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Space systems — Determination of test methods to characterize material or component properties required for break-up models used for Earth re-entry

Systèmes spatiaux — Détermination des méthodes d'essai pour caractériser les matériaux ou les éléments de structure nécessaires pour les modèles de calcul de « désintégration » utilisés pour la rentrée terrestre

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Foreword

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This document was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

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Introduction

New regulations require unmanned spacecraft and launch vehicle orbital stages, called space bodies in this document, to be designed and manufactured in such way that fragments generated during Earth re-entry cannot cause casualties, damage to property or environmental pollution on the ground (see ISO 24113).

Space bodies are submitted to high aero-thermodynamic fluxes, pressures and shear stresses that lead to their disintegration into fragments that can potentially reach ground after a re-entry. These fragments are generated by the effect of aero-thermal loads seen by components that constitute a space body. The assessment of the fragmentation and subsequent survivability of the fragments in terms of size and trajectory is based on simulation.

The methodology to determine the size of the debris is based on an idealized two-step process, called fragmentation and survivability.

- Fragmentation

  Based on the knowledge of the orbital (ballistic) trajectory of the space body and the knowledge of its design, the computation of temperature and stresses determine the most probable failure locations that will generate sub components. The breakup fragments prior to re-entry are termed debris.

- Survivability

  The objective is then to determine if debris can survive (no completely burned), and then if the final size and energy when touching down the Earth are in accordance with the international regulation.

  The computation of the final size and energy of the debris is based on generic geometry definition, homogenized properties and on the knowledge of their trajectories.

For both fragmentation and survivability, suitable thermal response models require a range of material properties for a full characterization of the material response.

Thermal tests used to determine material properties need to be well defined and shared between spacecraft manufacturers and regulation authorities.

There are a range of relevant spacecraft materials, from metals, organic and ceramics to composite materials.

As a result, the material or component properties used in break-up models is an essential model input.

Objects that separate during the ascent phase and impact the ground are addressed in ISO 14620-2.

Assessment, mitigating and control of potential risks created by the re-enter of objects from the orbit are addressed in ISO 27875.

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Space systems — Determination of test methods to characterize material or component properties required for break-up models used for Earth re-entry

1 Scope

This document defines the elementary thermal tests to obtain thermal properties of materials and composite materials used to manufacture space body to support the fragmentation and survivability analysis.

This document does not apply to spacecraft containing nuclear power sources[1].

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1.1
ablation
chemical change and removal of surface *material* (3.1.12) due to the action of external high temperature heating

Note 1 to entry: Ablation can be a chemical process, addressing chemical reaction (gas-solid or gas-liquid).

Note 2 to entry: Ablation can be a physical process addressing vaporization, melting, fusion.

Note 3 to entry: Ablation can be a mechanical process addressing phenomena of erosive process (applied to solid or liquid surface) linked to pressure effect and wall shear stress due to high-speed motion.

[SOURCE: EN 16603-31:2014, 3.2.4.2, modified — Notes 1, 2 and 3 to entry have been added.]

3.1.2
break-up
event that completely or partially destroys an object and generates *space debris* (3.1.18)

Note 1 to entry: Debris generated during earth *re-entry* (3.1.16) can survive and fall to the ground.

[SOURCE: ISO 24113:2019, 3.2, modified — Note 1 to entry has been added.]

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3.1.3

break-up altitude

altitude when the main structural fragmentation occurs leading several *components* (3.1.5)

Note 1 to entry: Altitude *break-up* (3.1.2) can occur on a wide range of values depending on trajectories and attitudes of the *spacecraft* (3.1.19).

3.1.4

ceramic

essentially inorganic and non-metallic *material* (3.1.12)

Note 1 to entry: The concept "ceramic" comprises products based on clay as raw material and also materials which are typically based on oxides, nitrides, carbides, silicides, borides, carbon etc.

