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**WORKSHOP**

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**AGREEMENT**

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## High temperature accelerated ageing of advanced ceramic specimens for solar receivers and other applications under concentrated solar radiation

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## **European foreword**

This CEN Workshop Agreement (CWA 17726:2021) has been developed in accordance with the CEN-CENELEC Guide 29 “CEN/CENELEC Workshop Agreements – A rapid prototyping to standardization” and with the relevant provisions of CEN/CENELEC Internal Regulations – Part 2. It was approved by a Workshop of representatives of interested parties on 2020-12-15, the constitution of which was supported by CEN following the public call for participation made on 2020-01-31. However, this CEN Workshop Agreement does not necessarily include all relevant stakeholders.

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The following organizations and individuals developed and approved this CEN Workshop Agreement:

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## Introduction

Concentrated solar power (CSP) is an important building block in installing a secure, competitive and sustainable energy system.

Advanced materials solutions for NEXT generation high efficiency concentrated solar power (CSP) TOWER systems (NEXTOWER) project is a four-year research and development project, funded by the European Commission, which aims at demonstrating high-performance durable materials for the next generation of concentrated solar power (CSP) air-based tower systems, making them commercially competitive in the energy market beyond 2020.

NEXTOWER comprises two main parts: steel and ceramic. The main objective related to ceramic is to develop new mechanically tough and highly thermally conductive ceramic receivers, working under extreme thermal cycling without failure at a maximum operating temperature of up to 1 400 °C and delivering up to 25 years of continued operation.

A general objective in NEXTOWER is the exploitation and standardization, addressing the integration of NEXTOWER in the standardization system.

Some of the results obtained in NEXTOWER regarding the use of advanced ceramics are:

1. Innovative ceramic for high-temperature open volumetric receivers based on all-SiC honeycomb manufactured by a mix of extrusion and slip casting through re-crystallization SiC more resistant to oxidation; better ceramics for high-temperature receivers, with superior thermal properties and reliability. Ceramic receivers optimized for oxidation, in terms of porosity and strength.
2. Innovative ceramic for high-temperature open volumetric receivers based on more flexible siliconized silicon carbide (Si-SiC) multiparts made by additive manufacturing (3D printing), especially designed for higher toughness, higher thermal conductivity and thermal shock resistance through a more open structure, with an optimized joining technique, improving lifetime and avoiding interfacial cracking.
3. Coating and surface treatments to improve thermomechanical properties and emissivity.
4. Proposal to amend ISO 18755 “Fine ceramics (advanced ceramics, advanced technical ceramics) – Determination of thermal diffusivity of monolithic ceramics by laser flash method”.
5. This CWA defines a methodology for testing the performance of the material ceramic materials and will be one of the standardization results. Both the standards community and NEXTOWER partners will benefit from it.

## 1 Scope

This document defines the requirements, operation and analysis for high temperature accelerated ageing of ceramic specimens for solar receivers and other applications under concentrated solar radiation, reaching a solar concentration up to 1 MW/m<sup>2</sup> peak and temperatures up to 1 400 °C.

This document also describes the structural and resistance post analysis of the irradiated samples.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 843-1, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength*

EN 843-2, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 2: Determination of Young's modulus, shear modulus and Poisson's ratio*

EN 843-5, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 5: Statistical analysis*

EN 843-6, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 6: Guidance for fractographic investigation*

EN ISO 9488, *Solar energy — Vocabulary*

EN ISO 22975-3:2014, *Solar energy — Collector components and materials — Part 3: Absorber surface durability*

ISO 9050, *Glass in building — Determination of light transmittance, solar direct transmittance, total solar energy transmittance, ultraviolet transmittance and related glazing factors*

ISO 20509:2003, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of oxidation resistance of non-oxide monolithic ceramics*

IEC/TS 62862-1-1, *Solar thermal electric plants — Part 1-1: Terminology*

ASTM E 903, *Standard test method for solar absorptance, reflectance, and transmittance of materials using integrating spheres*

