Corrosion of Metals and Alloys - Test method for Thermal Cycling Exposure Testing under High Temperature Corrosion Conditions for Metallic Materials

18th November 2005
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Corrosion of Metals and Alloys - Test method for Thermal Cycling Exposure Testing under High Temperature Corrosion Conditions for Metallic Materials

1 Scope

This international standard describes the methodology for Thermal Cycling Corrosion Testing (known as cyclic oxidation testing) of metallic materials in gaseous atmospheres between ambient and elevated temperatures (series of measurements on a single test piece with repeated, regular and controlled temperature cycles). It also may be applicable to other materials with some modifications. Tests with ultra short dwell times in the range of minutes or seconds are outside the scope of this standard.

2 Normative References


ISO 8044 Corrosion of Metals and Alloys - Terms and Definitions

ASTM E633-00 (Standard Guide for Use of Thermocouples in Creep and Stress-Rupture Testing to 1800°F (1000°C) in Air)

ASTM E220-02 (Standard method for calibration of thermocouples by comparison techniques)

ASTM E230-02 (Standard temperature-electromotive forces tables for standardized thermocouples)

ASTM E1350-97 (Standard test method for testing sheathed thermocouples prior to, during and after installation)

FEPA 43-1984 R 1993: Grit Sizes for Coated Abrasives

ISO 6344 Coated abrasives - Grain size analysis

JIS R6001-87: Bonded abrasive grain sizes

ANSI B74.12-92 - Specifications for the Size of Abrasive Grain -- Grinding Wheels, Polishing and General Industrial Uses


ISO/TC 156 N XXX – Corrosion of Metals and Alloys – Method for Metallographic Examination of Samples after Exposure to High Temperature Corrosive Environments
3 Definition

The definition of the main terms used in this Standard shall be as follows:

3.1 Scale
Surface film and corrosion products produced on the surface of test piece by high temperature corrosion.

3.2 Adherent scale
Scale adhering to the test piece even after cooling.

3.3 Spalled scale
Scale flaked from the test piece.

3.4 Metal loss
Cross sectional reduction of sound metal.

3.5 Delaminated scale
Scale fully or partially detached from the surface but still in contact with the test piece.

3.6 Gross mass change
Mass change of test piece after cooling including collected spalled scale.

3.7 Net mass change
Mass change of test piece after cooling without including the mass of spalled scale.

3.8 High temperature corrosion
Corrosion occurring when the temperature is higher than the dew point of aqueous phases of the environment but at least 373 K.

3.9 Breakaway
Rapid increase in corrosion rate following a change from protective to non-protective scale growth.

3.10 Thermal cycle
The sequence of temperatures that is repeated throughout the test. A single thermal cycle consists of the heating phase, the hot dwell time, the cooling time and the cold dwell time.

4 Test apparatus

4.1 Design of apparatus
The apparatus shall be comprised of a mechanism to alternate the test pieces between hot and cold environments. The heating device should ideally be equipped with a testing portion capable of separating the test piece from outside air (this assembly is referred to as a closed system), unless this is impracticable for the cyclic test planned. When applicable, a humidifying regulator should be used to continuously supply the gas kept at a constant humidity which should be monitored with a hygrometer. The gas supply shall be controlled by a gas flow meter. A facility to accelerate cooling may also be included. Examples of basic designs are shown in Figure 1.
4.1.1 The test piece chamber shall not be composed of a material that reacts with the test atmosphere during the test to a degree that it changes the composition of the atmosphere.

4.1.2 The heating device should be designed such that the test piece chamber is isolated from the external environment. It should also be ensured that a continuous gas flow within the prescribed range passes the test pieces.

4.1.3 If a closed system with a test piece chamber cannot be used, then the tests may be performed in an open system with laboratory air. In this case the humidity of the air shall be recorded and the laboratories should be kept free from temperature changes and influences from weather conditions as far as possible. Ideally, however, closed systems should be used.

4.1.4 The furnace shall be characterised prior to the testing to determine the temporal temperature profile at a position near to the test piece. This can be achieved by using dummy test pieces and appropriate thermometry.

4.1.5 The temperature regulating device shall be capable of guaranteeing that the temperature of the test piece is kept within the permissible range given in Table 1. The temperature of the furnace may vary or fluctuate due to movement of the furnace (less pronounced when test piece supports are moved). The control system used must ensure that the desired temperature inside the furnace is reached rapidly without being exceeded.

4.1.6 The heating device thermocouples for temperature control shall be as follows: The material for thermocouple shall withstand fully the test temperature. Moreover, the diameter of wire is recommended to be as small as possible, within the limit where the thermoelectric power does not change in service.
Table 1 — Permissible tolerance of temperature of test piece

<table>
<thead>
<tr>
<th>Temperature Range</th>
<th>Permissible Tolerance</th>
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<tr>
<td>≤ 573 K</td>
<td>±2</td>
</tr>
<tr>
<td>573 K &lt; T ≤ 873 K</td>
<td>±3</td>
</tr>
<tr>
<td>873 K &lt; T ≤ 1073 K</td>
<td>±4</td>
</tr>
<tr>
<td>1073 K &lt; T ≤ 1273 K</td>
<td>±5</td>
</tr>
<tr>
<td>&gt; 1273 K</td>
<td>±7</td>
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</table>

4.2 Temperature monitoring

4.2.1 The temperature shall be measured by a suitable device according to ASTM E633-00. Thermocouples of type S (Pt - 10%Rh/Pt) or type R (Pt - 13%Rh/Pt) are preferred for the temperature range room temperature up to 1700°C. A thermocouple should be positioned close to the test piece surface and must be calibrated according to 4.2.2. If however the environment does not allow the use of such thermocouples in this way, the test piece temperature during one complete temperature cycle has to be deduced from the furnace calibration using dummy test pieces and appropriate thermometry in an inert environment.

