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Test method for CMAS corrosion of thermal/environmental barrier coatings under dynamic thermal cycling

Méthode d'essai de la corrosion par les CMAS des systèmes barrières thermiques/environnementales dans le cadre d'un cyclage thermique dynamique

Reference number
ISO 21465:2025(en)

Contents

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document provides the test method for calcia–magnesia–aluminosilicate (CMAS) corrosion of thermal/environmental barrier coatings (T/EBCs) under dynamic thermal cycling. The CMAS corrosion behaviour affects the performance and service life of the T/EBCs. The multi-layer structure of the T/EBC is deposited on Ni-superalloys/SiC-based ceramic substrates using different methods such as atmospheric plasma spraying (APS), plasma spray-physical vapour deposition (PS-PVD), electron beam physical vapour deposition (EB-PVD), high-velocity oxygen fuel (HVOF). Therefore, the deposition methods and thickness of T/EBCs should meet the requirements of service conditions.

CMAS can be in the form of airborne sand, runway debris or volcanic ash in aircraft engines and ambient dust or fly ash in power generation engines. Gas turbine engines are attacked by the CMAS when the aerospace spacecraft or aircraft flies above desert and volcanic areas. The diffusion, reaction and viscosity of the molten CMAS can cause serious corrosion of T/EBC, resulting in the T/EBC's spallation and failure. Consequently, the operation lifetime of the gas turbine is reduced. Therefore, the behaviour of CMAS corrosion of T/EBCs is an important assessment index of T/EBCs performance. A unified international test standard is required to evaluate CMAS corrosion of thermal/environmental barrier coatings (T/EBCs) under dynamic thermal cycling. This document aims to formulate a standardized and unified test method, including the process and the failure determination criteria, for the performance of T/EBCs.

Test method for CMAS corrosion of thermal/environmental barrier coatings under dynamic thermal cycling

1 Scope

This document specifies requirements for the test method of the CMAS corrosion of thermal/environmental barrier coatings under dynamic thermal cycling, including the process and the determination of failure after corrosion.

The document does not apply to such coatings on plastics to be used for aerospace, electronics and other engineering fields.

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.

ISO 13123, *Metallic and other inorganic coatings — Test method of cyclic heating for thermal-barrier coatings under temperature gradient*

ISO 14188, *Metallic and other inorganic coatings — Test methods for measuring thermal cycle resistance and thermal shock resistance for thermal barrier coatings*

ISO 18555, *Metallic and other inorganic coatings — Determination of thermal conductivity of thermal barrier coatings*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13123, ISO 14188, and ISO 18555, and the following apply.

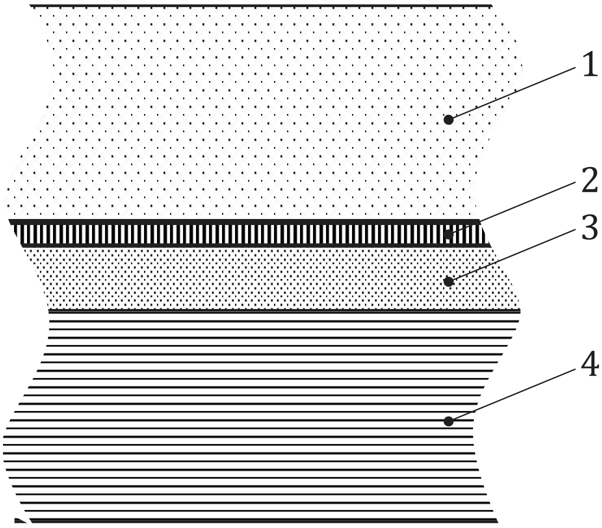
ISO and IEC maintain terminology databases for use in standardization at the following addresses:

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

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

3.1
thermal/environmental barrier coating
T/EBC
protective coating on the superalloy or SiC-based substrate to reduce the heat transfer from the outside topcoat layer through the coating to the substrate

Note 1 to entry: T/EBC inhibits the oxidation of the substrate, increases the operation temperature and improves the service life of the substrate when exposed to harsh environments, such as air, water vapour and molten *calcia-magnesia-aluminosilicate* (3.2) conditions. Generally, a bond coat layer is placed between the T/EBC and substrate to mitigate the coefficient of thermal expansion incompatibilities. Figure 1 shows the schematic diagram of the T/EBC on the substrate. The T/EBC is sprayed on the bond coat layer using APS, PS-PVD, EB-PVD, HVOF, etc.



