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European foreword

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Introduction

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

Today, the diverse solar thermal energy solutions commercially available differ with respect to concentration technology, receiver type and shape, nature of the heat transfer fluid (HTF) and capability to store thermal energy, to turn it later into process heat or electricity on demand.

However, more cost-effective solutions are required for a wider-scale deployment of the CSP technology.

Novel functional materials and material combinations throughout the manufacturing chain are therefore needed to enhance the efficiency of solar energy harvesting beyond that of the current state-of-the-art technologies.

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.

The main objectives of NEXTOWER are:

1. Durable solar receivers. Develop new mechanically tough and highly thermally conductive ceramic receivers, working under extreme thermal cycling without failure at a maximum operating temperature of at least 800 °C and delivering up to 25 years of continued operation.

2. High temperature steels for thermal storage by liquid lead. Develop coextruded-tubes and liner technologies from proprietary corrosion-resistant alumina forming steels (FeCrAl-alloys SMT) to build high-capacity, high-efficiency lead-based heat storage that can work together with high temperature receivers to supply heat suitable for gas turbines or industrial processes, thus expanding the boundaries of CSP technology.

3. New SOLEAD demo of CSP with lead loop. Set up a full scale CSP demonstrator (SOLEAD) for unprecedented field testing of materials for CSP lead-towers, encompassing a large solar receiver (ca 4 m^2, 100 tiles, up to 500 kWth for parallel testing of several receiver types) interfaced to a single-chamber lead storage pool, in turn connected to a secondary "heat sink".

4. Field testing SOLEAD for 12 months, running with lead at average 700 °C. Full proof at TRL 6 of all materials and for input data for levelized cost of energy (LCOE) and Life Cycle Assessment (LCA) computations.

5. Long term operation assessment. Non-destructive testing and multi-scale modelling are inter-twinned synergycally to optimize resources and provide predictive engineering tools based on unique Imputation by Chained Equations (ICE) multilevel approach.

6. Exploitation and Standardization. NEXTOWER will establish and maintain an exploitation culture throughout the project, treating IPR in a way that maximizes impact, and addressing the integration of NEXTOWER within the standardization system.

Some of the results obtained in NEXTOWER are:

1. Innovative ceramic for high-temperature open volumetric receivers based on all-SiC honeycomb manufactured by 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. Liquid lead as heat transfer fluid as technology transfer from nuclear fission to CSP of high-temperature lead-based thermal fluid.

6. Corrosion resistant alumina forming innovative steels based on: FeCrAl-alloys with better performance than reference commercial ones, getting raw material reduction compared to standard stainless steels and Ni superalloys, and better thermal efficiency.

7. Optimized robotic Gas Metal Arc Welding (GMAW) welding procedures and Submerged Arc Welding (SAW) Strip Cladding for avoiding liquation and solidification cracks, with greater repeatability and faster execution.

This CWA will be one of the standardization results, and both the standards community and NEXTOWER partners will benefit from it.

1 Scope

This document defines the requirements, operation and analysis for very high temperature accelerated ageing of flat ceramic specimens for solar receivers under concentrated solar radiation.

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.

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

IEC TS 62862-3-3, *Solar Thermal Electric plants — Part 3-3: Systems and components — General requirements and test methods for solar receivers*

EN ISO 9488, *Solar energy — Vocabulary*

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

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

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

ASTM E903-12, *Standard test method for solar absorptance, reflectance, and transmittance of materials using integrating spheres*

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

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 Method

4.1 Test pieces or slabs

The test pieces shall be as follow:

4.1.1 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), its thickness should not be too thick 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.

4.1.2 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

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.

4.2.1 Requirements for the test platform

Some of the requirements for the test bench for very high temperature accelerated ageing of flat ceramic specimens or small solar receivers by 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 ~~fracture~~ 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 at whole the 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 subjection 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.

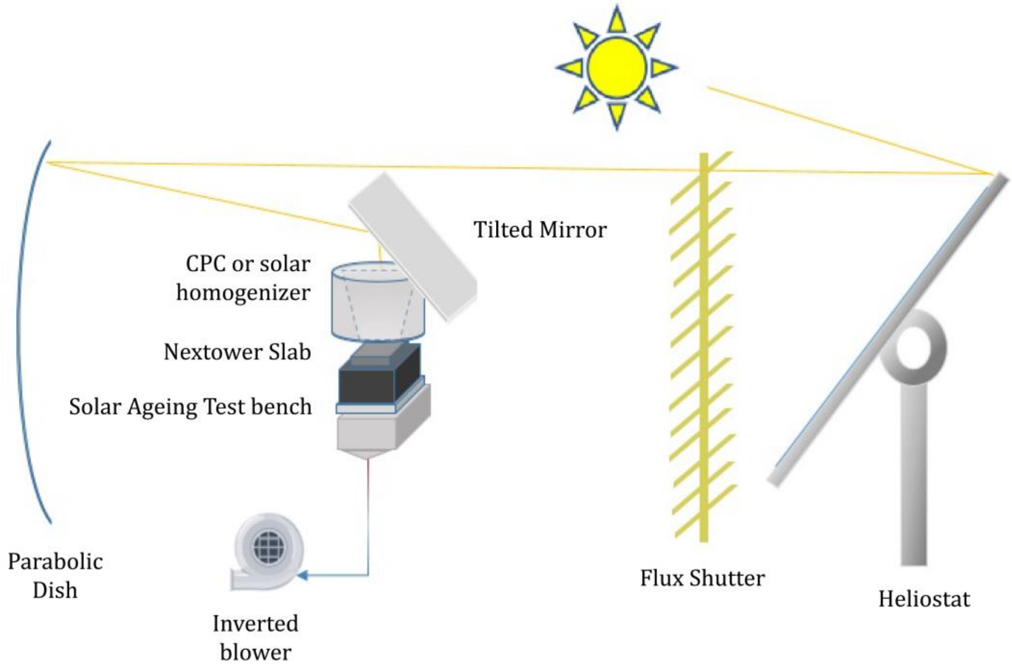

Figure 1 — Schematic diagram of the solar ageing test bench including a Compound Parabolic Concentrator (CPC) or a solar flux homogenizer