4.2.2 Calibration of thermocouples shall be performed in accordance with ASTM E220-02 (Standard method for calibration of thermocouples by comparison techniques), ASTM E230-02 (Standard temperature-electromotive forces tables for standardized thermocouples), ASTM E1350-97 (Standard test method for testing sheathed thermocouples prior to, during and after installation). In this case, a representative thermocouple taken up from the batch of wire may be calibrated.

It is recommended that they therefore be calibrated at the beginning and the end of each experiment if there is uncertainty about thermocouple stability.

4.2.3 The thermocouple shall be capable of confirming the temperature of the test piece to be within the range given in Table 1. It shall be on a defined, fixed place as close to the test pieces as possible.

4.2.4 The monitoring thermocouple shall withstand the gaseous test environment. Otherwise it shall be protected by an appropriate shield.

4.3 Gas supply for closed system operation

4.5.1 The gas supply system shall be capable of supplying the test gases at a constant rate to the test piece chamber (see example in Figure 1).

4.5.2 When a humidifying regulator is used it shall be capable of adjusting to the desired humidity. Deionised water of conductivity less than 1 µS/cm shall be used, unless otherwise specified.

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

4.5.4 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.

4.5.5 In the case that the gas is humidified the water vapour 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.

5 Test pieces

5.1 The test piece shall have a minimum surface area of 500 mm² and may be a rectangular plate, a disc or a cylinder.

5.2 The rectangular plate and the disc test pieces shall have a minimum thickness of 1.5 mm
5.3 If the test pieces in 5.2 cannot be made, the shape and dimensions of the test piece shall be in accordance with the agreement between the parties involved.

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

5.5 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 2.

Table 2 – Designation and mean diameter of particles of coated abrasives according to regional standards

<table>
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<th>Standard</th>
<th>Designation</th>
<th>Mean diameter [µm]</th>
<th>Region</th>
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<td>FEPA® 43-1984 R 1993: Grit Sizes for Coated Abrasives</td>
<td>P1200</td>
<td>15.3 ± 1.0</td>
<td>Europe</td>
</tr>
<tr>
<td>ISO 6344 Coated abrasives - Grain size analysis</td>
<td>#1000</td>
<td>15.5 ± 1.0</td>
<td>Japan</td>
</tr>
<tr>
<td>JIS R6001-87</td>
<td>600</td>
<td>16.0</td>
<td>America</td>
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<tr>
<td>ANSI B74.12-92 - Specifications for the Size of Abrasive Grain -- Grinding Wheels, Polishing and General Industrial Uses</td>
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a) Federation of European Producers of abrasives

Sharp edges of test pieces may give anomalous behaviour. These shall be removed during the final stages of test piece preparation.

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

5.7 The surface of the test pieces shall not be deformed by marking, stamping or notching. Identification of the test pieces shall be solely on the basis of recording the relative position within the test chamber; however holes for test piece support (figure 4) and or reference marking are permissible.

5.8 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.

5.9 The dimensions of the test piece 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 6906.

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

5.11 Prior to weighing and exposure the cleaned test pieces shall be stored in a desiccator (no longer than 72 hours).

5.12 The mass of the test pieces shall be determined prior to exposure. At least two measurements shall be made for each test piece. The difference between the measurements shall not exceed 0.05 mg.

5.13 It is recommend that duplicate test pieces are used for each time.
6 Test method

6.1 Definition of a thermal cycle

6.1.1 A thermal cycle consists of the heating phase, the hot dwell time, the cooling time and the cold dwell time. An example for a hot dwell temperature of $T_{\text{dwell}}=1273 \text{ K}$ is given in figure 4. The four phases of a temperature cycle are defined in 6.1.2 to 6.1.5.

![Figure 4 – Definition of a thermal cycle with a hot dwell temperature of 1273K; blue line with $T=323\text{K}=50^\circ\text{C}$; red line with $T=0.97\cdot T_{\text{dwell}}=1234\text{K}$.](image)

6.1.2 The heating time starts when the test pieces are heated e.g. by entering the furnace and ends with the beginning of the hot dwell time which is defined in 6.1.3.

6.1.3 The hot dwell time starts when the actual temperature exceeds 97% of the desired hot dwell temperature $T_{\text{dwell}}$ (measured in K). Extensive numerical calculations and comparison between hypothetical and real temperature cycles have shown that only those times of the temperature cycle contribute to oxidation of the test pieces where the temperature is close to the hot dwell temperature$^1$. The hot dwell time ends upon removal from the furnace.

6.1.4 The cooling time starts when the heating of the test piece is stopped e.g. by the removal of the test piece from the furnace and ends when the actual test piece temperature falls below 50°C.

6.1.5 The cold dwell time starts after the test pieces have cooled below 50°C and ends when the test pieces are heated again.

6.2 Types and dwell times of thermal cycles

6.2.1 Three general types of thermal cycles are typical for industrial applications. Long dwell time testing aims to simulate conditions in large-scale industrial facilities encountered in applications such as power generation plants, waste incineration or chemical industry. In these applications the metallic components are designed for extremely long term operation, e.g. for typically up to 100,000 h. Thermal cycling of materials occurs due to planned plant shut downs e.g. for regular maintenance or

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1 G. Strehl, *Materials at High Temperature*, accepted for publication
due to unplanned shut downs as a result of offset conditions. Therefore, the time intervals between various thermal cycles are relatively long and the number of cycles is, related to the long operation time of the components, relatively small, i.e. typically around 50 cycles. Thermal cycling with **short dwell times** is typically experienced in applications such as industrial gas turbines, jet engines, automotive parts, heat treatment facilities etc. The intervals between start and shut-down of the facilities are generally much shorter than in applications with long dwell times. Also, the design life and/or the time until complete overhaul/repair (typically 3,000–30,000 hours) are shorter and, depending on the specific practical application, the number of cycles is much higher than in the cases above.