Key

1 thermal/environmental barrier coating (T/EBC)

2 thermally grown oxide (TGO)

3 bond coat (BC)

4 substrate

Figure 1 — Diagrammatic representation of a section of T/EBC

3.2
calcia–magnesia–aluminosilicate
CMAS
mixture consisting of CaO, MgO, Al_2O_3 and SiO_2

Note 1 to entry: CMAS can be in the form of airborne sand, runway debris or volcanic ash in aircraft engines and ambient dust or fly ash in power generation engines.

3.3
dynamic thermal cycling
system comprising the heating, holding and cooling process

3.4
ratio of spalling area
proportion of the total spalling area relative to the effective area of the *thermal/environmental barrier coating (T/EBC)* (3.1), which is used to determine the failure of the T/EBC

4 Principle

The CMAS powder is coated on the surface of the T/EBCs, or a CMAS suspension is injected into a suitable flame and spread on the surface of the T/EBCs. One dynamic thermal cycle comprises the heating, holding and cooling processes. After cooling to room temperature using compressed air, the ratio of spalling areas is calculated to determine the failure of T/EBCs due to CMAS corrosion.

5 Test methods

5.1 CMAS corrosion under dynamic thermal cycling without thermal gradient

5.1.1 CMAS composition

The basic constituents of CMAS powder are CaO, MgO, Al_2O_3 and SiO_2. The composition of CMAS is uncertain and depends on different geographical locations. The compositions for the test shall be 22CaO-19MgO-7Al_2O_3-45SiO_2 or 33CaO–9MgO–13$AlO_{1.5}$–45SiO_2; however, the CMAS composition can also be adjusted through simulation, considering the natural variations in CMAS compositions observed in different geographical locations (expressed in mole fraction). Weigh the raw materials based on the above stoichiometric ratios. Mix 100 g of CMAS powder with 100 ml of ethanol and ball mill using ZrO_2 balls at 300 r/min for 4 h to 8 h. Dry the slurry at 90 °C to 100 °C for 2 h to 4 h to obtain the dried powders. Afterwards, place the powder on Pt-flakes and heat at 1 200 °C to 1 250 °C for 5 h to 10 h in an Al_2O_3 tube furnace or muffle furnace, then cool down to room temperature. Finally, grind the CMAS in a mortar and sieve with 200 to 400 mesh to obtain CMAS powders with a diameter of 30 µm to 120 µm.

5.1.2 CMAS coating

Apply the uniformly dispersed CMAS in ethanol on the surface of the T/EBC. Dry it in an oven at 80 °C for 2 h to 4 h to remove the ethanol and moisture. Measure the weight of each sample before and after the CMAS coating. Repeat the process until the loading of the CMAS powder is approximately 10 mg/cm^2 to 30 mg/cm^2.

5.1.3 Test method

The test is carried out in a constant temperature muffle furnace at 1 250 °C. Insert the CMAS-coated T/EBC specimen into the furnace by the displacement control system, then heat at a constant temperature for 5 min. Then pull out and cool the sample to room temperature for 5 min to complete a dynamic thermal cycle. Inspect the sample surface and repeat the test cycle if required. Calculate the ratio of spalling areas to determine the failure of T/EBCs attacked by CMAS. This test is easy to conduct in the muffle furnace. However, the surface temperature of T/EBC shall not exceed the superalloy substrate service temperature. An example of the microstructure of the T/EBC specimen after the CMAS corrosion attack is given in Annex A.

— For Ni-based superalloy substrate, the surface temperature of T/EBC should be lower than 1 300 °C.

— For SiC-based ceramic substrate, the surface temperature of T/EBC can be higher up to 1 500 °C.

5.1.4 Detection of the accurate temperature

The test temperature is monitored with thermocouples, which are connected to the substrate. Since the test temperature is higher than 1 250 °C, R or S-type thermocouples should be chosen.

5.1.5 Determination of failed samples

Repeat the heating and cooling processes until 20 % of the surface peels off. The damaged surface shall be observed through an optical or scanning electron microscope. Calculate the service life of T/EBC attacked by the molten CMAS at elevated temperatures as follows:

a) Measure the area of the T/EBC sample under test, S_1.

b) Calculate the remained T/EBC area after corrosion by molten CMAS, S_2.