[SOURCE: ISO 20507:2014, 2.1.7]

3.1.5

component

part

set of *materials* (3.1.12), assembled according to defined and controlled processes, which cannot be disassembled without destroying its capability and which performs a simple function that can be evaluated against expected performance requirements

[SOURCE: ISO 10795:2019, 3.48]

3.1.6

composite material

combination of *materials* (3.1.12) different in composition or form on a macro scale

Note 1 to entry: The constituents retain their identities in the composite.

Note 2 to entry: The constituents can normally be physically identified, and there is an interface between them.

[SOURCE: ISO 16454:2007, 3.6]

3.1.7

emissivity

ε

quotient of the radiant exitance of a radiator and the radiant exitance of a Planckian radiator at the same temperature, expressed by

$$\varepsilon = M/M_b$$

where M is the radiant exitance of a thermal radiator and M_b is the radiant exitance of a Planckian radiator (called hereafter black body) at the same temperature (ISO 80000-5)

Note 1 to entry: Emissivity of any surface is a function of wavelength, direction, temperature, and surface conditions.

Note 2 to entry: Emittance is a property of a particular object. It is determined by *material* (3.1.12) emissivity, surface roughness, of angle of incidence, oxidation, the sample's thermal and mechanical history, surface finish, and measured wavelength range. Although emissivity is a major component in determining emittance, the emissivity determined under laboratory conditions seldom agrees with actual emittance of a certain sample.

[SOURCE: ISO 80000-7:2019, item 7-30.1, modified — Notes 1 and 2 to entry have been added.]

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3.1.7.1
total directional emissivity
total radiance, L_{Ω}, emitted by the considered surface, divided by total radiance emitted by the black body, $L°_{\Omega}$ at the same temperature

Note 1 to entry: Determined in the domain of near infrared (NIR) and middle infrared (MIR) of the *infrared radiation* (3.1.10).

[SOURCE: ISO 9288:1989, 5.8, modified — Note 1 to entry has been added.]

3.1.7.2
spectral directional emissivity
spectral radiance, $L_{\Omega\lambda}$, of the considered surface divided by the spectral radiance emitted by the black body, $L°_{\Omega\lambda}$, at the same temperature

Note 1 to entry: Determined in the domain of near infrared (NIR) and middle infrared (MIR) of the *infrared radiation* (3.1.10).

[SOURCE: ISO 9288:1989, 5.9, modified — Note 1 to entry has been added.]

3.1.7.3
total hemispherical emissivity
total hemispherical exitance, M, of the considered surface divided by the total hemispherical exitance of the black body, $M°$ at the same temperature

Note 1 to entry: Determined in the domain of near infrared (NIR) and middle infrared (MIR) of the *infrared radiation* (3.1.10).

[SOURCE: ISO 9288:1989, 5.10, modified — Note 1 to entry has been added.]

3.1.8
glass ceramic
inorganic *material* (3.1.12) produced by the complete fusion of raw materials at high temperatures into a homogeneous liquid which is then cooled to a rigid condition and temperature treated in such a way as to produce a mostly micro crystalline body

[SOURCE: ISO 7086-2:2000, 2.11]

3.1.9
glass transition temperature
Tg
characteristic value of the temperature range over which the glass transition takes place

Note 1 to entry: Glass transition temperature characterizes the transition from true solid to very viscous liquid.

Note 2 to entry: The assigned glass transition temperature (*Tg*) may vary, depending on the specific property and on the method and conditions selected to measure it.

[SOURCE: ISO 11357-2:2020, 3.1, modified — Note 1 to entry has been added.]

3.1.10
infrared radiation
electromagnetic radiation of wavelength between 780 nm and approximately 1 mm

Note 1 to entry: For infrared radiation, the range between 780 nm and 1 mm is commonly subdivided into:

- near infrared (NIR): 780 nm to 3 000 nm;
- middle infrared (MIR): 3 000 nm to 50 000 nm;
- far infrared (FIR): 50 000 nm to 1 mm.

