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Standard Practice for Combined, Simulated Space Environment Testing of Thermal Control Materials with Electromagnetic and Particulate Radiation[1]

This standard is issued under the fixed designation E512; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Spacecraft thermal control coatings may be affected by exposure to the space environment to the extent that their radiative properties change and the coatings no longer control temperatures within desired limits. For some coatings, this degradation of properties occurs rapidly; others may take a long time to degrade. For the latter materials, accelerated testing is required to permit approximate determination of their properties for extended flights. The complexity of the degradation phenomena and the inability to characterize materials in terms of purity and atomic or molecular defects make laboratory exposures necessary.

It is recognized that there are various techniques of investigation that can be used in space environment testing. These range in complexity from exposure to ultraviolet radiation in the wavelength range from 50 to 400 nm, with properties measured before and after testing, to combined environmental testing using both particle and electromagnetic radiation and in situ measurements of radiative properties. Although flight testing of thermal control coatings is preferred, ground-based simulations, which use reliable test methods, are necessary for materials development. These various approaches to testing must be considered with respect to the design requirements, mission space environment, and cost.

1. Scope

1.1 This practice describes procedures for providing exposure of thermal control materials to a simulated space environment comprising the major features of vacuum, electromagnetic radiation, charged particle radiation, and temperature control.

1.2 Broad recommendations relating to spectral reflectance measurements are made.

1.3 Test parameters and other information that should be reported as an aid in interpreting test results are delineated.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E296 Practice for Ionization Gage Application to Space Simulators

E349 Terminology Relating to Space Simulation

E434 Test Method for Calorimetric Determination of Hemispherical Emittance and the Ratio of Solar Absorptance to Hemispherical Emittance Using Solar Simulation

E490 Standard Solar Constant and Zero Air Mass Solar

[1] This practice is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.04 on Space Simulation Test Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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Spectral Irradiance Tables
E491 Practice for Solar Simulation for Thermal Balance Testing of Spacecraft
E903 Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbed dose*—the amount of energy transferred from ionizing radiation to a unit mass of irradiated material.

3.1.2 *absorbed dose versus depth*—the profile of absorbed energy versus depth into material.

3.1.3 *bleaching*—the decrease in absorption of materials following irradiation because of a reversal of the damage processes. This results in a reflectance greater than that of the initially damaged material. Also referred to as *annealing*.

3.1.4 *equivalent ultraviolet sun (EUVS)*—the ratio of the solar simulation source energy to a near ultraviolet sun for the same wavelength region of 200 to 400 nm.

3.1.5 *far ultraviolet (FUV)*—the wavelength range from 10 to 200 nm. Also referred to as *vacuum ultraviolet* or *extreme ultraviolet*.

3.1.6 *far ultraviolet sun*—the spectral and energy content of the sun in the wavelength range from 10 to 200 nm. The spectrum is characterized by a continuum spectrum to approximately 160 nm and a line spectrum to 10 nm. The solar energy in the FUV fluctuates and for purposes of irradiation of thermal control coatings, the UV sun is defined as 0.1 W/m^2 for the wavelength range from 10 to 200 nm (see Tables E490) at 1 AU (astronomical unit) ($1.495\,988\,2 \times 10^{11}$ m) (**1**).[3]

3.1.7 *in situ*—within the vacuum environment. It may be used to describe measurements performed during irradiation as well as those performed before and after irradiation.

3.1.8 *integral flux*—the total number of particles impinged on a unit area surface for the duration of a test, determined by integrating the incident particle's flux over time. Also referred to as *fluence*.

3.1.9 *irradiance at a point on a surface*—the quotient of the radiant flux incident on an element of the surface containing the point, by the area of that element. Symbol: E_e, E; $E_e^1 = d\varphi_e/dA$; Unit: watt per square metre, W/m^2. (See Terminology E349.)

3.1.10 *near ultraviolet*—the wavelength range from 200 to 400 nm.