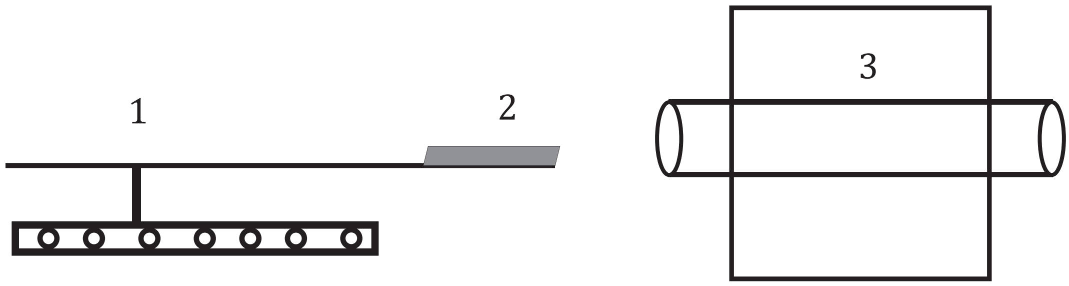
c) Calculate the peeled area ($S_1 - S_2$).

d) Calculate the peeling ratio $(S_1 - S_2)/S_1 \times 100$

A peeling ratio of ≥20 % means the failure of T/EBC after corrosion by molten CMAS.

5.1.6 Apparatus

The apparatus consists of a displacement control system and a muffle furnace (see Figure 2).



Key

1 displacement control system

2 sample

3 muffle furnace

Figure 2 — Devices for the CMAS corrosion test under dynamic thermal cycling without thermal gradient

5.2 CMAS corrosion under dynamic thermal cycling with thermal gradient

5.2.1 CMAS suspension and CMAS precursor solution

The CMAS suspension is composed of CMAS powder (see 5.1.1) and deionized water. The composition of the CMAS precursor is colloidal silica-$Ca(NO_3)_2{\cdot}4H_2O$-$Mg(NO_3)_2{\cdot}6H_2O$-$Al(NO_3)_3{\cdot}9H_2O$-$NaNO_3$-KNO_3-$Fe(NO_3)_3{\cdot}9H_2O$. The composition of the CMAS precursor should be $50SiO_2$(colloidal)-$38Ca(NO_3)_2{\cdot}4H_2O$–$5Mg(NO_3)_2{\cdot}6H_2O$-$4Al(NO_3)_3{\cdot}9H_2O$-$1NaNO_3$-$1KNO_3$-$1Fe(NO_3)_3{\cdot}9H_2O$. The other important composition of CMAS, located in different countries or regions, is also acceptable. The error of CMAS composition shall be within 0,1 % (in mole fraction). The water-based CMAS precursor solution comprises CMAS precursor and distilled water.

5.2.2 CMAS concentrations

The CMAS powder is suspended in deionized water, and the concentration of CMAS suspension is 0,1 % to 0,3 % (in mass fraction). The water-based CMAS precursor solution is diluted in distilled water to an overall concentration of 0,1 % to 0,3 % (in mass fraction).

5.2.3 CMAS injection rate

The injection rate of CMAS with the water-based precursor solution shall be 1,2 g/min ± 0,1 g/min.

5.2.4 Test method

Fix the T/EBC coated substrate on the sample stage. Heat the surface of T/EBC using the flame and spread the CMAS suspension or precursor solution on the surface of the T/EBCs through the CMAS injection system at the same time. Cool the back side of the sample (substrate side) with compressed air. Thus, a temperature gradient is formed between the T/EBC surface and substrate.

Measure the surface temperature of the T/EBC with an infrared pyrometer and the back side of the substrate with a thermocouple. Special care has to be taken due to the changing emissivity due to the CMAS coverage of the surface.

This test mode is close to the actual service environment of T/EBC attacked by the molten CMAS since the T/EBC surface temperature exceeds the melting point of CMAS, and the substrate temperature meets the service temperature. However, this test mode is relatively difficult.

In this case, the T/EBC surface temperature can reach 1 500 °C since the rear side of the substrate is cooled by compressed air.

