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Secretariat: CMSI

Corrosion of Metals and Alloys – test method for high temperature corrosion testing of metallic materials by thermogravimetry under isothermal or cyclic conditions

WD/stage

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Foreword

A boiler plate text will be inserted by ISO CS from DIS onwards.

List here any additional parts in the series.

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Introduction

Oxidation and corrosion tests at high temperature on metallic materials are often performed according to the isothermal or to the cyclic exposure methods specified in ISO 21608 and ISO 13573 respectively. These methods rely on measuring the mass change at the end of the exposures. An alternative approach is to employ a thermogravimetric method which enables a continuous monitoring and recording of mass as a function of time under isothermal or cyclic operation.

~~For~~At isothermal exposure, the thermogravimetric analysis allows the measurement of mass gain due to oxidation or corrosion ~~and of, or the~~ mass loss due to evaporation. The corresponding kinetics can be assessed. Moreover, it allows the detection of a mass change due to the loss of a part of the oxide scale during the high temperature dwell or during cooling. Re-oxidation~~s~~ subsequent to the formation of cracks in the oxide scale can also be detected.

~~During~~~~Under~~ thermal cycling conditions, the mass change of the sample can be measured ~~as well~~. The corresponding kinetics can be assessed thereby distinguishing between ~~the~~ mass gain due to oxidation and ~~the~~ mass loss due to spalling. The occurrence of breakaway oxidation can be also precisely identified, and the test is fully automated without ~~having the need~~ to take the samples out ~~of~~ the corrosive atmosphere for mass measurements.

The main areas of applications~~s~~ are the following:

- The test method describes the general conditions of analysis for materials such as pure metals and metallic alloys using ~~the~~ thermogravimetric techniques.
- The thermogravimetric test can be used in the isothermal mode (mass variation versus time at a given temperature) or in the cyclic mode (mass variation versus time according to defined thermal cycles).

Corrosion of Metals and Alloys — Thermogravimetric (continuous measurement) method for isothermal and cyclic exposure at high temperature under corrosive conditions

1 Scope

This international standard specifies the thermogravimetric method (continuous measurement) for isothermal and cyclic exposure of metals and metallic alloys at high temperature under corrosive conditions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 13573, *Corrosion of metals and alloys — Test method for thermal cycling exposure testing under high temperature corrosion conditions for metallic materials*

ISO 21608, *Corrosion of metals and alloys — Test method for isothermal-exposure oxidation testing under high-temperature corrosion conditions for metallic materials*

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply:

3.1 Thermogravimetry (TG)

Technique in which the mass of a test piece is measured with respect to temperature or time, the test piece being heated according to a given temperature program

3.2 TG curve

Thermogravimetric curve obtained by plotting the mass of the test piece divided by its surface area as the ordinate (y axis) and the elapsed time t as the abscissa (x axis) (Figure A.1)

3.3 Sample

Small part or fraction of a material coming from a series of products, designed to represent the whole amount

3.4 Test piece

Full product or unique fraction, taken from the sample and used for the test

3.5 Buoyancy effect

Apparent variation of the sample mass, related to the pressure effect on its volume in a given atmosphere.

4 Test method

4.1 Principle

The test piece is heated at a constant and controlled heating rate (at least 60 °C/min)^[dm1] up to an elevated temperature T_i in a controlled gaseous environment (inert, reducing, oxidative, or corrosive - dry or humid- atmosphere). If the reaction between the test piece and the atmosphere can be fully prevented during the heating (oxidation of pure metals with low stability oxides such as Cu, Ni, or Co), it is possible to use an inert gas during heating prior to the introduction of the reacting gas when the temperature T_i is reached. In all other cases, the atmosphere is established before the heating of the sample.

For an isothermal test, the temperature T_i is maintained constant during the test duration (Figure A.2).