Testing with **ultra short dwell times** mainly addresses applications of high temperature alloys as heating elements in the form of wires or foils. Another typical application in which such short cycles prevail would be catalyst foil carriers e.g. in cars. In such applications the number of cycles is, related to the overall design life (typically several hundred to a few thousand hours), extremely high and the time intervals between heating and cooling can be as low as minutes or even seconds. Such conditions are commonly encountered in a number of industrial applications such as e.g. burners and hot gas filters but also in a large variety of domestic applications where metallic heating elements are used e.g. in cooking plates, toasters, boilers, dryers, fryers, etc. This special case is not addressed by this standard.

### 6.2.2 Standard test parameters for long dwell time testing

The definition of 6.1 for a thermal cycle shall be applied to long dwell time testing. For practical reasons it is useful to stay within a 24 h operation rhythm. Therefore experiments shall be performed with a 20 h hot dwell time and 4 h period which includes the cooling time, the cold dwell time and the heating time. The heating and cooling times according to 6.1.2 and 6.1.4 shall be reported.

### 6.2.3 Standard test parameters for short dwell time testing

The definition of 6.1 for a thermal cycle shall be applied to short dwell time testing. The hot dwell time shall be 1 h, the cold dwell time shall be 15 min. Heating and cooling times according to 6.1.2 and 6.1.4 shall be reported. For weighing procedures for mass determinations the cold dwell time may be extended but shall be kept as short as possible.

### 6.2.4 Other dwell times

Dwell times that differ from the standard in 6.2.2 and 6.2.3 are allowed under exceptional circumstances if agreed between the parties concerned.

**BREAK 18. November 2005**

### 6.3 Testing duration

The testing duration shall be at least 300 h of accumulated hot dwell time to allow a significant oxidation of the test pieces. For more reliable results it is, however, recommended to extent the accumulated hot dwell time to at least 1000 h.

Note – For ultra short dwell testing the lifetime may be controlled by the number of cycles. Lifetimes less than 300h are often observed.

Figure 5 shows three types of different oxidation behaviour giving an indication of minimum testing duration. Testing is terminated when protective behaviour is no longer found for the materials.
6.4 Supporting of test pieces

6.4.1 The test pieces shall be supported according to the following principles:

6.4.2 The test piece shall be supported by a material that does not react at the test temperature. Contacts between test piece and support shall be minimised.

6.4.3 The supporter of the test piece to be used shall ideally be designed to be able to collect the scale if it flakes during testing or during cooling after finishing the test.

6.4.4 When spalled oxide is collected and multiple test pieces are exposed simultaneously, each test piece shall be inserted into an individual test piece supporter in order to enable the collection of all spall individually from each test piece.

6.4.5 The support design shall ensure that the test atmosphere flows evenly over the major faces of each test piece. This can be achieved by holes of 1-2 mm in length in width in the side wall of the test piece supporters in the bottom area.

6.4.6 Examples of suitable test piece supports and basic layout of test piece arrangement are shown in Figures 6 to 8.

Figure 5 – Different types of oxidation behaviour

Figure 6 — Test piece supporter and basic layout of test piece arrangement – tube design (this type of support is not suitable for rapid heating and cooling)
6.5 Test environment in air oxidation testing

6.5.1 The gas flow shall be high enough to ensure an overpressure at the exit of the furnace and that no 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 (if applicable) in some applications to reach equilibrium. For tubes with a diameter between 5 and 15 cm the volume flow rate shall be between 5 and 10 l of gas per hour resulting in a gas flow velocity of 0.02 to 0.35 mm s⁻¹.

6.5.2 For COTEST dry synthetic air shall be used as test gas as humidity of laboratory air varies significantly depending on the location of the laboratory and local weather conditions. If tests have to be performed in humidified air, the water content has to be reported in specific humidity (g of water per kg air).

6.5.3 For COTEST dry synthetic air shall be used within the validation testing for all materials except for P91 which shall be tested in humidified air with a specific humidity of 20 g water/kg air corresponding to a dew point of 25°C.

6.5.4 As synthetic air the following gas composition shall be used:
20 Vol-% O₂, 80% Vol-% N₂

6.6 Special test parameters in complex corrosive environments

6.6.1 Test environment
Test environments for testing in complex corrosive atmospheres, under deposits or burner rig burner rig conditions are given in the annex of this code of practice.

6.6.2 Dwell time test parameters
Dwell time test parameters for testing in complex corrosive atmospheres, under deposits or burner rig burner rig conditions are given in the annex of this code of practice.

6.7 Determination of mass change by oxidation
The use of tweezers is recommended. Test pieces shall never be touched with the hands to eliminate any contamination (grease, salts). Care has to be taken when using gloves as the contamination with
the separating agent of the gloves leads to falsification in mass determination. If consistency between individual measurements cannot be found the measurement environment has to be controlled.

6.7.1 Measurements prior to testing

6.7.1.1 New test piece supporters shall be put into the furnace for at least 24 h to remove water and other residues from production. It is recommended to subject each empty test piece supporter to be used to one thermal cycle before the test to remove water traces.

6.7.1.2 The mass of the test pieces shall be determined prior to exposure. Three individual measurements shall be made for each test piece with a precision of 0.02 mg. The standard deviation for the measurements shall not exceed 0.05 mg.

6.7.1.3 The mass of the test piece supporters shall be determined prior to exposure. Three individual measurements shall be made for each test piece supporter with a precision of 0.02 mg. The standard deviation for the measurements shall not exceed 0.05 mg.

6.7.1.4 The mass of the test piece supporter containing one test piece shall be determined prior to exposure. Three individual measurements shall be made for each test piece supporter containing one test piece with a precision of 0.02 mg. The standard deviation for the measurements shall not exceed 0.05 mg.

6.7.2 Intermediate and final mass change determination

6.7.2.1 After removing from the furnace the test piece supporters containing the test pieces shall be settled in the weighing room for 15 minutes to allow them to aclimatise. The test pieces shall not be descaled unless specified by the customer.

