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Designation: E423 – 71(Reapproved 2008)

Standard Test Method for Normal Spectral Emittance at Elevated Temperatures of Nonconducting Specimens[1]

This standard is issued under the fixed designation E423; 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

The general physical properties of ceramic materials combine to make thermal gradients a serious problem in the evaluation and use of thermal emittance data for such materials. Ceramic materials in general tend to be somewhat translucent, and hence emit and absorb thermal radiant energy within a surface layer of appreciable thickness. Ceramic materials in general also tend to have low thermal conductivity and high total emittance as compared to metals. These properties combine to produce thermal gradients within a heated specimen unless careful precautions are taken to minimize such gradients by minimizing heat flow in the specimen. The gradients tend to be normal to a surface that is emitting or absorbing radiant energy. As a further complication, the gradients tend to be nonlinear near such a surface.

When a specimen is emitting from a surface layer of appreciable thickness with a thermal gradient normal to the surface, it has no unique temperature, and it is difficult to define an effective temperature for the emitting layer. Emittance is defined as the ratio of the flux emitted by a specimen to that emitted by a blackbody radiator at the same temperature and under the same conditions. It is thus necessary to define an effective temperature for the nonisothermal specimen before its emittance can be evaluated. If the effective temperature is defined as that of the surface, a specimen with a positive thermal gradient (surface cooler than interior) will emit at a greater rate than an isothermal specimen at the same temperature, and in some cases may have an emittance greater than 1.0. If the thermal gradient is negative (surface hotter than interior) it will emit at a lesser rate. If the "effective temperature" is defined as that of an isothermal specimen that emits at the same rate as the nonisothermal specimen, we find that the effective temperature is difficult to evaluate, even if the extinction coefficient and thermal gradient are accurately known, which is seldom the case. If spectral emittance is desired, the extinction coefficient, and hence the thickness of the emitting layer, changes with wavelength, and we have the awkward situation of a specimen whose effective temperature is a function of wavelength.

There is no completely satisfactory solution to the problem posed by thermal gradients in ceramic specimens. The most satisfactory solution is to measure the emittance of essentially isothermal specimens, and then consider the effect of thermal gradients on the emitted radiant flux when attempting to use such thermal emittance data in any real situation where thermal gradients normal to the emitting surface are present.

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1. Scope

1.1 This test method describes an accurate technique for measuring the normal spectral emittance of electrically nonconducting materials in the temperature range from 1000 to 1800 K, and at wavelengths from 1 to 35 μm. It is particularly suitable for measuring the normal spectral emittance of materials such as ceramic oxides, which have relatively low thermal conductivity and are translucent to appreciable depths (several millimetres) below the surface, but which become essentially opaque at thicknesses of 10 mm or less.

1.2 This test method requires expensive equipment and rather elaborate precautions, but produces data that are accurate to within a few percent. It is particularly suitable for research laboratories, where the highest precision and accuracy are desired, and is not recommended for routine production or acceptance testing. Because of its high accuracy, this test method may be used as a reference method to be applied to production and acceptance testing in case of dispute.

1.3 This test method requires the use of a specific specimen size and configuration, and a specific heating and viewing technique. The design details of the critical specimen furnace are presented in Ref (**1**),[2] and the use of a furnace of this design is necessary to comply with this test method. The transfer optics and spectrophotometer are discussed in general terms.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E349 Terminology Relating to Space Simulation

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *spectral normal emittance*—The term spectral normal emittance (Note 4) as used in this specification follows that advocated by Jones (**2**), Worthing (**3**), and others, in that the word emittance is a property of a specimen; it is the ratio of radiant flux emitted by a specimen per unit area (thermal-radiant exitance) to that emitted by a blackbody radiator at the same temperature and under the same conditions. Emittance must be further qualified in order to convey a more precise meaning. Thermal-radiant exitance that occurs in all possible directions is referred to as hemispherical thermal-radiant exitance. When limited directions of propagation or observation are involved, the term directional thermal-radiant exitance is used. Thus, normal thermal-radiant exitance is a special case of directional thermal-radiant exitance, and means in a direction perpendicular (normal) to the surface. Therefore, spectral normal emittance refers to the radiant flux emitted by a specimen within a narrow wavelength band and emitted into a small solid angle about a direction normal to the plane of an incremental area of a specimen's surface. These restrictions in angle occur usually by the method of measurement rather than by radiant flux emission properties.