In cyclic mode, the temperature is first increased to a temperature T_j at a constant heating rate. T_j is maintained constant during a time to be defined (cold dwell). The temperature is then increased at a given heating rate ≥ 60 °C/min to the temperature T_i and maintained constant during a time to be defined (hot dwell). The final stage is a cooling from T_i to T_j with a controlled initial cooling rate at least equal to 60 °C/min. The temperature cycle can be repeated a number of times to be defined (Figure A.2).

At the end of either procedure, the test piece is cooled down to room temperature and the mass variation of the test piece is measured versus time t during the duration of the test and recorded as a TG curve.

~~The thermogravimetric data can be used for the development and the control of industrial processes, for the development of materials, for the determination of their properties in respect to resistance against oxidation and corrosion in controlled atmospheres.~~

4.2 Test pieces

The test pieces are in solid form and may be of different geometries: sheets, films, rectangular cuboid, cylinders, and discs. The dimensions of the test piece shall be restricted to a value less than the diameter of the furnace used for the test, avoiding contact with the walls of the furnace.

The test pieces shall be finished by machining so that the strata affected by cutting do not remain.

The final finishing of the surface of the test pieces shall be performed with abrasives with mean particle diameter of approximately 15 µm. This can be achieved by the use of abrasives according to Table 1.

If another surface finish is required by the parties involved, the surface finish condition shall be described.

Table 1 – Designation and mean diameter of particles of coated abrasives according to regional standards

Standard	Designation	Mean diameter µm	Region
FEPA ^a 43-1984 R:1993, <i>Grit Sizes for Coated Abrasives</i>	P1200	15,3 ± 1,0	Europe

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ISO 6344-3:1998, <i>Coated abrasives – Grain size analysis – Part 3: Determination of grain size distribution of microgrits P240 to P2500</i>			
JIS R6001-87, <i>Bonded abrasive grain sizes</i>	#1000	15,5 ± 1,0	Japan
ANSI B74.12-92, <i>Specifications for the Size of Abrasive Grain – Grinding Wheels, Polishing and General Industrial Uses</i>	600	16,0	America
^a Federation of European Producers of abrasives			

Sharp edges of test pieces may give anomalous behavior. These shall be slightly rounded during the final stages of test piece preparation.

The surface of the test pieces shall not be deformed by marking, stamping, or notching. However holes for test piece support and or reference marking are permissible.

Where holes are used for test piece support they shall be drilled prior to final finishing or application of coatings. These have to be taken into account when calculating the surface area.

The dimensions of the test pieces shall be measured prior to exposure at a minimum of three positions for each dimension with a precision of ±0,02 mm by means of the measuring instruments specified in ISO 3611 and ISO 13385-1.

The test pieces shall be dried after degreasing by ultrasonic cleaning using iso-propanol or ethanol.

If it is suspected that specimens may adsorb significant amounts of atmospheric contaminants such as water, it is recommended that the cleaned test pieces are stored in a desiccator prior to weighing and exposure.

A hole is machined in the top part of the test piece to allow it to be suspended from the balance (Figure A.3). Platinum wire or an intermediary alumina piece shall be used to suspend the test piece. When it is not possible to machine a hole in the test piece (coated test piece for example), a cradle shall be used to hang the test piece (Figure A.3). If a holder is used, it shall be adapted to the shape and the size of the test piece. It should not limit the access of the reactive gas to the sample or prevent the spalling spalled parts of the sample to fall from falling down.

Note: the vaporization of Pt as a volatile oxide can be significant for-at temperatures higher than 1_100_°C in oxygen-rich atmospheres. In this case, alumina shall be preferred to platinum. Moreover depending on the specimen materials the reaction with Pt (eutectic formation for example) has to be carefully addressed before thermogravimetry test.^[DM2]

5 Test apparatus

5.1 Thermobalance

The thermobalance shall be able to measure the mass variation with an accuracy equal to 10⁻³ of the expected total mass variation. For every condition of temperature and gas flow a blank test shall be performed with an inert sample. The resulting drift of the balance under isothermal conditions shall be at least 100 times less than the instantaneous rate of mass loss or gain over the analysed test period.