3.1.11 *near ultraviolet sun*—for test purposes only, the solar irradiance, at normal incidence, on a surface in free space at a distance of 1 AU from the sun in the wavelength band from 200 to 400 nm. Using the standard solar-spectral irradiance, the value is 8.73 % of the solar constant or 118 W/m^2 (see Terminology E349). This definition does not imply that any spectral distribution of energy in this wavelength band is satisfactory for testing materials.

3.1.12 *particle flux density*—the number of charged particles incident on a surface per unit area per unit time.

3.1.13 *reciprocity*—a term implying that effect of radiation is only a function of absorbed dose and is independent of dose rate.

3.1.14 *solar absorptance (α_s)*—the fraction of total solar irradiation that is absorbed by a surface. Use the recommended spectral-solar irradiance data contained in Tables E490.

3.1.15 *solar constant*—the solar irradiance, at normal incidence, on a surface in free space at the earth's mean distance from the sum of 1 AU. The value is 1353 ± 21 W/m^2 (see Tables E490).

3.1.16 *synergistic*—relating to the cooperative action of two or more independent causal agents such that their combined effect is different than the sum of the effect caused by the individual agents.

3.1.17 *thermal emittance (ε)*—the ratio of the thermal-radiant exitance (flux per unit area) of the radiator (specimen) to that of a full radiator (blackbody) at the same temperature.

4. Summary of Practice

4.1 The most typical approach in performing this test is to measure the radiative properties of the specimen under consideration, then to place the specimen in a vacuum chamber and expose it to the desirable simulated space environments. The specimen temperature is controlled during the period of exposure. The radiative property measurements are performed in situ without exposing the specimen to atmospheric pressure, after exposure and before measurement. Unless it has been established that the material under investigation is not affected by postexposure measurements, the in situ approach is the preferred method. Usually only the radiative property of solar absorptance, α_s, is of interest, and the net result of the test is a measurement of change in solar absorptance, $\Delta\alpha_s$. For detailed discussions of methods of determining radiative properties, see Test Method E903 and Refs. (**2**), (**3**), and (**4**).

4.2 The most effective method is to combine the radiation components of the space environments and investigate the synergistic effects on radiative properties of the thermal control materials.

5. Specimen Analysis

5.1 A method characterizing the behavior of thermal control materials during space environment exposure is through spectral reflectance measurements. The two parameters of engineering importance are total solar absorptance (α_s) and total hemispherical emittance (ε_h). Solar absorptance is generally determined from spectral reflectance measured under conditions of near normal irradiation and hemispherical viewing over the wavelength range from 0.25 to 2.5 µm. For these measurements, an integrating sphere with associated spectrophotometer is commonly used. For reflectance measurements beyond 2.5 µm, a blackbody cavity or parabolic reflectometer is frequently used.

5.2 *Postexposure Measurements:*

[3] The boldface numbers in parentheses refer to the list of references at the end of this practice.

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5.2.1 Although in situ measurements are necessary, many measurements must be performed after removal of the specimen from the test chamber. The accuracy of such measurements should be verified by in situ measurements because of possible bleaching.

5.2.2 Postexposure measurements of properties should be accomplished as soon as possible after the exposure. Where delays allow the possibility of bleaching, it is necessary to minimize atmospheric effects by maintaining the specimens in the dark and in vacuum until measured. In the event that evacuation is impractical, it is desirable that the specimens be maintained under a positive pressure of dry argon. Note that bleaching by diffusion of oxygen or nitrogen into the system has been observed to occur in the dark, although more slowly, than in the light.

5.3 *In Situ Analysis:*

5.3.1 Calorimetric measurements of thermal-radiative properties have received some attention in connection with in situ studies of thermal-radiative property changes. A calorimetric determination gives a direct measure of α_s/ε and therefore indicates the in situ changes in thermal-radiative properties. If *e*does not change, then the change in α_s/ε shows the change in α_s. If the electromagnetic radiation source provides a good match to the air-mass zero solar-spectral irradiance, then *a* will be equal to α_s. The limiting factors in calorimetric α_s/ε determinations are the deviation of the spectral irradiance produced by the simulated solar source from that of the solar irradiance and the accuracy of the irradiance measurement (see Test Method E434).