6.7.2.2 For each mass change determination at intermediate stages and final stage the mass of the test piece supporter containing one test piece and spalled scales, the test piece supporter including spalled scales and the mass of the test piece including adherent scales shall be determined as shown in Figure 9. Three individual measurements shall be made for each test piece supporter containing one test piece, each test piece supporter and each test piece with a precision of 0.02 mg for each measurement. The standard deviation for each set of measurements shall not exceed 0.05 mg.

6.7.2.3 Care must be taken during mass measurements to avoid spallation of oxide scales caused by mechanical contacts with tweezers etc. A test piece supporter with a rod support design as shown in figure 8 circumvents the problem and allows the determination of mass changes without direct contact of the tweezers with the test piece as shown in figure 10.

![Figure 9 — Mass determination – variant I](image_url)

\[ m_{ST} \]: mass of test piece supporter with test piece

\[ m_{S} \]: mass of test piece supporter

\[ m_{T} \]: mass of test piece
Figure 10 — Mass determination – variant II (test piece can be removed by holding the rod with tweezers)

6.7.2.4 Gross mass change $\Delta m_{\text{gross}}$ as defined in 3.5 is determined according to equation 6.1.

$$\Delta m_{\text{gross}}(t_n) = m_{ST}(t_n) - m_{ST}(t_0)$$  \hspace{1cm} \text{Eq. 6.1}

where,

- $\Delta m_{\text{gross}}(t_n)$: gross mass change at time $t_n$ (mg)
- $m_{ST}(t_n)$: mass of test piece supporter and test piece at time $t_n$ (mg)
- $m_{ST}(t_0)$: mass of test piece supporter and test piece prior to the test (mg)

6.7.2.5 The mass of spalled oxide $\Delta m_{\text{spall}}$ is determined according to equation 6.2.

$$\Delta m_{\text{spall}}(t_n) = m_{S}(t_n) - m_{S}(t_0)$$  \hspace{1cm} \text{Eq. 6.2}

where,

- $\Delta m_{\text{spall}}(t_n)$: mass change of spalled material at time $t_n$ (mg)
- $m_{S}(t_n)$: mass of test piece supporter at time $t_n$ (mg)
- $m_{S}(t_0)$: mass of test piece supporter prior to the test (mg)

6.7.2.6 Net mass change $\Delta m_{\text{net}}$ as defined in 3.6 is determined according to equation 6.3:

$$\Delta m_{\text{net}}(t_n) = m_{T}(t_n) - m_{T}(t_0)$$  \hspace{1cm} \text{Eq. 6.3}

where,

- $\Delta m_{\text{net}}(t_n)$: net mass change at time $t_n$ (mg)
- $m_{T}(t_n)$: mass of test piece at time $t_n$ (mg)
- $m_{T}(t_0)$: mass of test piece prior to the test (mg)

or when a rod supported design as shown in figure 8 is used by equation 6.4.

$$\Delta m_{\text{net}}(t_n) = \Delta m_{\text{gross}}(t_n) - \Delta m_{\text{spall}}(t_n)$$  \hspace{1cm} \text{Eq. 6.4}

where,

- $\Delta m_{\text{net}}(t_n)$: net mass change at time $t_n$ (mg)
- $\Delta m_{\text{gross}}(t_n)$: gross mass change at time $t_n$ (mg)
- $\Delta m_{\text{spall}}(t_n)$: mass of spalled material at time $t_n$ (mg)
6.7.3 Frequency of mass change determination

6.7.3.1 For long dwell time testing mass change measurements shall be made once each working day for the initial part of the test, measurements may be made less frequently as the test progresses.

6.7.3.2 In short dwell time testing the extension of the cold dwell phase due to the mass change measurements must be taken into account. Therefore the number of mass change measurements shall be small enough to minimize these possible influences but high enough to obtain meaningful mass change curves. Mass change measurements on a daily basis have proven to be practicable.

6.7.3.3 In ultra short dwell testing pieces cannot be remounted in the test facility after mass change measurement. Therefore individual test pieces shall be removed after a range of cycles / exposure times to determine mass change and correlate this with the change in resistance while the rest of the initial number of test pieces are continued to be subjected to thermal cyclic oxidation testing. Test pieces shall be removed after 10, 20, 40 and 100 h of accumulated hot dwell time.

6.7.3.4 It is recommended to document the macroscopic appearance of the test piece surface by macro photographs.

6.7.3.5 Thermal cyclic oxidation testing in complex corrosive environments shall follow 6.6.5.1 to 6.6.5.3.

6.7.4 Analysis of mass change

6.7.4.1 Net mass change of test pieces shall be plotted versus time as shown in figure 11. According to the common use in high temperature oxidation testing the mass change is usually described mathematically by the equation given in the box in figure 11 (Eq. 6.5). The values for the oxidation rate \( k \), the exponent of the growth law \( n \), the protective oxide growth time \( t_{\text{protective}} \) and the corresponding number of cycles \( N_{\text{protective}} \) shall be reported. Determination of these values is described in 6.7.4.2 to 6.7.4.4.

\[
\Delta m(t) = \Delta m_{\text{net}}(A) \quad \text{[mg/cm}^2]\]

\[
\Delta m_{\text{net}}(A) = \frac{1}{A} \int_{0}^{t} k^n t^n dt \quad \text{Eq. 6.5}
\]

A change in the mechanism (spallation, break away oxidation) becomes apparent by a change of the slope in the double logarithmic plot. Analysis of the linear part of the curve by linear regression with
means of simple spreadsheet calculations yields the slope $b=1/n$ and the y-axis intercept as shown in figure 12.

\[ \log (\Delta m/A)_{\text{prot.}} - \log (\Delta m/A)_{300h} = t_{\text{300h}} - t_{\text{protective}} \]

**Figure 12 — Double logarithmic plot of mass change vs. time**

6.7.4.3 From the y-axis intercept the oxidation rate constant $k$ for the linear region shall be calculated as given in Eq. 6.6

\[ a = \log k = \frac{1}{n} \log k \rightarrow \frac{1}{n} \log k = 10^{a/n} \quad \text{Eq. 6.6} \]