Note: It is recommended to install the thermobalance in a controlled temperature room. Depending on the thermobalance and the accuracy that is required for the measurement, it may be necessary to set up the instrument on a stable-vibration-reduced table.

The thermobalance shall allow a constant flow of gas around the test piece in order to allow a homogeneous interaction on the whole surface of the test piece in the reactive gaseous atmosphere.

Issues with buoyancy and convection effects in the gas flow **can be** resolved through the use of a symmetrical thermobalance built with two identical furnaces (Figure A.4). In this case the test piece is introduced in the measuring furnace and a reference piece with identical dimensions, but inert in the given temperature range and gas, is introduced to the reference furnace. The gas flow rates are adjusted in the two furnaces to be equal. With such a device, as the buoyancy effect is identical in both furnaces, the TG curve does not show a significant deviation.

5.2 Furnace

The furnace shall be able to achieve **controlled** [dm3] heating and cooling cycles. The furnace shall be characterized at the exposure temperature prior to the testing to determine the length of the isothermal zone inside the furnace. This can be achieved through the use of an independent moveable thermocouple.

The temperature shall be controlled such that the temperature of the test piece is kept within the permissible range given in Table 2.

Table 2 – Permissible tolerance of temperature of test pieces

Temperature range, °C	≤ 300	300-600	600-800	800-1_000	1_000-1_200	> 1_200
Temperature tolerance, °C	±2	±3	±4	±5	±7	By agreement

5.3 Temperature probe

The temperature shall be measured by a suitable device, which is separate from the thermocouple used for control [DM4], according to ASTM E633-00. Thermocouples of type K (Ni/Cr - NiAl) up to 800 °C, type S (10_%Rh/Pt - Pt) and type R (Pt/13_%Rh - Pt) up to 1_100 °C or type B (Pt/30_%Rh - Pt/6_%Rh) above 1_100 °C are preferred. The thermocouple shall be capable of confirming the temperature of the test piece to be within the range given in Table 2.

A thermocouple should be positioned close to the test piece surface and **must-shall** be calibrated according to the paragraph 6.2. [DM5]

The type of thermocouple used shall be noted and included in the test report. The temperature probe shall be resistant to the oxidizing or corrosive atmosphere in dry or humid conditions. Thermocouple sheaths shall be used to protect the thermocouple wires. The thermocouple sheaths shall withstand fully the test temperature and environment.

5.4 Data acquisition device

A data acquisition device is required in order to achieve the automatic acquisition of the mass of the test piece and **of** the temperature and/or time and graphically represent the mass variation versus temperature or time. It shall be able to record at least 1_000 data points over the test duration. It shall have a sufficient acquisition rate to enable accurate monitoring of the TG curve.

5.5 Test piece holder

The test piece holder is attached to the balance and located in the furnace. The test piece can also be directly hung to the balance in order to have the whole surface in contact with the reactive atmosphere

(Figure A.3). The test piece holder shall be made of a material that does not react with the given atmosphere or the material under investigation in the whole temperature range defined for the test.

5.6 Reactive gas and gas flow

The gas supply system shall be capable of supplying the test gases at a constant rate to the test piece chamber.

When a humidifying regulator is used it shall be capable of adjusting to the desired humidity. Deionized water of a conductivity less than $1 \mu\text{S cm}^{-1}$ shall be used, unless otherwise specified.

The space between humidifying regulator and test piece chamber shall be kept above the dew point in order to avoid condensation.

The gas flow shall be high enough to ensure that no significant depletion of reaction species will occur. At the same time the gas flow shall be slow enough to allow the gas mixture to preheat and in some applications to reach equilibrium. The flow rate of the test gas shall be sufficient to ensure complete replacement of the test gas within the test chamber at least three times an hour.^[dm6]

The gas flow shall be monitored by a gas flow meter. The flow meter shall be located as close as practicable to the inlet of the test piece chamber except where a humidifying regulator is used, in which case it shall be located upstream to the humidifier.

