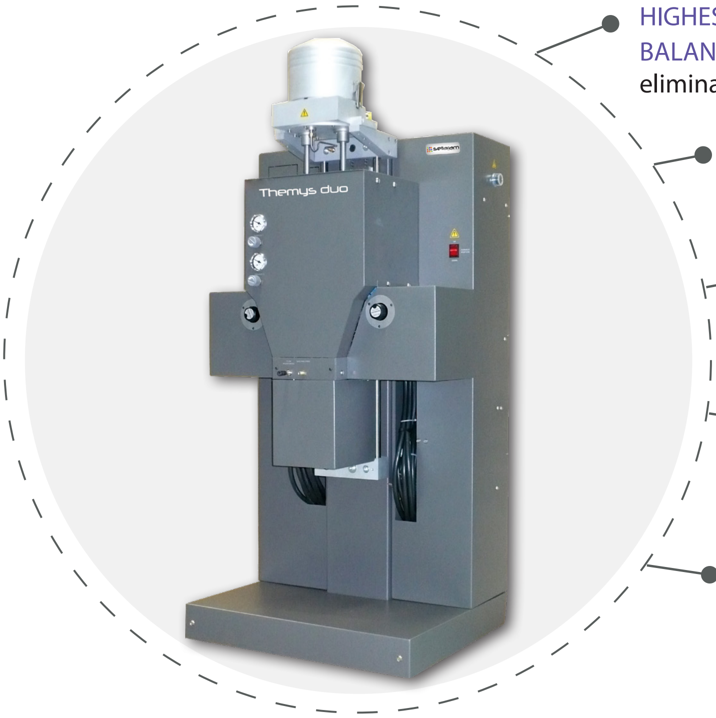


Fig. 3. Enlargement of Fig. 2 with superimposition of the recorded temperature during 2 cycles. Definition of the mass gain due to oxidation ($\Delta M_{148} = (M_{148}^{end} - M_{148}^{bg})$), mass of spalled oxide ($SOX_{148} = (M_{149}^{bg} - M_{148}^{end})$), buoyancy effect during heating (Bh) and cooling (Bc).

Reference: D. Monceau and D. Poquillon, Oxidation of Metals, Vol. 61, Nos. 1/2, Feb. 2004

INSTRUMENT

THEMYS DUO



HIGHEST ACCURACY WITH ITS HANG-DOWN SYMMETRICAL BEAM BALANCE
eliminate drift & buoyancy effect, improve gas/sample interaction

ULTRA-HIGH TEMPERATURE CAPABILITY
to ***1750°C*** with a single furnace

MODULAR ADAPTIONS ALLOWING
TGA only, DTA only, TG-DTA up to 1750°C, DSC only and TG-DSC up to 1600°C all in one instrument

ACCURATE AND SENSITIVE ULTRA-HIGH TEMPERATURE
heat flow measurement with Tri- Couple DTA technology

EXTERNAL COUPLING CAPABILITY
designed for evolved gas analyzers (FTIR, MS, GCMS, MSFTIR, or FTIR-GCMS)