6.7.4.4 For the determination of $k$, $n$ and the protective oxide growth time $t_{\text{protective}}$ an iterative procedure shall be applied. In a first step a best fit line shall be drawn through the data points in the "linear range" using the functionality of simple spreadsheet calculations. The correlation coefficient defined by Eq. 6.7 shall be maximized by including or excluding data points near to the point where data points leave the linearity area.

\[ \rho_{\log t, \log \Delta m/A} = \frac{\text{cov}(\log t, \log \Delta m/A)}{\sigma_{\log t} \cdot \sigma_{\log \Delta m/A}} \quad \text{Eq. 6.7} \]

with the standard deviations $\sigma_{\log \Delta m/A}$ and $\sigma_{\log t}$ and the covariance $\text{cov}(\log t, \log \Delta m/A)$ between two data sets defined by Eq. 6.8.

\[ \text{cov}(X, Y) = \frac{1}{n} \sum_{j=1}^{n} (x_j - \mu_x)(y_j - \mu_y) \quad \text{Eq. 6.8} \]

The data points included in the calculation of the correlation coefficients shall be used to finally calculate $k$ and $n$. The latest data point included in the linear regression shall be reported as $t_{\text{protective}}$ (protective oxide growth time).

6.7.4.5 The number of cycles $N_{\text{protective}}$ that corresponds to the protective oxide growth time $t_{\text{protective}}$ and which is the last data point that is included in the linear regression shall be reported.

6.7.4.6 Additionally the following net mass changes difference shall be reported as given in Eq. 6.7 and 6.8 and shown in Figure 13.

\[ \Delta m_{\text{net}}(t_{300h} - t_{\text{protective}}) = \Delta m_{\text{net}}(t_{300h}) - \Delta m_{\text{net}}(t_{\text{protective}}) \quad \text{Eq. 6.7} \]

where, \( \Delta m_{\text{net}}(t_{300h}) \) : net mass change of test piece after 300 h (mg)

\( \Delta m_{\text{net}}(t_{\text{protective}}) \) : net mass change of test piece at $t_{\text{protective}}$ (mg)
\[ \Delta m_{\text{net}}(t_{1000h} - t_{\text{protective}}) = \Delta m_{\text{net}}(t_{1000h}) - \Delta m_{\text{net}}(t_{\text{protective}}) \]  \quad \text{Eq. 6.8} \\

where,  
\[ \Delta m_{\text{net}}(t_{1000h}) \] : net mass change of test piece after 1000 h (mg)  
\[ \Delta m_{\text{net}}(t_{\text{protective}}) \] : net mass change of test piece at \( t_{\text{protective}} \) (mg) 

6.8 Determination of Resistance Change due to Oxidation (Ultra Dwell Tests) 

6.8.1 During the hot dwell period of the first thermal cycle the current and voltage readings are adjusted to reach the required test temperature 

a) The initial power (\( P_I \)) is calculated from \( V_{\text{rms}} \times I_{\text{rms}} \), assuming A.C. Joule heating. 

b) The initial resistance (\( R_I \)) is calculated from \( V_{\text{rms}} / I_{\text{rms}} \), assuming A.C. Joule heating. 

6.8.2 Control of the rapid cyclic oxidation test is through control of the power. The power level is adjusted to keep the upper dwell temperature constant, to within specified tolerance. 

6.8.3 Frequency of resistance measurement 

6.8.3.1 Resistance measurements should be determined every cycle until stabilized (stress relief during early cycles can lead to a drop in resistance). The stabilized, minimum resistance should be recorded (\( R_o \)). 

6.8.3.2 During the ultra short dwell cyclic oxidation tests, hot resistance should be determined periodically as necessary to keep power control of temperature within \( \pm 10^9 \)K. It is recommended that such measurements are taken every 30-50 cycles throughout the test until burnout. More frequently if rapid changes in resistance are observed.
6.8.4 Analysis of Resistance Change

6.8.4.1 Fractional change in resistance \( \frac{\Delta R}{R_o} \) is defined as \( \frac{R(t_n) - R_o}{R_o} \) \hspace{1cm} \text{Eq. 6.9}

where \( R(t_n) \) is the measured hot resistance \( \left( \frac{V_{rms}}{I_{rms}} \right) \) at time \( t_n \), measured in ohms, where \( n \) is the cycle number. \( R_o \) is the minimum resistance determined in or shortly after cycle 1 (see Figure 14 for definition). \( R_o \) may be less than \( R_i \) due to thermal annealing during the first few cycles.

6.8.4.2 Fractional change in resistance shall be plotted versus number of cycles (time) as shown in Figure 15. The fractional resistance change plot is a dimensionless measure of the effect of oxidation rate on the resistance of the wire and thus can normalize the performance of wires for different diameters. This is not the case for foil materials, where change in cross section area must be taken into account.

![Graph showing resistance vs. number of cycles](image1)

**Figure 14 - Definition of \( R_i, R_o \) and \( R(t_n) \)**

![Graph showing change in resistance vs. number of cycles](image2)

**Figure 15 - Change in resistance with number of cycles.**
6.8.4.3 The resistance of a wire, foil or ribbon sample is given by:

\[ R = \rho \frac{L}{A} \]  

Eq. 6.10

where \( R \) = resistance, \( \rho \) = resistivity, \( L \) = length of the wire, foil or ribbon and \( A \) is the cross section area.