Note 2 to entry: These limits are also specified in ISO 20473.

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Note 3 to entry: For *break-up* (3.1.2) *models* (3.1.14), NIR and MIR are the must representative wavelength domains related to *re-entry* (3.1.16) phenomena.

[SOURCE: ISO 9488:1999, 3.9, modified — Notes 1, 2 and 3 to entry have been added.]

3.1.11
launch vehicle orbital stage

complete element of a launch vehicle that is designed to deliver a defined thrust during a dedicated phase of the launch vehicle's operation and achieve orbit

Note 1 to entry: Non-propulsive elements of a launch vehicle, such as jettisonable tanks, multiple payload structures or dispensers, are considered to be part of a launch vehicle orbital stage while they are attached.

[SOURCE: ISO 24113:2019, 3.13]

3.1.12
material

raw, semi-finished or finished purchased item (gaseous, liquid, solid) of given characteristics from which processing into a functional element of the product is undertaken

Note 1 to entry: Gas is not concerned for *break-up* (3.1.2) *models* (3.1.14).

[SOURCE: ISO 10795:2019, 3.148, modified — Note 1 to entry has been added.]

3.1.13
melting point

temperature at which solid changes state from solid to liquid

Note 1 to entry: At the melting point the solid and liquid phases exist in equilibrium for a single substance. The melting point of a solid depends on pressure and is usually specified at standard pressure.

Note 2 to entry: Solids, which differ from single substances and eutectics, do not have a sharp melting point because fusion occurs in a wide temperature range. Therefore, there is a temperature of beginning of fusion called solidus and an end temperature of fusion called liquidus.

Note 3 to entry: Amorphous solids (including many polymers) do not have a sharp melting point. When these pass from a solid to fluid state, they do so over a wide temperature range, centred roughly about the *glass transition temperature* (3.1.9).

Note 4 to entry: The determination method of the melting point used in the *break-up* (3.1.2) *model* (3.1.14) shall be mentioned if it is liquidus state, solidus state or even as an average of solidus and liquidus.

Note 5 to entry: By conservatism, liquidus is preferred in order to state that no solid debris can fall on ground.

Note 6 to entry: Carbon does not melt at any temperature under standard pressure; instead it sublimes around 4 000 K.

3.1.14
model

physical or abstract representation of relevant aspects of an item or process that is put forward as a basis for calculations, predictions, or further assessment

Note 1 to entry: The term "model" can also be used to identify particular instances of the product, e.g. flight model.

[SOURCE: ISO 10795:2019, 3.155]

3.1.15
pyrolysis

chemical change caused by heat

Note 1 to entry: Sometimes used in a more restricted sense to describe chemical changes resulting from heat treatment in the absence of oxygen.

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[SOURCE: ISO 11074:2015, 6.4.33]

3.1.16

re-entry

return of a *spacecraft* (3.1.19) or other space object into the Earth's atmosphere

Note 1 to entry: Several alternative definitions are available for the boundary between the Earth's atmosphere and outer space.

[SOURCE: ISO 10795:2019, 3.197]

3.1.17

reversible specific heat capacity

specific heat capacity determined with DSC where all heat contributions (endothermic and exothermic) due to chemical transformation of the *material* (3.1.12) are not taken into account to measure the specific heat capacity

3.1.18

space debris

non-functional fragments of, or residue from, a space segment element, or launch segment element, in Earth orbit or re-entering the Earth's atmosphere

[SOURCE: ISO 10795:2019, 3.219, modified — The deprecated term has been removed.]

3.1.19

spacecraft

manned or unmanned vehicle designed to orbit or travel in space

[SOURCE: ISO 10795:2019, 3.224, modified — The abbreviated term and Note 1 to entry have been removed.]

3.2 Abbreviated terms

DSC	differential scanning calorimetry
LFA	laser flash analysis
TGA	thermogravimetry analysis

4 Methodology of material characterization

4.1 General

Unmanned spacecraft and launch vehicle orbital stages are made with different components based on metals, polymers, ceramics and composite materials made with polymers, metals and ceramics.