For testing in air a specific humidity (mass fraction of water in air) of $\sim 20 \text{ g/kg}$ is recommended. This corresponds to a relative humidity of 100% at 25°C (dew point) and is easy to obtain by bubbling through a water bath of 25°C .

If any other humidity is employed, it shall be agreed between the parties concerned.

In the case that the gas is humidified the water vapor content shall be measured. This can be achieved by e.g. the use of a hygrometer before the test piece chamber or by measuring the amount of water after condensation of the exhaust gases or by measuring the water consumption of the humidifier over the course of the experiment.

WARNING: Before any use of reactive gases, it is required to check that the thermobalance is well adapted to this type of measurements ~~for~~ regarding the safety of the operators and that there is no risk of corrosion or degradation of parts of the instrument, especially metallic parts (thermocouple, balance, and furnace).

5.7 Protection of the thermobalance

If an inert gas is used to protect the metallic parts of the balance during the test, it shall be ensured that the atmosphere surrounding the sample is not modified.

6 Calibration

6.1 Mass calibration

Calibration of the balance shall be performed using standard masses over an appropriate range without gas flow in the thermobalance (in order to prevent any buoyancy and/or convection effects). Calibration shall be performed at least once a year or whenever significant changes have occurred to the equipment.

6.2 Temperature calibration

Calibration of thermocouples shall be performed in accordance with ASTM E220-02, ASTM E230-03, or ASTM E1350-97. A representative thermocouple taken from the batch of wire may be calibrated.

It is recommended that thermocouples are recalibrated annually or at the beginning and the end of each experiment if there is uncertainty about thermocouple stability.

In a thermobalance, it is possible to run an in-situ calibration in the same atmosphere, the same flow rate, and with the same temperature heating rate as used during the test itself:

a) Select at least two standard reference materials among the 5 GM761 magnetic materials listed in table A.1 that exhibit a Curie temperature in the vicinity of the temperature range to be investigated.

b) Start the heating at the same heating rate than the one used for the test itself and run the calibration on the basis of the onset temperature T_A , the mid point temperature T_C and the end set temperature T_B for the transition corresponding to the Curie temperature (Figure A.5).

NOTE: The production of a magnetic field through a magnet set closed to the furnace is needed to measure the magnetic transition that occurs with the measurement of a force variation (equivalent to a mass) on the TG curve.[17]

7 Procedure

7.1 General

The test method shall be adapted to the instrument and to the experimental conditions. Two modes are described: the test in isothermal mode and the test in cyclic mode.

A preliminary test (called blank test) shall be run using a test piece of identical volume (preferably with the same dimensions) made of an inert material (with respects to the reactive atmosphere), at the same temperature, heating rate, and with the same gas flow rate as used for the test itself in order to measure the apparent mass variation due to buoyancy and /or convection effects.

7.2 Isothermal test

- a) The mass of the test pieces shall be determined prior to exposure. At least two individual measurements shall be made for each test piece with a precision of 0,02 mg. The maximum difference between the measurements shall not exceed $\pm 0,05$ mg.
- b) Install the test piece in the holder (if used) or hang the test piece to the balance suspension.
- c) Close the furnace and zero the balance.
- d) Select the gas flow rate, start the gas circulation.[DM8]
- e) Define the temperature profile to be run. The program must include the initial and final temperatures, the temperature scanning rates between the two temperatures, and the duration of the isothermal level (hot dwell time) at the final temperature.
- f) Start the program and record the thermogravimetric curve (Figure A.2).
- g) Weigh the test piece at room temperature without gas flow, in the same conditions as in c).
- h) Remove the test piece from the thermobalance and weigh it in the same conditions as in a).

7.3 Cyclic test

Run the operations defined in 7.2 a), b), c), and d).

- e) In order to prepare the temperature cycle to run in the cyclic mode, the program shall include the T_i and T_j temperatures, the temperature scanning rates (heating and cooling for the cycle), the level durations Δt_{hot} , Δt_{cold} respectively at T_i and T_j , [and](#) the number of cycles to be run.