The fractional resistance defined in Eq.6.9 can be related to specimen dimensions by:

\[ \frac{\Delta R}{R_o} = \frac{R(t_n) - R_o}{R_o} = -\frac{\frac{L(t_n)}{A(t_n)} - \frac{L_o}{A_o}}{\frac{L_o}{A_o}} \]  

Eq. 6.11

where \( L(t_n), A(t_n) \) relate to the specimen length and cross sectional area at time \( t_n \) (after \( n \) cycles), \( \rho \) is the wire resistivity for a thermally annealed wire. \( L_o \) and \( A_o \) are the initial wire length and cross sectional area. [Note: \( \rho \) may be less than \( \rho_o \), the initial resistivity of the wire, due to thermal annealing effects over the first few thermal cycles]

Thus changes in wire cross section area \( A(t_n) \) at time \( t_n \), due to oxidation processes, can be directly correlated with measured \( \frac{\Delta R}{R_o} \) values.

6.8.4.4 For wire samples equation 6.11 can be simplified:

\[ \frac{\Delta R}{R_o} = 4\rho \frac{\left( \frac{L(t_n)}{(d(t_n))^2} - \frac{L_o}{(d_o)^2} \right)}{\pi} = \frac{L(t_n)}{L_o} \frac{d_o^2}{(d(t_n))^2} - 1 \]  

Eq. 6.12

where \( d_o \) and \( d(t_n) \) are the wire diameters initially and at time \( t_n \).

6.8.4.5 For small changes in wire dimensions this permits an estimate of metal loss from oxidized wires. The metal loss \( (\Delta x) \) would be \( (d_o - d(t_n))/2 \). Thus equation 6.12 simplifies to

\[ \Delta x = d_o \frac{\Delta R}{4 \frac{R_o}{R_o}} \]  

Eq. 6.13

Thus a plot of \( \frac{\Delta R}{R_o} \) versus time can be treated in a similar manner to mass change data (section 6.7.4) to study oxidation kinetics for the early stages of oxidation where the assumptions of small section loss hold true. For a more exact calculation or for foil and ribbon samples equation 6.11 has to be used to calculate changes in cross section area.

7 Post-test evaluation of test pieces

7.1 Macroscopic evaluation

The macroscopic appearance of the test piece surface shall be photographed.
7.2 Metallographic cross section

7.2.1 Scale thickness measurements shall be made by metallographic cross-sections following the TESTCORR recommendations. Care has to be taken in mounting the specimen orthogonal to the primary axis of the test piece. The cross sectioned test pieces shall be analysed using conventional light microscopy. Measurements shall consist of:
- deposit thickness
- scale thickness
- depth of internal penetration
- depth of grain boundary attack measured
- depth of any depleted zone
- remaining cross section of unaffected material
A minimum of 8 measurements per test piece shall be obtained. In addition, the position of maximum attack shall be measured.

8 Report

8.1 Matters to be described

The following data, where known shall be included in the report on the test results.

8.1.1 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
f) Processing condition
g) Heat treatment condition
h) Microstructure of the material determined according to ASTM E3-01 and etching according to ASTM E407-00.

8.1.2 Test piece

a) Designation of test piece
b) Dimensions and surface area of test piece
c) Surface finish condition of test piece
d) Degreasing method of test piece
e) Method of test piece support
f) initial mass

8.1.3 Testing environments

a) Test temperature, and maximum and minimum temperatures during the test
b) Test duration
c) Hot dwell time, cold dwell time, heating time, cooling time
d) Dew point temperature of humidified air or humidity of laboratory air
e) Flow rate of test gas
f) Volume flow rate of test gas
g) Open or closed system according to 4.1.3

8.1.4 Test result

a) Plot of mass change per area in mg cm⁻² vs. time according to 6.7.2.

b) Results of any metallographic investigations performed according to 7.2.1.

c) Amount of spalled scale in mg

d) Photograph of appearance after testing

e) Photograph of cross section including the surface layer of the metallographic section of test piece after testing

f) Oxidation rate $k$, exponent of the growth law $n$, cycles $N_z$ and the accumulated hot dwell time $t_z$ that corresponds to the onset of spallation or breakaway oxidation according to 6.7.2.

8.2 Supplementary note

It is desirable additionally to describe the following matters in the report on the test results:

8.2.1 Mechanical properties of the raw material at room temperature

8.2.2 Sampling conditions of the test piece from raw material

8.2.3 Outline of the test apparatus

8.2.4 SEM/EDX analysis
ANNEX A: Thermal Cycling Oxidation Testing with Deposits

A.1 Scope

A.2 Application of Code of practice

Sections 1 to 5 of the Code of Practice shall apply.

A.3 Test Method

Section 6 of the Code of Practice shall apply with the following modifications:

A.3.1 Dwell time of the thermal cycle

The methodology of long dwell time testing shall be applied [Section 6.2 of the Code of Practice].

A.3.2 Dwell time test parameters

The definition of 6.1 of the Code of Practice shall apply. For practical reasons, the 24 h operation rhythm is maintained with a hot dwell time of 18 h and a 6 h period, including the cooling time, the cold dwell time and the re-heating time. The heating and cooling times according to 6.1.2 and 6.1.4 of the Code of Practice shall be reported [Section 6.2.2 of the Code of Practice].

A.3.3 Testing duration

For COTEST, the minimum number of cycles for deposit testing shall be 17 giving a hot dwell duration total of 306 h [Section 6.3 of the Code of Practice].

A.3.4 Test piece support

The rod supported design of Figure 6 is recommended [Section 6.4 of the Code of Practice].

A.3.5 Test environment

For COTEST, dry synthetic air containing 0.5vol-% SO2 humidified to contain 2.5vol-% H2O is recommended. If difficulties are encountered with the formation of H2SO4 in the furnace, the SO2 or the H2O may be omitted [Section 6.5 of the Code of Practice].

A.3.6 Definition of deposit

For COTEST, a salt mixture of Na2SO4:K2SO4 corresponding to the eutectic composition with a theoretical melting point of 830°C [75 mol-% Na2SO4 and 25 mol-% K2SO4] shall be applied at a target level of 0.6 mg.cm-2.