The test matrix (type of test and environmental experimental conditions) shall be determined with respect to the complexity of the break-up model and the re-entry mode that provides the temperature range.

4.2 Temperature range

Depending on the type of re-entry (controlled versus natural) of the spacecraft, one can define two temperature domains associated with high dynamic pressure and aerothermal loads or high energy, respectively.

For controlled re-entry, the entry speed is maximized when the flight path angle allows the temperature to reach over 1 500 K and below approximately 2 500 K.

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For uncontrolled re-entry, the trajectory speed and low flight path angle are naturally decaying slowly.

In these conditions, the theoretical heat fluxes and dynamic pressure can lead to lower break-up altitude of the main parts and consequently the temperature of the parts can be lower than 1 500 K.

Even if the reality is more complex, for practical reasons, two domains of temperature can be defined to specify the temperature range of the test matrix. This temperature boundary of 1 500 K is not fixed but should be adapted to take into account important evolutions of the material and its thermo-physical properties.

4.3 Type of tests

Elementary thermal tests can characterize basic properties, such as thermal conductivity, specific heat capacity and chemical effects inclusive of melt and pyrolysis. These bulk properties are independent of the surface state or any surface interactions with the environmental gas.

In order to produce a reasonable prediction of the survivability of an object during the re-entry, it is necessary to take into account mass and energy transfer occurring between the object surface and the environmental gas. Surface properties and characteristics, such as emissivity, catalicity of the object, shall be measured in representative atmosphere.

The available data is often restricted to a temperature domain, which is generally well below the real or predicted temperature, met by the debris during the terminal re-entry phase.

Technological tests (complementary to elementary tests) are consequently required to extend the domain temperature in a more relevant surface state so that surface mass and energy transfer are ideally well captured.

These technological tests can be divided in two types.

The first type is radiative (generally in the infrared wavelength domain), where a furnace may be used to expose one face of the material sample to highly calibrated pure radiative heat flux in air at ambient pressure or in vacuum. This allows capturing of the material emissivity, the roughness and some chemical effects that contribute to the surface mass and energy transfer. Convective flow effects on the surface phenomena in such a test set-up are not assessed.

The second method to generate high temperature and pseudo flight representative aerothermal loads consists in the use of plasma wind tunnel facilities. These facilities can capture effects such as the material chemical catalicity to the recombination of air molecules at the surface, and the thermo-mechanical flow effects on the material.

4.4 Test conditions

a) Tested samples shall be representative to the materials used in the real part even if the thickness has to be adapted to test devices.

b) Elementary thermal tests shall be performed on test samples of monolithic material to solve the energy balance conservation equation (including Fourier's law in case of multidimensional object) by knowing the density, reversible specific heat capacity, thermal conductivity, glass transition temperature, melting point, and enthalpy of fusion[2]. Total hemispherical emissivity tests are also required[3].

NOTE Thermal conductivity is often determined indirectly with diffusivity measurement. Thermal conductivity is then calculated knowing the density and specific heat.

c) Due to the uncertainties of the measurements of thermo-physical material properties, the number of test samples to identify each thermal property measured in laboratories shall be selected with respect to the expected statistical uncertainty of the material property.

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In case of material scattering, a minimum of 5 measurements for each characteristic determined for each test condition are recommended if the statistic distribution can be fitted by a Gaussian law.

d) Test methods and environmental conditions shall be described.

4.5 Mechanisms of degradation

Degradations are based on different mechanisms: fusion, pyrolysis and chemical ablation. Mechanical ablation is not addressed in this document even if it exists.

a) For metal and metal composite materials, oxidation, melting and chemical ablation shall be considered.

b) For ceramic and ceramic composite materials, oxidation, melting and chemical ablation shall be considered.