ASTM E 1441-19, *Standard Guide for Computed Tomography (CT)*

ASTM C 1499-09, *Standard test method for monotonic equibiaxial flexural strength of advanced ceramics at ambient temperature*

ASTM G 173-03, *Standard Tables for Reference Solar Spectral Irradiances: Direct Normal and Hemispherical on 37° Tilted Surface*

ASTM C 1322, *Standard practice for fractography and characterization of fracture origins in advanced ceramics*

### **3 Terms and definitions**

For the purposes of this document, the terms and definitions given in IEC TS 62862-1-1 and EN ISO 9488 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp/ui>

**3.1**  
**thermal cycling**  
temperature which is simply and cyclically repeated between a specific high value and a specific low value

[SOURCE: ISO 17841:2015]

### **3.2**

#### **heating rate**

variation in temperature as a function of time during heating cycle

[SOURCE: ISO 4885:2018, 3.78; ISO/TR 14745:2015, 3.2]

### **3.3**

#### **cooling rate**

variation in temperature as a function of time during cooling cycle

[SOURCE: ISO 4885:1996, 3.37; ISO/TR 14745:2015, 3.1]

### **3.4**

#### **holding temperature**

temperature at which the product or component is kept in order to achieve specified properties

[SOURCE: ISO 4885:1996, 3.78; ISO/TR 14745:2015, 3.3]

### **3.5**

#### **oxidation resistance**

resistance against oxidation of a non-oxide ceramic material due to reaction with oxygen in the surrounding atmosphere, including any internal reactions as a result of the presence of open porosity or of diffusion of ions to or from the ceramic surface

[SOURCE: ISO 20509:2003, 3.1]

### **3.6**

#### **flexural strength**

maximum nominal stress at fracture of a specified beam loaded in bending

[SOURCE: ISO 20509:2003, 3.2]

## **4 Test layout description**

### **4.1 Test pieces or slabs**

#### **4.1.1 General**

The test pieces shall be as follows:

#### **4.1.2 Shape and dimensions**

The test pieces should have a flat form of dense or reticular material.

The total surface area of the test pieces must be smaller than the characteristic solar focus area. In the case of testing of dense flat specimens, as concentrated solar energy will be applied on one side of the sample (on the front side, directly exposed to concentrated solar radiation), its thickness should not be higher than 10 mm for continuous materials (a lower thickness is recommended for ceramic samples, where the thermal gradient between the exposed and unexposed side to solar radiation is usually more pronounced) and up to 30 mm for porous materials in order to get a homogenous temperature gradient across the sample height. This is related to the low intrinsic thermal conductivity of most ceramic materials.

The shape and dimensions of the test pieces shall be in accordance with the agreement between the parties involved.

NOTE The dimensions used in NEXTOWER for small flat specimens were 50 mm × 50 mm × 5 mm.

#### **4.1.3 Environmental conditions**

Testing shall be carried out in air or any other controlled atmosphere, depending on the final application of the specimens.

NOTE The ageing testing in NEXTOWER was in air, in order to compare the CSP real conditions in a Central Receiver facility.

### **4.2 Test platform**

#### **4.2.1 General**

The test platform shall consist of a set-up that will enable transition of the test pieces between maximum and minimum temperature (hot and cold environment) with a controlled heating and cooling rates, in a practical and reproducible manner.