4.2.2 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

Some requirements to carry out solar the accelerated ageing tests are:

5.1 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 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 regulated 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 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.

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.

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 distribution. 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.

5.2 Heating and cooling rates

The heating and cooling rates should be normally suggested by the material manufacturer. Since ceramics fails 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, °C/cm, a threshold value not to be overcome.

An experimental method to determine the thermal gradient across the samples under CSP cycling should be established[1)]. 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[2)], which it is taken as a threshold value.

2) A method to experimentally measure the thermal gradient should be defined. By drilling, or other non-evasive method, a practical way to experimentally measure such thermal gradient through the sample ~~is~~ must be established, for instance by using appropriate sensors.

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1) One of the methods used in NEXTOWER consists of measuring the temperature at different depths of the material through blind holes using thermocouples. and obtaining real thermal gradients through the sample. The inconvenience 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 material (a blind drilled sacrificed slab), which is subsequently sacrificed during solar thermal ageing cycles.

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

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 °C/cm), together with heating and cooling rates (expressed as °C/min).

5) 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.

Taking the thermal gradient into account, 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 in the specimens.

5.3 Number of cycles

The number of cycles for an accelerated ageing test 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.

5.4 Temperature measurement

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

- Direct methods:
  - Thermocouples
  - Phosphor thermometry
  - Fiber 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.

5.5 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 (Energy, Environmental and Technological Research Centre in Spain) 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 [1].
- 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 [2].

6 Analysis

It is of utmost importance to carry out the analysis of tested samples that can provide more suitable and reliable information concerning the behaviour and durability of ageing ceramic materials.

It is recommended to take into account the industrial analysis methods for the determination of the durability and degradation of ceramics materials under real operating conditions.

The failure of flat ceramic specimens exposed to concentrated solar radiation can result from several mechanisms including corrosion, creep, fatigue and ultimately fracture when the fracture toughness is exceeded [3]. In predicting service life, it is necessary to establish which of these mechanisms is the slowest, operating during most of the service life, and which is the fastest, causing failure. In case the slowest mechanism is responsible for the damage accumulated, the risk of sudden failure is not so high.

In predicting the lifetime of a component it is the total damage that is of interest. This means that one type of inspection would suffice if the property inspected is related closely enough to the service life. In practice it is advisable to study damage accumulation by monitoring as many properties as can easily and cheaply be measured.

A good knowledge of failure mechanisms is therefore important as well as a clear identification of the active damage mechanisms. In order to predict the changes in properties or the rate of degradation of a given material over a long period of time, it is generally necessary to evaluate critically the applicability of extrapolation of results obtained in service-condition tests. Because of the limited time available for service-condition tests relative to the required service life, such tests have to be complemented by **accelerated tests**, which simulate higher levels of damage experience by the material over its lifetime.

Based on the establishment of reliable correlation between the results of accelerated tests and service-condition tests, the long-term behaviour of materials can be predicted. Therefore, comparison between data obtained from normal ageing tests and those obtained from accelerated ageing tests is essential for this purpose. Statistical treatment of data must be envisaged whenever possible to predict lifetime through extrapolation methods. Hence, there should be enough samples available for statistics. Samples with differences in material composition compared to state of the art may lead to non-foreseen degradation modes and rates.

6.1 Tolerance to thermal ageing

For evaluating tolerance to thermal ageing of porous SiC ceramic receivers, the following two standards were 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.*

At first, 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 of a ceramic slab (with a geometry 50 mm × 50 mm × 5 mm) were evaluated.

After some tests, it seemed more appropriate to also perform equibiaxial flexural strength tests in accordance with the ASTM C 1499 *standard test method for equibiaxial flexural strength of advanced ceramics at ambient temperature*, 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)[3)].

Regardless EN 843-1 and EN 843-2 or ASTM C 1499, for any conclusion about ceramic material reliability for the CSP application, it is very important to include in the report the number of standard samples which broke during machining and to evaluate the source of failure by fractographic analysis.

6.2 Optical characterization analysis

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 transmittance (or reflectance) are

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 = 30 mm and thickness h = 2.4 mm, was employed.

made over the spectral range from 300 to 2500 nm with an integrating sphere spectrophotometer (ASTM E903 – 12).

— 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. (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 µm 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).

— 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.

6.3 Material characterization 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 and the flexural strength of the aged samples (ISO 20509:2003), microstructural analyses ought 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 aging, 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).

6.4 Oxidation kinetic

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 subsequent strength, according to ISO 20509:2003, or not.

NOTE Mass changes recorded by thermogravimetry TGA may be used to measure oxidation rates coupled to mass spectrometer (O_2/CO).

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