NOTE 1 Glass and glass ceramic materials are considered as ceramic materials.

NOTE 2 Carbon materials like graphite are considered as ceramic materials.

NOTE 3 Carbon/carbon composite materials are considered as ceramic composite materials.

c) For organic and organic composite materials, melting, pyrolysis and chemical ablation driven by oxidation shall be considered.

NOTE 4 It is assumed that non-reinforced organic materials are fully consumed during any re-entry conditions but often decompose endo-thermally and as such can act to postpone the degradation of spacecraft components.

5 Definition of the elementary tests

5.1 General

a) All relevant thermo-physical material properties shall be characterized under well-defined laboratory conditions (temperature, heating rate, pressure, gas composition, number of tests for each condition).

b) Elementary test measures shall be performed at atmospheric pressure and under inert gas or high vacuum conditions.

For foamy or fibrous material, pressure and nature of the environmental gas can affect diffusivity measurement. In that case, measured diffusivity or conductivity includes conductivity and/or convective terms due to the gas transport properties.

c) In order to gain in representativeness, thermal properties shall be measured in atmospheric gas pressure in accordance with expected break-up altitude.

NOTE 1 Initial conditions of re-entry are not well defined due to the global uncertainties on the atmosphere, attitude and flight behaviour. An error made at this stage does not influence the critical failure event, which occurs at high temperatures. Therefore, characterization of the material at the beginning of the re-entry, which is below 300 K, is not mandatory and is not worth doing.

NOTE 2 Based on the currently available observation of natural re-entry of spacecraft or launch vehicle orbital stage, the altitude of 78 km can be taken as a reference altitude to specify the environmental conditions that need to be reproduced on ground to have a fair representability of the tests.

NOTE 3 Room temperature can be considered as the minimum temperature for the characterization matrix even if during operational orbit, the initial temperature can be very low.

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The maximum temperature test is determined:

- when the degradation threshold generally obtained via mass loss measurement is reached for organic material and composite;
- until the melting point for metal and composite with metal matrix is reached;
- until the glass transition temperature for glass and composite with glass is reached;

  NOTE 4 Choosing melting point as a fragmentation/demisability criterion is a conservative approach. When viscosity is low enough compared to the aerodynamic shear stress at the wall, the liquid layer is blown off. This thermal physical mechanism can only be characterized with non-elementary thermal tests. Glass behaves like simple liquid at temperature where viscosity is lower than 10 Pa·s.

  NOTE 5 Thermal characterization of the material close to the melting temperature is difficult, this is why the characterization at the glass transition temperature is chosen.

- until the melting point for ceramic and composite with ceramic matrix is reached.

  NOTE 6 These measures are generally done under low velocity in inert gas flow.

d) The geometry and the dimensions of the test samples are defined in the recommended standards reported in Clauses 6, 7 and 8.

5.2 List of the elementary tests

a) The following tests shall be performed in an appropriate temperature domain depending on the family of materials:

   1) Density: at room temperature.

      NOTE 1 Density at elevated temperatures is determined from density measured at room temperature corrected by thermal expansion. Linear thermal expansion from room temperature to the maximum temperature or melting temperature is obtained via thermal mechanical analyser (dilatometer).

      NOTE 2 Measure of the density at high temperature is justified for high thermal expansion materials.

   2) Specific heat capacity from room temperature to maximum temperature.

   3) Heat of fusion in the melting range.

   4) Heat of solid-state phase transitions before solidus.

   5) Thermal diffusivity from room temperature to maximum temperature.

   6) Thermal conductivity from room temperature to maximum temperature

   7) Total hemispherical emissivity from room temperature to maximum temperature in representative conditions of pressure and gas atmosphere.

b) Thermal diffusivity, conductivity and bulk density shall be determined in steady-state conditions.

c) The evolution of total hemispherical emissivity of the exposed surface shall be measured in case of mechanisms of oxidation.