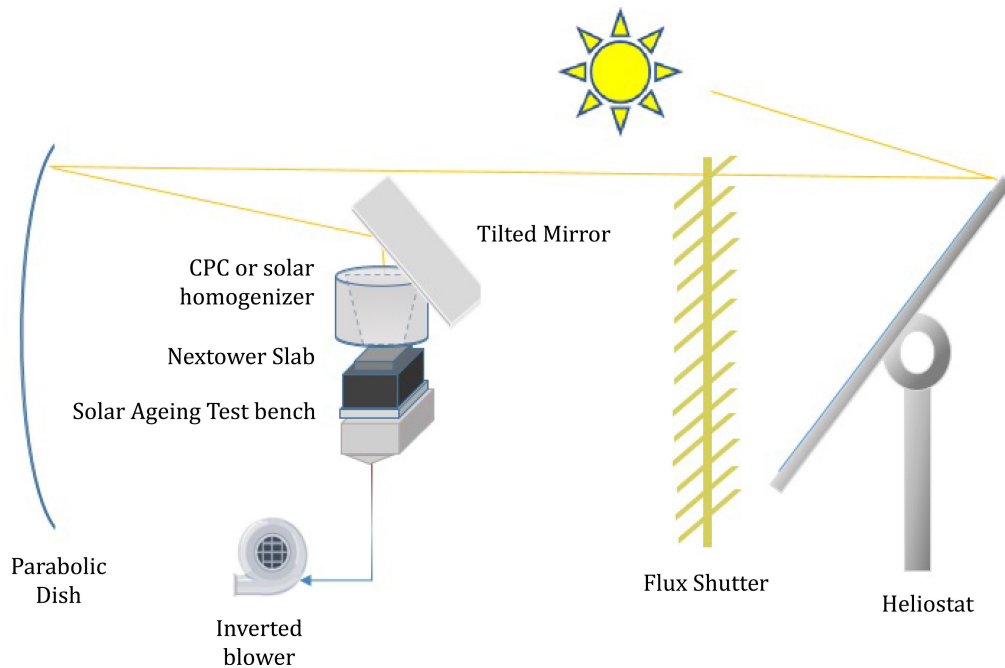
#### **4.2.2 Requirements for the test platform**

Some of the requirements for the test bench for high temperature accelerated ageing of ceramic specimens for solar receivers and other applications under concentrated solar radiation are:

- The test bench shall include a support able to hold or maintain the samples without taking away degrees of freedom for thermal expansion, avoiding causing stresses that lead to premature failure.
- The compatibility between the material of the samples to be tested and the support shall be checked. The support material shall not react with the samples for the whole test temperature range.
- The support shall allow the samples to cool down homogeneously. This is achieved with porous supports or with holes that allow the working fluid (air or other gasses) going through it.
- It is recommended to work in a horizontal position, so that gravity favours the subsection of the samples without applying additional stresses.
- Whenever possible, it is recommended to use point focus CSP facilities in which the energy supply is vertical, such as vertical solar furnaces or beam down facilities. In the case of horizontal axis solar furnaces, the use of a tilted mirror is recommended, which allows the treatment of the samples in the horizontal plane.

Figure 1 shows a general scheme of the test platform required.





**Figure 1 — Schematic diagram of the solar ageing test bench including a Compound Parabolic Concentrator (CPC) or a solar flux homogenizer**

#### 4.2.3 Post test

Some of the requirements for the end of the test are defocusing and access position to the samples and waiting for the samples to be cooled (mechanical and/or electrical safety lock).

## 5 Procedure

### 5.1 General

Some requirements to carry out solar accelerated ageing tests are:

### 5.2 Flux shape

Solar ageing shall be carried out to guarantee homogenous temperature distribution over the surface bulk of the samples. For this purpose, the following focal solar systems are recommended:

- Solar furnaces, such as SF40 [1], are the most suitable CSP facilities for accelerated ageing of materials using concentrated solar radiation.
- Solar furnaces are optical systems that concentrate solar radiation in such a way that the highest concentration rates are reached in the focus. It is therefore very important to accurately measure the size and distribution of the flux density in the focus as it determines the concentration of heat obtainable within the focal area.
- The solar flux density on the samples is modulated by a shutter system. The flux on the focal plane usually has a Gaussian shape. The use of a compound parabolic concentrator (CPC) or a flux homogenizer are recommended in order to get a homogenous flux. It is of utmost importance that ageing tests are carried out in the most suitable focal plane to obtain a homogeneous solar flux, according to the surface area of the exposed samples.