Two modes are available:

— Mode 1: Start with a cold dwell time at T_i (see Figure A.2)

— Mode 2: Start with a hot dwell time at T_i .

Choose the frequency of acquisition with the program in order to record at least two specific data points at each cycle: the mass at the beginning of the high temperature dwell, $m(t_i)$, and the mass at the end of the high temperature dwell $m(t_i + \Delta t_{\text{hot}})$.

Run the operations defined in 7.2 f) to h).

8 Expression of results

8.1 Graphical presentation

Present the obtained thermogravimetric data as a curve giving $\Delta M/S$ (S = initial test piece surface area) versus time, including the temperature profile used for the test.

Note: The curve of the derivative of $\Delta M/S$ ~~with the time~~ versus time can be added to the plot.

9 Analysis of the mass change

9.1 General

Note: The objective of the analysis is to convert the raw data consisting in mass change versus time (see 8.1) into oxidation kinetics parameters.

9.2 Isothermal exposure

Note: Reference can be made to ISO 21608 isothermal testing (discontinuous measurement). Nevertheless, because thermogravimetry allows much more precise measurements with more data points, a specific analysis can be done.

An initial check of the results shall be performed by plotting $\Delta M/S$ (S = test piece surface area) versus time for the high temperature dwell ~~shall be plotted~~.

If mass loss occurs from the start of the exposure or after a period of mass gain, evaporation of the oxide scale, of the metal, or of the corrosion product should be considered before proceeding further. The occurrence of evaporation should be proven before further analysis of the oxidation kinetics.

For analysis of the kinetics the data are plotted in a log-log plot: $\log(\Delta M/S)$ versus $\log(t)$ for the high temperature dwell. If a linear regression is found between time 0 and time t^* , the parameters (k, n) of the law $\Delta M/S = k.t^n$ can be given as an interpolation between 0 and t^* . The log-log plot is used for interpolation only and before any breakaway or change of slope. The results are expressed as follows:

“Between time 0 and t^* , the oxidation kinetics can be interpolated by a power law $\Delta M/S = k.t^n$ with the following values of k , n and t^* .”

If no linear fit can be found using the log-log plot, no standard analysis is provided.

For some values of the coefficient “ n ”, a more precise analysis of the kinetics can be provided as follows, [and the kinetics parameters found with the following analysis can be used for extrapolations:](#)

Linear kinetics - for a value of “ n ” close to 1 (between 0.9 and 1.1), a linear regression of $\Delta M/S = f(t)$ is performed and the result is expressed as follow:

“Between time 0 and t^* , the oxidation kinetics are linear $\Delta M/S = k_l.t$ with k_l = value expressed in $\text{mg}/\text{cm}^2/\text{s}$ (k_l is called the linear rate constant).”

Parabolic kinetics - for a value of “ n ” close to 0.5 (between 0.35 and 0.65), the user plots the data in a parabolic plot, i.e. $\Delta M/S$ versus \sqrt{t} , and applies a linear regression. The result is then expressed as follows:

“Between time 0 and t^* , the oxidation kinetics are parabolic $\Delta M/S = \sqrt{k_p.t}$ with k_p = value expressed in $\text{mg}^2/\text{cm}^4/\text{s}$. (k_p is called the parabolic rate constant).”

In order to get an improved fit or to perform subsequent extrapolations, the data may be represented in a plot of $\Delta M/S - t$ and the “complete parabolic law” may be fitted, i.e.:

$$t = A + B \Delta M/S + C(\Delta M/S)^2$$

between time t_i and the end of the experiment. t_i is chosen in order to obtain a good quality fit. If a good fit is obtained, the result is expressed as:

“After [an incubation time a transient regime of duration \$t_i\$](#) , the oxidation kinetics follows a complete parabolic law: $t = A + B \Delta M/S + C (\Delta M/S)^2$ with $k_p = 1/C$ is expressed in $\text{mg}^2/\text{cm}^4/\text{s}$. (k_p is called the pure parabolic rate constant corresponding to the final stationary state regime of oxidation kinetics).”