   NOTE 3 The required emissivity data is the total (integrated over wavelengths) hemispherical (integrated over all directions above the surface) emissivity as a function of temperature. Typical measurements are made either on a total or spectral basis, with the spectral data being integrated over all measured wavelengths to obtain total properties.

d) Because the total hemispherical emissivity is linked to the surface state (roughness, oxide coating) and its evolution during flight condition, these properties shall be measured in a relevant oxidized state. It is recommended to measure the total hemispherical emissivity in post-test state that is more relevant than the pre-test state.

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NOTE 4 Post-test measurements can give only indication and are not representative of the real time surface state (roughness, oxide nature, temperature).

NOTE 5 The most representative surface state can be achieved in arc-jet heated wind tunnel, inductive plasmatron or in microwave plasma where conditions of heat exchange with temperature, pressure, flow and dissociated air are closer to real re-entry conditions. Hence, various degrees of surface state change can be analysed during the emissivity test campaign. Contribution of chemical effect can also be captured when the material is exposed to highly calibrate pure radiative heat flux in air at ambient pressure or in vacuum.

e) The total hemispherical emissivity coefficient shall be provided with the condition of the surface state and gaseous environment.

f) When the total hemispherical emissivity cannot be measured directly, the reference of the type of measure (total directional emissivity, spectral directional emissivity) and the method to obtain total hemispherical emissivity shall be provided with the emissivity value.

NOTE 6 Obtaining the total hemispherical emissivity at high temperatures is difficult. Typically, only directional data at specific viewing angles (normal, near normal or at an angle between 45° and 60°) and in particular surface state is generated at high temperatures. The calculation of hemispherical emissivity from the total directional emissivity is an approximation. Usually, the ratio of measured hemispherical to directional emissivity at room temperature is assumed to be applicable at higher temperatures[4]. Therefore, the directional total emissivities measured at elevated temperatures are multiplied by the room-temperature ratio of hemispherical to directional emissivity[5].

g) The environmental conditions, such as oxygen partial pressure, gas flow rate and surface state (roughness before and after test) shall be mentioned.

NOTE 7 Verification of the surface state (roughness) after the test can indicate any evolution of the surface (oxidation) that can occur during the test.

5.3 Composite materials specifications

Composite materials are heterogeneous and anisotropic materials.

a) Measurements shall be done in different directions for thermal expansion, thermal diffusivity and thermal conductivity.

b) When thermal diffusivity or conductivity are measured in temperatures different from room temperature, the geometrical correction applied to the apparent measurements, due to thermal dilatation of the sample, shall be explicitly stated.

c) Anisotropic properties are required for advanced models dealing with fragmentation.

NOTE Linear thermal expansion in different directions is measured to calculate density at high temperatures.

d) If specific means or test methodology are used because of material features or type of test conditions, this information shall be provided with material properties. Scattered values are observed depending on the test set-up and sample geometry and preparation.

e) The size of the samples shall be defined according to heterogeneity of the material and the fibrous architecture.

f) The dimensions of the test set-up shall be in agreement with the representative dimension of the fibrous architecture of the composite in order to minimize scattering of measurement generated by the heterogeneity.

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6 Metallic and metallic composite materials

6.1 Test conditions

a) Density, linear thermal expansion, reversible specific heat capacity and thermal diffusivity or thermal conductivity, total hemispherical emissivity shall be measured up to the melting point.

b) For materials with high melting temperatures, the maximum temperature can be limited by the standardized experimental set-up. Therefore, the value at high temperatures shall be extrapolated and labelled as extrapolated measure including the extrapolation methodology (constant, linear, polynomial).

NOTE 1 Standardized methods to determine thermal properties of liquid metals are practically non-existent.

NOTE 2 For refractory materials (e.g. platinum, tungsten, tantalum, some carbides and nitrides) the extremely high melting point (typically above 2 000 K) can be determined by heating the material in a black-body furnace and measuring the black-body temperature with an optical pyrometer.