5.3.2 In situ measurements allow the determination of the reflectance or absorptance in a vacuum environment. The environment maintained for in situ measurements should have no effect on the property being measured. The annealing of the specimen after irradiation may occur sufficiently fast to make the posttest measurements misleading. In situ reflectance measurements allow the investigator to plot a curve of the change in thermal radiative properties as a function of the exposure or absorbed dose. Posttest measurements limit the data to one point at the total dose.

5.4 *Physical Property Analysis:*

5.4.1 The complete evaluation of thermal control coatings does not depend only on thermal-radiative property measurements; coatings must have the adhesion and stability required for retention on a specified substrate. One method used to evaluate the ability of the coating to remain firmly attached to the substrate in space is through thermal cycling of the specimens either during or after radiation exposure in a vacuum.

5.4.2 The loss of mass of thermal control coatings can be measured, to provide an indication of the amount of decomposition products leaving the coating during exposure. This may be important in the study of the curing, outgassing, and contamination potential of thermal control coatings.

5.4.3 Vacuum gas analysis (mass spectroscopy or residual gas analysis, RGA) can be used to assess the type and concentration of decomposition products.

5.5 *Surface Analysis of Specimens*—X-ray photoelectron specotroscopy (XPS), auger electron spectroscopy, and secondary ion mass spectrometry (SIMS) are some techniques that can be used to determine the composition of materials on the surface of the specimens. This information can then be used to identify any contamination that may be present on the specimens.

5.6 *Auxiliary Methods of Specimen Analysis*—Several other techniques for specimen characterization and analysis are available to the investigator. As a rule, these are usually used in studies of damage mechanisms rather than engineering tests. They are included in Table 1 to give a more complete account of methods for analysis of thermal control surfaces damaged by electromagnetic or particle irradiation, or both.

SIMULATION SYSTEM

6. Vacuum System

6.1 *General Description*—The vacuum system shall consist of the specimen test chamber, all other components of the simulation system that are joined to the chamber without vacuum isolation during specimen exposure, and the transition sections by which these components are joined to the chamber. The vacuum system must perform the following functions:

6.1.1 It must provide for a reduction of pressure of atmospheric gases in the test chamber to a level in which none of the constituents can react with the specimen material to affect the validity of the tests. This provision implies a pressure no greater than 1×10^{-6} torr (133 μPa) at the specimen position.

6.1.2 It must provide that the specimen area be maintained as free as possible from contaminant gases and vapors. These gases and vapors may originate anywhere in the system including from the test specimens themselves.

6.1.3 It must promptly trap or remove any volatiles outgassed from the test specimens.

6.1.4 It must provide for accurate pressure measurements in the chamber. (See Practice E296.)

6.2 *Test Chamber:*

6.2.1 *Construction*—The specimen test chamber should be constructed of materials suitable for use in ultra-high vacuum. Metals, glasses, and ceramics are used. Tables E490 contain information on materials for vacuum applications. Austenitic-stainless steels, such as Type 304, are frequently used for vacuum-chamber construction.

6.2.1.1 Welding and brazing should be performed in accordance with good high-vacuum practice and the temperature requirements of the chamber. Materials to be joined must be properly cleaned so that sound, leaktight, nonporous joints can be made. Inert gas arc welding (TIG), using helium or argon, and electron beam welding have been used. Brazing materials and cleaning techniques are discussed in Refs (**5**) and (**6**). Welds should be on the vacuum side to eliminate the possibility of trapping gas in cracks and crevices, thus creating a virtual leak. Parts must be absolutely clean before welding. An oil film can cause gas to evolve and result in a porous, leaky weld.