9.3 Thermal cycling exposure

Note: Reference can be made to ISO 13573 thermocyclic exposure (discontinuous measurement). Nevertheless, because thermogravimetry allows more precise measurements with more data points, and because crucibles are not used, a more specific analysis can be done.

A graph of $\Delta M/S$ (S = test piece surface area) versus time is plotted for the entire duration of thermal cycling.

A Net Mass Change (NMC) plot versus time or versus the number of cycles is obtained by keeping only one data point $\Delta M/S$ for each thermal cycle. Because of buoyancy, these data points are taken always at the same temperature. It is recommended to take the data point $\Delta M/S$ at the end of each high temperature dwell. This set of data can be analyzed in the same manner as a result of discontinuous measurements using the ISO 13573 thermocyclic exposure (discontinuous measurement) [method](#).

A Gross Mass Gain (GMG) plot versus time or versus the number of cycles is obtained by keeping two data points $\Delta M/S$ for each thermal cycle. Because of buoyancy, these data points are taken always at the same temperature. It is recommended to keep the data point $M_i^{\text{beg}} = (\Delta M/S)$ at the beginning of each high temperature dwell “ i ” and the data point $M_i^{\text{end}} = (\Delta M/S)$ at the end of each high temperature dwell “ i ”. Using these notations, the NMC and GMG at the cycle “ i ” are calculated as:

$$NMG_i = M_i^{\text{end}}$$

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And

$$GMG_i = \sum_{j=1}^i (M_j^{end} - M_j^{bg})$$

This set of data can be analyzed as a result of discontinuous measurements using the ISO 13573 thermocyclic exposure (discontinuous measurement) [method](#), or [it](#) can be analyzed in more details using the following note.

Note: Using this technique, from the knowledge of the Net Mass Change and the Gross Mass Gain at each cycle, it is possible to calculate the mass of spalled oxide, the mass of consumed metal, [and](#) the kinetics of reduction of the sample thickness. Assuming parabolic oxidation kinetics during the high temperature dwells, it is also possible to calculate and plot the parabolic rate constant " k_p " and the fraction of the oxide scale which spalls at each cycle " P " as a function of the cycle number. A parametric plot in a $\log(k_p)$ - $\log(P)$ diagram can be built in order to evaluate the resistance of the material to cyclic oxidation. [\[no scientific reference?\]](#)

10 Report

10.1 Matters to be described

10.1 [The following data, where known, shall be included in the report on the test results:](#)

10.1.1 [The following data, where known, shall be included in the report on the test results:](#) Test material

- a) Manufacturer
- b) Name of material (Manufacturer designation; ASTM, DIN etc.)
- c) Grade or symbol
- d) Heat number / Batch number
- e) Chemical composition (analysis technique)
- f) Processing condition
- g) Heat treatment condition
- h) Microstructure of the material determined according to ASTM E3-01 and etching according to ASTM E407-07e1

10.1.2 Test piece

- a) Designation of test piece
- b) Sampling conditions of the test piece from raw material (Crystallographic orientation, rolling direction etc.)
- c) Dimensions, shape and surface area of test piece (including the surface area of the hole if used)
- d) Surface finish condition of test piece
- e) Degreasing and cleaning method of test piece
- f) Method of test piece support

g) Initial mass

10.1.3 Testing environments

a) Type of the thermobalance used, manufacturer

f) Diameter of the furnace tube

g) Dimensions and material of the test piece holder (if used)

h) Type and location of the temperature probe

i) Atmosphere and gas flow rate used for the test, as well as the gas velocity towards the sample:

— Specify the times and nature of the performed gas switching;

— Specify the gas purity;

— Specify the total pressure in the furnace;

j) For the isothermal test: Temperature scanning rates (heating and cooling), duration of the isothermal level, temperatures of hot dwell T_i

For the cyclic test: Temperature scanning rates (heating and cooling), duration of the isothermal level, temperatures of hot dwell T_i and cold dwell T_j , durations of the isothermal levels at T_i and T_j , the number of cycles

k) Standard reference material used for the temperature calibration (if any)

l) Any observation relative to the instrument, the test conditions or the behaviour of the test piece;