5.2.5 Heating temperature and time

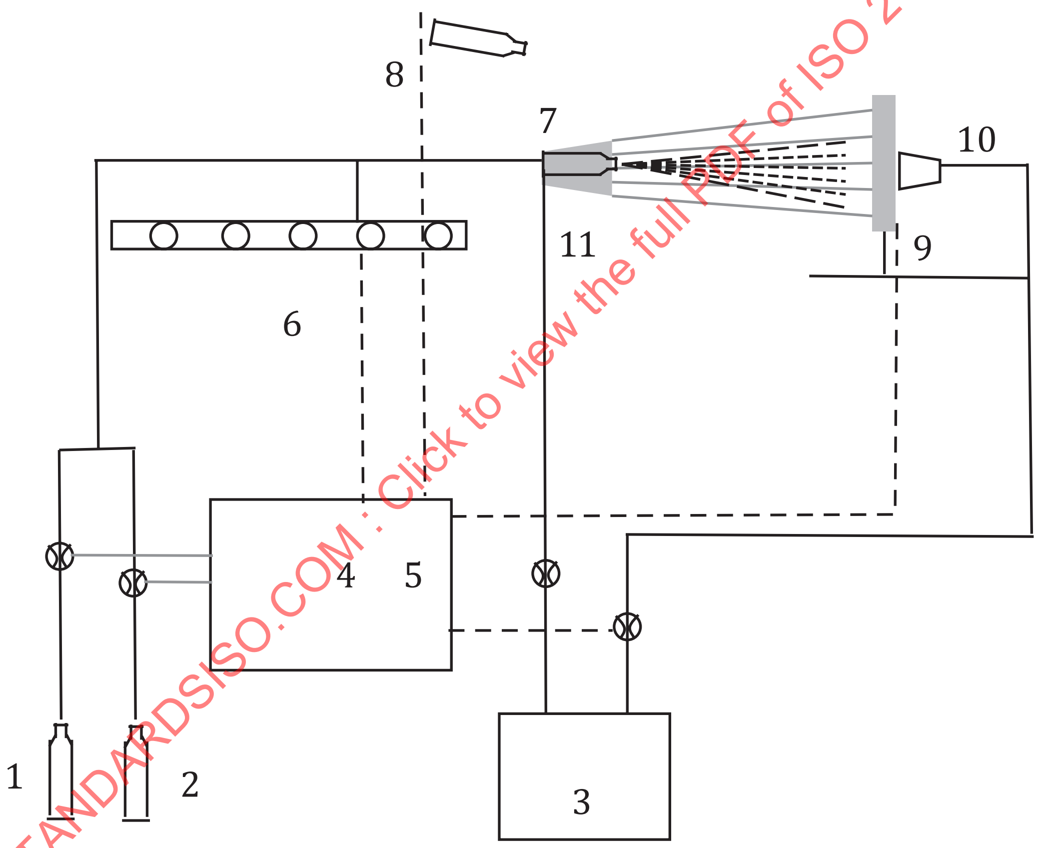
The surface temperature of the T/EBC shall not be less than 1 250 °C or higher than 1 500 °C, and the holding time is 5 min for each cycle. Afterwards, remove the flame, stop the CMAS injection and cool down to room temperature using compressed air.

5.2.6 Determination of failed samples

Inspect the sample surface until 20 % of the surface area peels off to determine the sample failure (see 5.1.5).

5.2.7 Equipment design

The equipment consists of heating, cooling, displacement and CMAS injection systems, as shown in Figure 3. Furthermore, the injection through an additional nozzle in the centre can be added to support the optimal heating of the CMAS if necessary.



Key

1	C_3H_8, C_2H_2: LPG	7	heating system
2	O_2	8	infrared pyrometer
3	cooling system	9	thermocouple
4 and 5	control system	10	compressed air
6	displacement system	11	CMAS injection system

Figure 3 — Schematic of CMAS corrosion test setup for dynamic thermal cycling with thermal gradient

6 Test report

The test report shall contain the following information:

a) general information: test principle and the test method used;

b) a reference to this document, i.e. ISO 21465:—;

c) T/EBC sample information:

— topcoat or coating material;

— substrate material;

— coating method;

— shape and dimension of the test piece;

— thickness of T/EBC;

— bond coat material and thickness;

d) test equipment and parameters:

— test temperature;

— composition and concentration of the CMAS powder and suspension;

— CMAS powder/suspension loading method;

— CMAS suspension injection rate;

— holding temperature and time;

— number of thermal cycles up to the 20 % peeling tolerance limit of the top coat;

— photo or illustration showing the top coat damage pattern of the test piece;

e) all operating details not specified in this document, or regarded as optional, together with details of any incidents which can influence the test result(s);

f) any deviations from the procedure;

g) any unusual features (anomalies) observed during the test;

h) the date of the test.