Other point focus facilities include:

- Solar central receiver facilities: They are devoted to bigger surfaces. It is suggested to operate the heliostat field getting a homogenous distribution of flux map on the samples.
- Parabolic dish facilities: It is recommended to operate in the most homogeneous focal plane, according to the surface of the samples. A CPC or a solar flux homogenizer could be included.
- Point focus Fresnel facilities: Ageing tests should be done in the most suitable focal plane to get a homogeneous solar flux on the samples surface.
- Solar simulators: Solar simulators are testing devices using halogen lamps which offers both an intensity level and a spectral composition close to that of natural sunlight, similar to that of solar radiation to irradiate the samples. Sizes and geometries of samples must be adapted to the focal size of the solar simulator.

It is recommended to measure the flux map with an appropriate equipment, either using indirect methods (camera-target) or direct methods (sensors). The maximum surface exposed to concentrated solar radiation should be adapted to the size of the focus and should not exceed the area where the flux has a homogeneous distribution. The measured flux distribution shall be reported as a result and should be taken into account in the samples post analysis.

NOTE To avoid undesired temperature gradient across the material, structures that enable penetration and volumetric absorption of concentrated solar radiation are recommended in order to obtain a more homogeneous temperature along the height of the exposed sample. An appropriate thermal measurement is recommended. Therefore, it is convenient to optimize the thermal measurement and the heating cycle in order to guarantee homogeneous temperatures along the thickness of the materials being tested.

### 5.3 Heating and cooling rates

The heating and cooling rates should be normally suggested by the material manufacturer. Since ceramics often fail due to the differential expansion, the maximum heating and cooling rate that is well tolerated will be better expressed in term of “maximum thermal gradient along the thickness of the sample” and measured as a value, K/mm, a threshold value not to be overcome.

An experimental method to determine the thermal gradient across the samples under CSP cycling should be established. To make the ageing test condition really reproducible, the thermal gradient along the thickness of the sample during heating and cooling should be kept as constant as possible. In terms of operational procedure and logic path:

- 1) First the solar receiver or ceramic material producer ought to suggest the maximum thermal gradient it is expected to tolerate<sup>1)</sup>, which is taken as a threshold value.
- 2) An experimental method for measuring the thermal gradient through the sample and during the solar ageing test should be defined. Preferably, a non-invasive method should be selected, or another practical way to experimentally measure the thermal gradient through the sample using appropriate sensors should be established. Thermal gradient is calculated as the temperature different between two points divided by the distance between them<sup>2)</sup>.

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1) For instance, LiqTech recommended that in the case of their porous SiC, the maximum thermal gradient along its thickness should not exceed 50-70 K/mm.

2) One of the methods used in NEXTOWER consists of measuring the temperature at different depths of the material through blind holes drilled in the sample, and using thermocouples, in order to obtaining real thermal gradients across the sample. The drawback is that the sample will not be valid for some of the subsequent mechanical analyses because it has been drilled. An alternative is to use another slab (a blind drilled sacrificed slab), which will be aged but it will not be taken into account in the mechanical analyses.

- 3) Based on preliminary tests, the thermal gradient should be below the threshold value, at which the specific ageing is going to be performed. An experimental apparatus could determine the standard deviation around the mean value which it is possible to be achieved. The more spread out a data distribution is, the greater its standard deviation. Thence, the lower the standard deviation is the better, since the result will be more easily compared and reproduced.
- 4) The suggested way of collecting reproducible ageing data is keeping both heating and cooling parameters in such a way that the thermal gradient of each cycle should be minimum and as constant as possible. The measured values could slightly differ anyway, and both thermal gradients are to be reported (expressed as K/mm), together with heating and cooling rates (expressed as K/min). This “thermal gradient” should be kept as constant as possible in all the range of temperature, although it is the thermal gradient at the highest T which it is expected to cause the material failure.

Cooling rate shall be as fast as it is allowed by the material, within the test conditions and the threshold of the sample. A controlled forced cooling is recommended for accelerating solar ageing tests, taking into account the working limits of the material, and without exceeding the thermal gradient along the specimens. An inverter blower will allow a controlled forced cooling.

## 5.4 Number of cycles

The number of cycles for a high frequency test under concentrated solar extreme or controlled conditions comparing with real operation conditions will be estimated by multiplying the number of days envisaged by the number of cycles which can be performed per day, which would correspond to the daily solar cycle itself. To this amount, a correction factor should be added, corresponding to the energy flux transients caused by the presence of clouds.