10.1.4 Test results

a) Reference to the standard;

b) Date of the test;

c) Calculated net mass variation during the entire test (difference between mass after and before the thermogravimetric test)

d) Numerical file of the raw data (t , T , mass change);

e) TG curve (net mass change per area in mg cm^{-2} vs. time)

f) Results of the data analysis as described in §8.19.2 (isothermal) or §8.29.3 (cyclic);

g) In case of a more detailed analysis of the cyclic test (§8.29.3)-

- Plot of gross and net mass change per area in mg cm^{-2} vs. time

- Plot of the amount of spalled scale in mg cm^{-2} vs. time

h) Image of appearance after testing

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- | j) Image of cross section including the surface layer of the metallographic section of test piece after testing. The chosen magnifications must clearly show the extent of the total attack in a single micrograph
- | k) Results of any metallographic investigations performed [DM9]

Annex A (informative)

Standard reference materials for the temperature calibration

A.1 NIST GM761 reference materials for the magnetic transition used with the thermogravimetric analyzers

T_A is the onset temperature, T_B is the endset temperature, T_C is the mid-point temperature for the transition corresponding to the Curie temperature

Table A.1 – NIST GM761 reference materials for the magnetic transition used with the thermogravimetric analyzers (NIST: National Institute of Standards and Technology)

Reference material	T_A (°C)	T_C (°C)	T_B (°C)
Permanorm 3	253	259	267
Nickel	351	353	355
Mumétal	378	382	386
Permanorm 5	451	455	458
Trafoperm	749	750	751

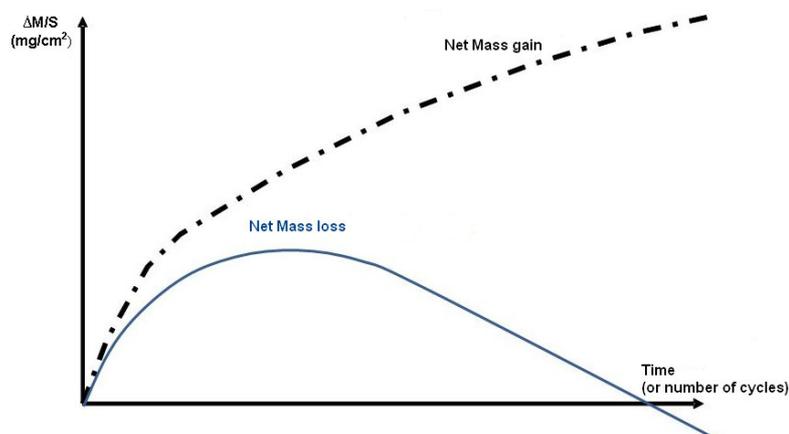


Figure A.1 — Two examples of TG curve with a net mass gain or a net mass loss after an initial mass gain (Net Mass Change NMC versus time or versus number of cycles)

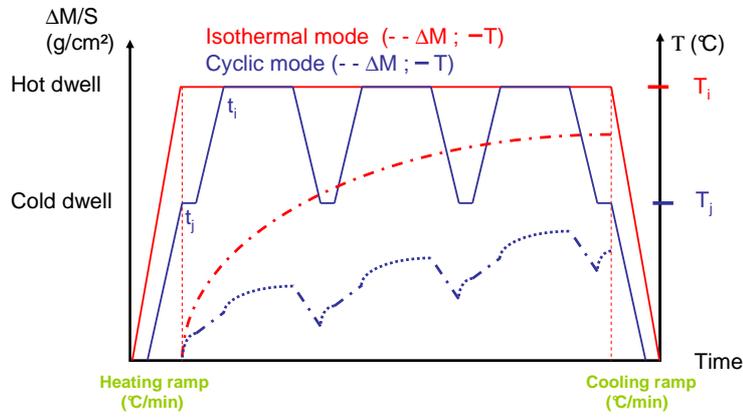


Figure A.2 — Temperature and Net Mass Change as a function of time during a thermogravimetric test in isothermal or cyclic mode