A.3.7 Method of deposit application

For COTEST, it is recommended that the deposit application be made by dissolving 1 wt-% of the deposit according to 2.6 in water and spraying the salt solution obtained with a small paint sprayer using N2 (or compressed air) as a carrier gas. The target amount of deposit is 0.6 mg/cm2. Deposit should cover the whole specimen surface. Detailed instructions for deposit application on rectangular samples are given as follows.

Deposit application is made by spraying with a small paint sprayer using N2 (or compressed air) as a carrier gas, Fig. 1. Deposit should cover the whole specimen surface. The test specimens are preheated at 150-200°C for 10 -15 min in their alumina support crucible to obtain an even deposit layer on the specimen surface. Only the specimens are taken out (with tweezers) from the preheating
furnace to prevent unnecessary cooling. The preheating furnace should be located near (within 1 to 5 m) to the spraying set-up. The target amount of deposit is 0.6 mg/cm².

Before the deposit spraying, test sprayings with different spray distances (distance "a" in Fig A-2.) and times are done to ensure the uniformity of spray and the adequate, even coverage area. The right distance a can be determined by spraying the deposit on a piece of colourful paper at different distances a. If the deposit dries up quickly, it might block the paint sprayer. Therefore, the behaviour of the deposit should be observed during the distance tests. Test sprayings are also done to preheated samples to determine the spraying time. The time should not exceed the point where liquid deposit droplets start to form on the sample surface. If necessary, the sample can be reheated for a few minutes before spraying the other side of the sample.

Specimens are weighed at room temperature before and after the deposit application to obtain required amount of deposit. The spraying step and weighing are repeated if necessary.

Prepared specimens should be stored overnight in a desiccator to ensure a complete drying of deposit.
A.3.8 Frequency of re-application

For COTEST, the deposit shall be re-applied every 5 cycles [once a week].

A.3.9 Determination of mass change

In addition to the intermediate mass change measurements described in the Code of Practice [Section 6.7.2], the mass of the test piece supporter with test piece held by an alumina rod after the re-application of deposit \([m_{STD}]\), and the mass of the test piece supporter after completion the test and weighed after any spall has been taken out from the test piece supporter by applying gentle mechanical means, such as brushing, \([m_{TDF}]\), shall be measured.

A.3.10 Frequency of mass change determination

Mass measurements at the completion of each cycle are recommended for the first 5 cycles. Thereafter the frequency may be reduced to every 2 or 3 cycles, except that measurements must be made before and after the re-application of deposit [Section 6.7.3 of the Code of Practice].

A.3.11 Analysis of mass change

The guidelines of the Code of Practice [Section 6.7.4 of the Code of Practice] hold regarding that values of mass changes used in the analysis shall be calculated using modified Equations 6.1_{DEP} - 6.4_{DEP} as follows.

Gross mass change \(\Delta m_{\text{gross}}\) as defined in 3.5 of the Code of Practice is determined according to equation 6.1_{DEP}.

\[
\Delta m_{\text{gross}}(t_1) = m_{ST}(t_1) - m_{ST}(t_0) - m_{DEP}(t_1) \quad \text{Eq. 6.1}_{\text{DEP}}
\]

where,
- \(\Delta m_{\text{gross}}(t_1)\) : gross mass change at time \(t_1\) (mg)
- \(m_{ST}(t_1)\) : mass of test piece supporter and test piece at time \(t_1\) (mg)
- \(m_{ST}(t_0)\) : mass of test piece supporter and test piece prior to the deposit applications (mg)
- \(m_{DEP}(t_1)\) : calculated cumulative mass of deposit by time \(t_1\) (mg); \(m_{DEP}(t_1) = \Sigma (m_{STD}(t_1)-m_{ST}(t_1))\)

6.7.2.5 The mass of spalled and that of material eventually absorbed in the test piece supporter \(\Delta m_{\text{spall}}\) is determined according to equation 6.2_{DEP}.

\[
\Delta m_{\text{spall}}(t_1) = m_{S}(t_1) - m_{S}(t_0) \quad \text{Eq. 6.2}_{\text{DEP}}
\]

where,
- \(\Delta m_{\text{spall}}(t_1)\) : mass change of spalled and absorbed material at time \(t_1\) (mg)
- \(m_{S}(t_1)\) : mass of test piece supporter at time \(t_1\) (mg)
- \(m_{S}(t_0)\) : mass of test piece supporter prior to the first deposit application (mg)
6.7.2.6 Net mass change $\Delta m_{\text{net}}$ as defined in 3.6 is determined according to equation 6.3$_{\text{DEP}}$ or 6.4$_{\text{DEP}}$.

\[ \Delta m_{\text{net}}(t_1) = m_T(t_1) - m_T(t_0) - m_{\text{DEP}}(t_1) \quad \text{Eq. 6.3}_{\text{DEP}} \]

where,
- $\Delta m_{\text{net}}(t_1)$ : net mass change at time $t_1$ (mg)
- $m_T(t_1)$ : mass of test piece at time $t_1$ (mg)
- $m_T(t_0)$ : mass of test piece with no deposit applied prior to the test (mg)
- $m_{\text{DEP}}(t_1)$ : calculated cumulative mass of deposit by time $t_1$ (mg);
  \[ m_{\text{DEP}}(t_1) = \Sigma (m_{\text{STD}}(t_1) - m_{\text{ST}}(t_1)) \]

\[ \Delta m_{\text{net}}(t_1) = \Delta m_{\text{gross}}(t_1) - \Delta m_{\text{spall}}(t_1) \quad \text{Eq. 6.4}_{\text{DEP}} \]

where,
- $\Delta m_{\text{net}}(t_1)$ : net mass change at time $t_1$ (mg)
- $\Delta m_{\text{gross}}(t_1)$ : gross mass change at time $t_1$ (mg)
- $\Delta m_{\text{spall}}(t_1)$ : mass of spalled material at time $t_1$ (mg)

**A.4 Post-test evaluation of test pieces**

The methodology described in section 7 of the Code of Practice shall apply. It is recommended that non-aqueous media be used for the grinding/polishing of cross-sections to avoid loss of water-soluble deposit/oxidation product.