## 5.5 Temperature measurement

There are different direct and indirect methods for temperature measurement under concentrated solar radiation:

- Direct methods:
  - Thermocouples
  - Phosphor thermometry
  - Fibre optics thermometry (Raman Scattering, Stimulated Brillouin Scattering or femtosecond Fibre Bragg Gratings)
  - Multisensor-resistance thermometry: The system consists of ultrasonic techniques together with single Pt100 sensors
- Indirect methods:
  - Pyrometers
  - Infra-Red (IR) Cameras

NOTE In the frame of NEXTOWER, both thermocouples and IR cameras were used. Further temperature data was collected using a solar blind pyrometer at temperatures exceeding 400 °C.

## 5.6 Incident flux measurement

There are different direct and indirect methods for incident heat flux measurement [1]:

- Direct method: two clearly differentiated ones can be highlighted:
  - Stationary and distributed water-cooled radiometers, installed on the surface of the receiver to be evaluated. Numerous sensors are required to get accurate results.
  - A moving bar equipped with sensors that scan the aperture surface during the movement.
- Indirect method consists of capturing the irradiance distribution on a white moving lambertian target using a high resolution digital camera. In order to measure the physical features of the beam, the system must be calibrated.

New methods are being developed to determine irradiance distributions on receivers and targets, measuring directly on receiver surfaces avoids moving parts, namely:

- A method developed by CIEMAT allows characterizing a large target and quantifying its degree of homogeneity and diffusivity. The measurement system consists of a digital camera and a radiometer, with favourable information about the receiver diffusivity [2].
- The PHLUX method - developed at Sandia National Laboratories - uses a recorded image of the sun, a direct normal irradiance (DNI) reading, and the reflectivity of the target or receiver to calibrate the brightness distribution of target or receiver surfaces [3].

## **6 Test reporting**

A testing result report must be prepared including, at least, the following documentation:

1. Facility description:
  - a) Name of the facility
  - b) Type of facility: Solar furnace, parabolic dish, solar simulator, ...
  - c) Concentration and total power
2. Concentrated Solar flux values: Peak, mean value and flux distribution (flux maps)
3. Temperature and cycles: max. and min. temperatures and number of cycles

## **7 Analysis**

### **7.1 General**

Thermal cycling effects and different ceramics durability (meaning tolerance to thermal ageing conditions) have to be evaluated applying industrial analysis methods and standards, for modelling expected reliability into expected operating conditions.

Failure of ceramics exposed to concentrated solar radiation might be caused by several phenomena, including differential thermal expansion, corrosion, creep and fatigue. Some of these, however, usually prevail over the others and it is this the main cause of final failure, when the fracture toughness is exceeded [4, 5]. In predicting service life, it might be advisable to try establishing each of these degradation mechanisms speed, and specifically it is necessary to determine which is prevailing, by monitoring as many properties as possible. This preliminary analysis about main failure mechanisms is necessary especially in the case of CSP, being virtually impossible to perform service-condition tests which are long enough to be significant. So predictions of service life can only be achieved by performing accelerated tests, which focus and speed up prevailing degradation mechanisms to reliably

simulate the damage evolution of material over time. After a proper correlation of these results, modelling and extrapolation is also needed, to predict lifetime. The comparison between data obtained from normal versus accelerated ageing tests is essential for validation of this model and identify proper statistical treatment of data.

As outlined, this analysis should consider enough samples, for each material and ageing condition, for achieving a representative statistic. Different materials (e.g. state-of-the art and innovative ones) might also be compared in this way, but a special attention should be devoted to main degradation mechanisms, since they might depend on materials and on expected working conditions, making a comparison of the results in the same accelerated ageing tests potentially misleading. Samples with different material composition or microstructure might lead to un-foreseen degradation modes and rates.

## 7.2 Mechanical analyses

For evaluating tolerance to thermal ageing of porous SiC ceramic receivers, the following three standards are applied:

EN 843-1, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength*.