Several methods, such as the oscillating viscometer, the levitated drop apparatus to measure surface tension, density and viscosity, and the laser flash to measure thermal diffusivity and reversible specific heat capacity, are being used.

c) Reversible specific heat capacity shall be measured from room temperature to the first solid-liquid transition.

NOTE 3 These measurements are done at a slow heating rate of about 0,083 K/s.

d) Heat of fusion and the solid fraction shall be measured from solidus temperature up to the complete fusion of the material under inert atmosphere with pristine material.

e) Linear thermal expansion is measured under inert atmosphere.

NOTE 4 These measurements are done with a dilatometer at a slow heating rate of about 0,083 K/s.

f) Thermal diffusivity is measured with LFA under inert atmosphere.

g) Total hemispherical emissivity shall be measured up to solidus temperature on pristine material in inert atmosphere at least in the infrared wavelength range.

h) Total emissivity of the melting phase shall be measured if it is different from the solid material and shall be measured in the same conditions.

NOTE 5 Some metals can react at high temperatures with molecular nitrogen and can affect emissivity measures.

NOTE 6 It is important to consider phase transition in metal composites (e.g. beta- transition of Ti6Al4V).

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6.2 List of the recommended standards

- Density: ISO 3369 and ISO 2738
- Specific heat capacity: ASTM E1269 – 11 and ASTM E2716 – 09
- Thermal conductivity: ASTM C 518-98
- Thermal diffusivity: ASTM E1461 – 13
- Emissivity: ISO 16378, ASTM C835-06
- Thermal expansion: ASTM E228 – 17, ASTM E289 – 17 and ASTM E831 (2019)
- Heat of fusion: ASTM E793-06 (2018) and ASTM E794-06 (2018)

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7 Organic and organic composite materials

7.1 Organic materials

7.1.1 General

Organic materials address the family of thermosetting polymers and thermoplastic polymers, also named plastic, and also natural based material such as wood, hemp, flax.

7.1.2 Test conditions

If charred material is removed under aerodynamic flow, it is not necessary to characterize pyrolysis phenomena. Characterization is limited to density, reversible specific heat, melting enthalpy and conductivity.

- Thermosetting polymer

Thermosetting polymers decompose with temperature instead of melting.

- Thermoplastic polymer

Thermoplastic polymers melt at high temperatures before decomposition, even if at high temperatures decomposition can occur simultaneously with or before melting.

- Natural based material

Natural based materials decompose with temperature.

7.2 Organic composite materials

7.2.1 General

Organic composite materials made with a matrix of thermosetting polymers, thermoplastic polymers or natural based materials are reinforced with organic materials (aramid, natural based materials) or inorganic materials (carbon, ceramic). During the re-entry, they can be in three states: virgin state, reacting state and charred state.

Virgin state is reached when the mass loss measured by thermogravimetric analysis under inert atmosphere of extreme purity is less than 5 %. Mass loss is measured by TGA set-up under inert atmosphere.

Reacting state is defined as an unsteady state where the micro-structure of the material (mass and elementary composition) is evolving under thermal decomposition, a process known as pyrolysis. The evolution of the thermal and physical properties during decomposition is not addressed in this document.

Charred state is reached when the mass loss is stabilized after pyrolysis heat treatment in inert atmosphere of extreme purity. Mass loss is measured by TGA set-up. The thermal decomposition testing is performed at a wide range of heating rate in order to obtain decomposition kinetic constants that are the closest to the flight conditions.

7.2.2 Test conditions in virgin state

a) Density, linear thermal expansion, reversible specific heat capacity and thermal diffusivity or thermal conductivity, total hemispherical emissivity shall be measured up to the beginning of the thermal degradation of the sample.

b) The heating rate shall be chosen to minimize the thermal gradient in the sample.

NOTE Typical heating rate is between 0,083 K/s to 0,333 K/s.

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c) The size of the sample shall be sufficiently large with regard to the heterogeneity scale of the material.