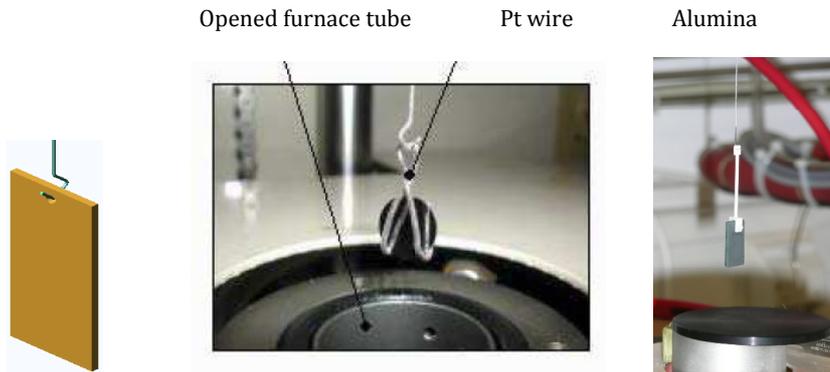


Figure A.3 — Test pieces hung to the balance a) using a hole drilled in the specimen b) using a Pt wire to hold the specimen c) using a hole drilled in the sample and an alumina sample holder

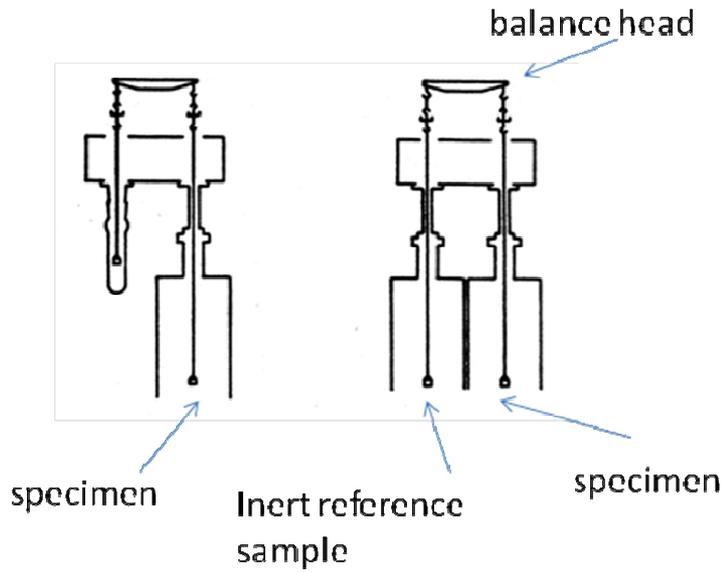


Figure A.4 — Thermobalance with one furnace and symmetrical thermobalance (two

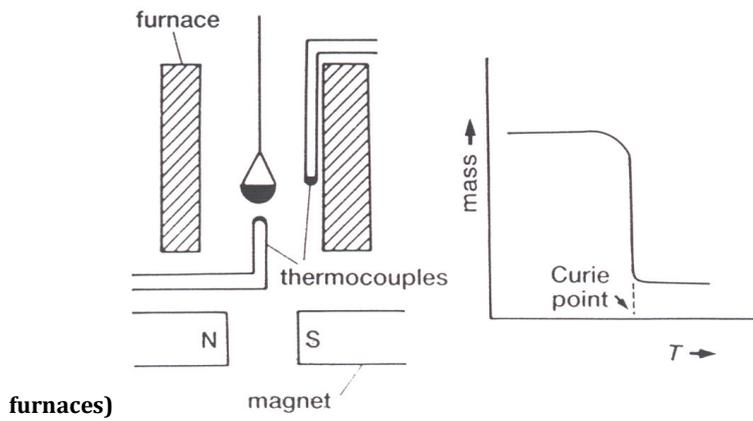


Figure A.5 — Temperature calibration with the Curie point method