EN 843-2, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 2: Determination of Young's modulus, shear modulus and Poisson's ratio*.

ASTM C 1499, *Standard test method for equibiaxial flexural strength of advanced ceramics at ambient temperature*.

One option is to carry out both flexural strength (nominal fracture strength) and Young's modulus (applying EN 843-1 and EN 843-2, 4-point bending tests on standard samples 4 mm × 3 mm × 45 mm) before and after thermal ageing cut from a ceramic slab (with a geometry 50 mm × 50 mm × 5 mm).

The EN 843-1 standard suggests, for material development, characterization or quality control, a minimum number of 10 test samples. While, for statistical evaluation of data (e.g. Weibull parameters), in accordance with EN 843-5, the minimum number of samples shall be 30.

Another option is to perform equibiaxial flexural strength tests in accordance with the ASTM C 1499, since most engineering applications of ceramics frequently involve biaxial tensile stresses. Equibiaxial tests also minimize the effects of test specimen edge preparation as compared to uniaxial flexural tests. Although the test results of equibiaxial test specimens may not totally represent the strength properties in the entire full-size component or its in-service behaviour in different environments, it provides more reliable data compared to uniaxial flexural strength, considering that a bigger volume of material is being evaluated. ASTM C 1499 standard proposes a test method that covers the determination of the equibiaxial strength, using concentric ring configurations under monotonic uniaxial loading. The relative dimensions of the round test samples and the loading fixture should be chosen to ensure behaviour reasonably described by simple plate theory (in accordance to paragraphs 6 and 8 of ASTM C1499 standard)<sup>3)</sup>.

ASTM C 1499 standard recommends a minimum of 10 specimens validly tested for estimating mean biaxial flexural strength, and a minimum of 30 test specimens validly tested for estimating Weibull parameters.

Regardless to EN 843-1, EN 843-2 or ASTM C 1499, for taking any conclusion about ceramic material reliability for CSP application, it is very important to include in the report the number of standard

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3) In NEXTOWER project, in order to be compliant to ASTM C 1499 standard, a loading fixture with support ring diameter DS = 24 mm and loading ring diameter DL = 10 mm and a machined round test sample with diameter D = 33 mm and thickness h = 2.4 mm, was employed.

samples which broke during machining and to enquire the source of these failures by fractographic analysis.

A special attention should be devoted to position and fracture origin of each sample. EN 843-1 standard suggests correcting the equations to determine nominal fracture strength calculation, according to the origin of fracture (Note 1 pg. 15). Typical fracture patterns in ceramic test samples are explained in Annex 1 of the same standard.

Further guidance for fractography analysis may be obtained from EN 843-6 and ASTM C 1322 standards.

ASTM C 1499 standard suggests reporting, among information about tests, location of fracture in each test specimen centre (if applicable). Fig. 4 (pg. 9) of this standard shows pictures of typical failure patterns in concentric ring test specimens.

### **7.3 Optical analyses**

Solar-energy absorptance, reflectance, and transmittance are important in the performance of all solar energy systems ranging from passive building systems to central receiver power systems. There are several techniques to carry out such analysis:

- Optical characterization of ceramics materials absorbers at ambient using spectrophotometers. Discrete measurements of spectral near normal-hemispherical reflectance are made over the spectral range from 300 to 2 500 nm with an integrating sphere spectrophotometer (ASTM E903 – 12).
- Calculation of thermal emittance of the absorber at nominal temperature by measuring the spectral hemispherical reflectance in the wavelength ranges 2 to 50  $\mu\text{m}$  with an integrating sphere spectrophotometer. The thermal emittance is calculated at any temperature by weighting spectral measurements with the corresponding blackbody radiation spectrum, defined by Planck's law (ISO 22975-3, Annex A).
- Calculation of solar absorptance. The solar transmittance, reflectance, or absorptance is obtained by calculating a weighted average with a standard solar spectral irradiance as the weighting function by either the weighted or selected ordinate method. (IEC 68268-1-1, ASTM G173 and ISO 9050).
- Fourier-transform infrared (FTIR) spectrometers allow materials to be identified quickly and easily in the near, mid and far infrared regions.