7.2.3 Test conditions in charred state

In order to measure the properties in the charred state, the sample shall be charred at the maximum heating rate that does not lead to structural damage and that allows easy handling.

a) Density, linear thermal expansion, reversible heat capacity, thermal diffusivity or thermal conductivity and total hemispherical emissivity shall be measured up to maximum temperature.

NOTE 1 Typical heating rate is between 0,083 K/s to 0,333 K/s.

NOTE 2 Maximum temperature at laboratory scale can reach 2 273 K.

Any extrapolation of the property value shall be defined by the extrapolation method.

b) The heating rate shall be chosen to minimize thermal gradient in the sample.

c) The size of the sample shall be sufficiently large with regard to the heterogeneity scale of the material.

7.3 List of the recommended standards

- Density: ISO 1183-1 and ASTM E792-13
- Specific heat capacity: ISO 11357-4, ASTM E2716 – 09 and ASTM E1269 – 11:
- Thermal conductivity: ISO 22007-2 and ASTM D5930-97
- Thermal diffusivity: ISO 22007-4 and ASTM E1461 – 13
- Emissivity: ISO 16378
- Thermal linear expansion: ISO 11359-2, ASTM E228 – 17, ASTM E289 – 17 and ASTM E831 (2019)
- Mass loss: ISO 11358-1

8 Ceramic and associated composite materials

8.1 Test conditions

a) For ceramic and associated composite materials, density, linear thermal expansion, reversible specific heat capacity and thermal diffusivity or thermal conductivity and total hemispherical emissivity shall be measured up to the melting temperature or sublimation for representative environmental conditions (temperature and pressure).

b) Glassy material can exhibit a semi-transparent molten layer at the surface at high temperature and under low wall shear stress. In that case, care must be taken to account for the not well-defined geometry of the interface and the real bulk radiative transfer for which emissivity cannot be strictly defined.

NOTE 1 Ceramic and ceramic composite materials react chemically with air. The most representative surface state can be achieved in an arc-jet heated wind tunnel where conditions of heat exchange with temperature, pressure, flow and dissociated air are closer to real re-entry conditions. Hence, various degrees of surface state change can be analysed during the emissivity test campaign.

NOTE 2 Linear thermal expansion is measured at a heating rate of 0,083 K/s.

b) For ceramic and associated composite materials, the heat of fusion and melting temperature shall be measured under inert atmosphere with pristine material.

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NOTE 3 For glass, glass ceramic and associated composite materials, the properties can be limited to glass transition temperature.

NOTE 4 These measurements are done with DSC at a slow heating rate between 0,083 K/s and 0,333 K/s.

c) If the melting temperature cannot be reached with the experimental set-up, the value at the melting temperature shall be extrapolated and labelled as extrapolated measure including the extrapolation methodology (constant, linear, polynomial).

8.2 List of the recommended standards

8.2.1 Ceramic

- Density: ISO 18754
- Specific heat capacity: ISO 19628, ASTM E1269 – 11 and ASTM E2716 – 09
- Thermal conductivity: ISO 8302
- Thermal diffusivity: ISO 18755 and ASTM E1461 – 13
- Emissivity: ISO 19618, ISO 16378 and ASTM E423-71
- Thermal linear expansion: ISO 17139, ISO 17562, ASTM E289 – 17 and ASTM E831 (2019)
- Glass transition: ISO 7884-8
- Heat of fusion: ASTM E793-06 (2018) and ASTM E794-06 (2018)

8.2.2 Ceramic composite

- Density: ISO 18754
- Specific heat capacity: ISO 19628
- Thermal diffusivity: ISO 19629
- Emissivity: ISO 16378 and ASTM E423-71
- Thermal linear expansion: ISO 17139, ISO 17562 and ASTM E831 (2019)
- Glass transition: ISO 7884-8
- Heat of fusion: ASTM E793-06 (2018) and ASTM E794-06 (2018)

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