**A.5 Report**

The information described in section 8 of the Code of Practice shall be reported. In addition, the following shall be reported:

8.1.2 (g) the weight of the deposit applied at each application [in mg.cm$^{-2}$]

(h) the calculated total mass of deposit (mg); $m_{\text{DEP}} = \Sigma (m_{\text{STD}}(t_1) - m_{\text{ST}}(t_1))$, where the summation is taken over all the times $t_1$

(i) the calculated mass change of the test piece supporter (mg); $\Delta m_{\text{SD}} = m_{\text{S}}(t_0) - m_{\text{SDF}}$

8.1.3 (h) the composition of the test gas
Annex Test method for testing in low velocity burner rigs

Scope

Test Apparatus/Principle of Test

Low Velocity Burner Rig testing allows cyclic oxidation to be carried out with the additional influence of continuous deposition of salts. The test involves injection of fuel and salt solution into a heated chamber where combustion occurs. The combined action of the injection and the flow of the combustion atmosphere then carry the salt downstream where it may react with the fuel and/or vaporise and is then subsequently deposited/condensed onto the test pieces. The combustion and stabilisation zones of the burner rig are maintained at a higher temperature than the test piece zone in order to provide the driving force for deposition. The test pieces are rotated to ensure homogenous deposition of salt around their circumference.

Test Pieces

Test pieces shall, subject to limitations in available material, be of circular section and have a domed upper surface avoiding any sharp edges. Grooves/notches are allowable to secure the specimen in the holder. A suitable geometry is shown in Figure B-1.

Test conditions

The test is defined by the following parameters:

(a) Temperature cycle
(b) Chemical composition of fuel and salt
(c) Fuel/air ratio
(d) Salt deposition rate

Thermocycles in burner rig testing

According to the definition in section 6.1.1 of the code of practice, a thermocycle consists of the heating phase, the hot dwell time, the cooling time and the cold dwell time. An example is given in Figure B-2 for a hot dwell temperature of 1273 K. The four phases of a temperature cycle are defined in A.4.2 to A.4.5.
B.5.2 The heating time starts when the test pieces are heated e.g. by entering the furnace and ends with the beginning of the hot dwell time which is defined in B.5.3.

B.5.3 The hot dwell time starts when the actual temperature exceeds 97% of the desired hot dwell temperature $T_{dwell}$. Extensive numerical calculations and comparison between hypothetical and real temperature cycles have shown that only those times of the temperature cycle contribute to oxidation of the test pieces where the temperature is close to the hot dwell temperature\(^2\). The hot dwell time ends upon removal from the furnace.

B.5.4 The cooling time starts when the heating of the test piece is stopped e.g. by the removal of the test piece from the furnace and ends when the actual test piece temperature falls below 50°C.

B.5.5 The cold dwell time starts after the test pieces have cooled below 50°C and ends when the test pieces are heated again.

B.5.6 For burner rig testing the cycle is constrained by a daily/weekly cycle.

B.5.7 The recommended cycle is 20 h hot dwell and 4 h total for cooling time, cold dwell and heating time.

B.5.8 The first cycle of the week should start on Monday afternoon allowing specimens to be removed for measurements the following morning. Unless weekend working is envisaged, the final cycle of the week should finish on Friday morning. After measurement the specimens shall be stored in a desiccator until cycling recommences the following Monday.

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\(^2\) G. Strehl, *Materials at High Temperature*, accepted for publication
B.6 Fuel Supply and Salt Deposition

B.6.1 The chemical composition of the fuel and salt, together with the fuel/air ratio and salt deposition rate shall be agreed with the customer prior to commencement of the test programme.

B.6.2 Fuel shall be delivered continuously at the required rate for at least 15 minutes prior to introduction of the specimens to the chamber and throughout the exposure. The fuel may be switched off after removal of the specimens from the chamber.

B.6.3 The deposition rate of the salt is controlled by the contaminant flux rate, salt composition and temperature. The contaminant flux rate for the required deposition rate shall be established prior to the experiment using inert pins in the specimen chamber.

B.6.4 Deposition during the exposure shall be monitored using an inert pin positioned in the specimen holder together with the testpieces.

B.7 Testing duration

The testing duration shall be at least 300 h of accumulated hot dwell time to allow a significant oxidation of the test pieces. For more reliable results it is, however, recommended to extent the accumulated hot dwell time to 1000 h.

B.8 Supporting of test pieces

The test pieces shall be supported according to the following principles:

B.8.1 The test piece shall be supported by a material that does not react at the test temperature.

B.8.2 The specimens shall be continuously rotated to ensure even distribution of deposit around the circumference of the specimens.

B.8.3 Additional restraint in the form of heat resistant wire may be necessary to secure the test pieces in the rotating holder.

B.9 Determination of mass change by oxidation

B.9.1 Test pieces shall never be touched with the hands to exclude any contamination (fat, salts). Care has to be taken when using gloves as the contamination with the separating agent of the gloves leads to falsification in mass determination. The use of tweezers is recommended.

B.9.2 The mass of the test pieces shall be measured at pre-determined intervals. Measurement shall be more frequent at the early stages of the test.

B.9.3 The mass change due to oxidation/corrosion is calculated by subtracting the mass change due to salt deposition from the total mass change. The mass of salt deposited is measured on the inert pins that are exposed in parallel with the test pieces.

B.9.4 Some test pieces may be washed after mass measurements. In this case the mass change prior to and after washing shall be recorded.

B.10 Metallographic Examination Post Test Evaluation of Test Pieces

The methodology described in section 7 of the Code of Practice shall apply. It is recommended that non-aqueous media be used for the grinding/polishing of cross-sections to avoid loss of water-soluble deposit/oxidation product.
B.11 Reporting of Results

The information described in section 8 of the Code of Practice shall be reported. In addition, the following shall be reported:

B.11.1 Testing environments

f) Composition of fuel