At the beginning of the test, after each 3 cycles, and at the end of test, solar absorptance and thermal emittance are calculated.

### **7.4 Thermal oxidation degradation**

The assessment of the mass and dimensional changes of test pieces following oxidation at high temperature in an oxidizing atmosphere is crucial to determine the extent of material degradation after ageing. In addition, it might be convenient to assess whether oxidation has a significant effect on the retained strength, according to ISO 20509:2003, or not. The ability to design and use ceramic components is dependent upon the availability of a sound base of valid materials test data. Such data is particularly useful for materials development, quality control, characterization, and design data generation purposes, and it can only be generated using standard test methods which allow reproducible and comparable results to be obtained. Oxidation kinetics of non-oxide ceramics are often dependent on various key test parameters [6]. In fact, it has been shown that the effect of thermal cycling on the kinetics of oxidation of non-oxide ceramics is usually negligible in view of the fact that cracks formed during cooling from the high temperature, either did not penetrate to the ceramic surface, or that healing occurred quickly upon re-exposure at high temperature preventing an

acceleration in the rate of oxidation. On the other hand, materials used to support test pieces may have a significant influence on the oxidation behaviour. The choice of an appropriate supporting material is critical (e.g. alumina ought to be avoided).

**NOTE** Mass changes recorded by thermogravimetry TGA may be used to measure oxidation rates coupled to mass spectrometer ( $O_2/CO$ ). It is common practice to use TGA analysis to address the influence of key test parameters that are likely to affect oxidation kinetics, namely, the level of humidity of the oxidizing atmosphere, heating/cooling rates, test piece geometry, surface condition, and the presence of sodium and sulphur bearing species (e.g.  $Na_2SO_4$ ).

## 7.5 Microstructural analysis

In addition to the evaluation of the Young modulus (through the impulse excitation technique according to EN 843-2), the normal solar absorptance (ASTM E903 – 12), the oxidation resistance and the flexural strength of the aged samples (ISO 20509:2003), microstructural analyses ought to be carried out in order to assess the effect of corrosion and thermal stresses on the aged materials. To this end, two techniques shall be used for characterizing the aged test-pieces:

- Crystalline phase analysis by means of X-ray diffraction analysis.
- Microstructure by means of Scanning Electron Microscopy (SEM-EDS).

The impulse excitation technique (IET) is a non-destructive characterization technique used to determine the elastic properties of a material. It measures the resonant frequencies in order to calculate the Young's modulus, shear modulus and Poisson's ratio of predefined shapes, such as rectangular bars, cylindrical rods and disc shaped samples according to the EN 843-2 standard. The measurement principle is based on tapping the sample and recording the induced vibration signal with a microphone. The acquired vibration signal is then converted to the flexural and torsional resonance frequency domains by a fast Fourier transformation. IET is mostly used in research and as quality control tool to study the changes in elastic moduli as function of time.

In some cases, it is recommended to assess the material's structure by means of X-ray Computed Tomography (CT). Indeed, it is a non-destructive technique suitable to locate and size planar and volumetric features within solid parts, and for obtaining digital information on their 3-D internal geometries and properties. It is thus an optional technique that can be used for non-destructive evaluation of samples, before or after thermal ageing, as it is capable of visualising internal defects (voids), and cracks that may be obscured by coatings. There is a standard guide to perform such CT analysis (ASTM E1441-19 – Standard Guide for Computed Tomography (CT)). Because the full scan field for CT is a stack of circular fields of view, the optimal geometry to scan is a cylinder, although samples of rectangular section can also be observed quite satisfactory. In addition, little or no sample preparation is required. Major drawbacks include: resolution is limited to about 1 000-2 000x the object field of view, which is usually the largest cross-section dimension; image artefacts can complicate data acquisition and interpretation and the large data volumes (gigabytes+) require considerable computer resources. With suitable resolution, CT can be particularly useful also to provide inputs for modelling the mechanical properties of cellular materials when CT is coupled with the finite element method (FEM).

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