6.2.1.2 Dimensions of the test chamber should be sufficiently large in relation to those of the specimen holder, so that contaminants outgassed from any of the specimens cannot be reflected back from windows or walls to the surface of other specimens.

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TABLE 1 Potential Techniques Used for Specimen Analysis in Ground-Based Simulated-Solar Ultraviolet Studies on Thermal-Control Coatings

NOTE 1—Bidirectional reflectance is influenced by the changes in geometrical distribution of the reflected energy, as well as the change in spectral reflectance. Extreme care must be used in interpreting results for degradation evaluation.

Measurement Techniques	Laboratory Equipment	Properties Investigated[A]	Materials[B,C]
General sample analysis:			
Spectral reflectance measurements (pre- and post-test)	integrating sphere, Hohlraum, Coblentz hemisphere	α, ε	P, B, P/B
In-situ analysis:			
Calorimetric	vacuum, cryogenic apparatus	α, ε	P, B, P/B
Spectral	bidirectional reflectance, integrating sphere	...	P, B, P/B
Physical property analysis:			
Thermal cycling-mass loss (pre- and post-test and in situ)	thermal cycling, apparatus, radiation exposure apparatus	flexibility, adhesion (qualitative), weight	B, P/B
Auxiliary methods of analysis:	radiation exposure apparatus or simple thermal vacuum with or without radiation		P, B, P/B
X-ray diffraction		a, b, c	
X-ray powder diffraction		a, c, e	
X-ray fluorescence		a	
Electron microscopy		b, c, g, h	
Conventional microscopy		g, h	
Metallography		c, d, e, f, h	
Particle size analyzers		g	
Particle counter analyzers		g	
Gas absorption		f, g	
Porosimeter		f, g	
Spectrograph		a	
Extensometer		i	
Profilometers		d	
Density measurement		f	
Thermal conductance		p	
Resistance measurements		j	
Electron paramagnetic resonance		k, m	
Photoconductivity		d	
Seebeck and Hall coefficients		l	
Stoichiometry		n, o	
Oxidation-reduction capacity		n	
Optical absorption		n	
Low-energy electron diffraction		d	
X-ray photoelectron spectroscopy		q	
Auger electron spectroscopy		q	
Secondary ion mass spectroscopy		q	

[A] a= purity
b = crystal lattice
c = physical structure
d = surface structure
e = phases
f = void volume
g = particle size
h = particle shape
i = coefficient of expansion
j = electrical resistivity
k = free radicals
l = excess carriers
m = trapped unpaired electrons or holes
n = defect centers per unit volume
o = chemical structure
p = thermal conductivity
q = surface analysis

[B] P = pigment
B = binder
P/B = pigment/binder

[C] The laboratory equipment used and the types of materials investigated vary considerably and therefore will not be discussed in detail in this table.

6.2.1.3 The chambers should contain a cryogenic shroud, or be of an insulated double-wall (annular) construction, to provide for reducing wall temperature by the use of coolant fluids. The walls should preferably be cooled with liquid nitrogen during all tests. This feature is particularly essential if there are condensable contaminants in the test chamber arising from any part of the system or from the specimens. The temperature of the wall should always be lower than that of the test specimens to reduce the probability of contaminants preferentially condensing on the specimens. The use of a residual gas analyzer to measure the partial pressures of gases and vapors in the system may prove of use in interpreting the results of the tests.

6.2.1.4 The test chamber construction should also provide for bakeout to a temperature of at least 150 °C and preferably to 400 °C. Bakeout should be conducted before installing the test specimen for each test. Adequate bakeout can be accomplished in a shorter time at higher temperatures.

6.2.2 *Chamber Pumping System*—The pumping capacity of the test-chamber pumping system, including the cold wall, must be adequate not only for chamber evacuation, but to handle outgassing loads from specimen materials and gases or vapors entering the chamber from other system components.

6.2.2.1 The pumping system should be selected or designed to maintain the test-specimen contamination at levels below those which would affect the test results.

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