NOTE 1—All the terminology used in this test method has not been standardized. Terminology E349 contain some approved terms. When agreement on other standard terms is reached, the definitions used herein will be revised as required.

4. Summary of Test Method

4.1 The principle of the test method is direct comparison of the radiance of an isothermal specimen at a given temperature to that of a blackbody radiator at the same temperature. The details of the method are given by Clark and Moore (**1**,**4**).

NOTE 2—With careful attention to detail, overall accuracy of ±2 % can be attained.

4.2 The essential features of the test method are (*1*) the use of a cylindrical sample that rotates in an electrically heated furnace and attains essentially isothermal conditions, and (*2*) the use of electronic controls to maintain the host specimen and blackbody reference at the same temperature.

4.3 A theoretical analysis (**5**) was made of thermal gradients in the rotating cylinder, supplemented by measurements of the temperature and temperature changes indicated by a small thermocouple imbedded 0.025 mm below the surface of a specimen of alumina, as the specimen rotated in front of a water-cooled viewing port. In brief, it was found that (*1*) the temperature fluctuations at the surface of the specimen were inversely related to the speed of rotation, and because negligibly small (2 K or less) at speeds of rotation greater than 50 r/min, and (*2*) the temperature indicated by a radiation shielded thermocouple suspended in the center of the rotating specimen was the same within 1 K as the average temperature indicated by the embedded thermocouple at speeds of rotation greater than 10 r/min.

NOTE 3—An electronic-null, ratio-recording spectrophotometer[4] is preferred to an optical-null instrument for this use. Special precautions may be necessary to obtain and maintain linearity of response of an optical-null instrument if the optical paths are not identical to those of the instrument as manufactured. Clark and Moore (**1**) describe linearity calibration of an optical-null instrument.

5. Significance and Use

5.1 The significant features are typified by a discussion of the limitations of the technique. With the description and

[1] This test method 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.

Current edition approved May 1, 2008. Published July 2008. Originally approved in 1971. Last previous edition approved in 2002 as E423-71(2002) DOI: 10.1520/E0423-71R08.

[2] The boldface numbers in parentheses refer to the references listed at the end of this test method.

[3] 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.

[4] The Perkin-Elmer Model 13-U prism spectrophotometer is one of several instruments found suitable for this test method.

arrangement given in the following portions of this test method, the instrument will record directly the normal spectral emittance of a specimen. However, the following conditions must be met within acceptable tolerance, or corrections must be made for the specified conditions.

5.1.1 The effective temperatures of the specimen and blackbody must be within 1 K of each other. Practical limitations arise, however, because the temperature uniformities are often not better than a few kelvins.

5.1.2 The optical path length in the two beams must be equal, or, preferably, the instrument should operate in a nonabsorbing atmosphere, in order to eliminate the effects of differential atmospheric absorption in the two beams. Measurements in air are in many cases important, and will not necessarily give the same results as in a vacuum, thus the equality of the optical paths for dual-beam instruments becomes very critical.

NOTE 4—Very careful optical alignment of the spectrophotometer is required to minimize differences in absorptance along the two paths of the instrument, and careful adjustment of the chopper timing to reduce "cross-talk" (the overlap of the reference and sample signals) as well as precautions to reduce stray radiation in the spectrophotometer are required to keep the zero line flat. With the best adjustment, the "100 % line" will be flat to within 3 %.

5.1.3 Front-surface mirror optics must be used throughout, except for the prism in prism monochromators, and it should be emphasized that equivalent optical elements must be used in the two beams in order to reduce and balance attenuation of the beams by absorption in the optical elements. It is recommended that optical surfaces be free of SiO_2 and SiO coatings: MgF_2 may be used to stabilize mirror surfaces for extended periods of time. The optical characteristics of these coatings are critical, but can be relaxed if all optical paths are fixed during measurements or the incident angles are not changed between modes of operation (during 0 % line, 100 % line, and sample measurements). It is recommended that all optical elements be adequately filled with energy.

5.1.4 The source and field apertures of the two beams must be equal in order to ensure that radiant flux in the two beams compared by the apparatus will pertain to equal areas of the sources and equal solid angles of emission. In some cases it may be desirable to define the solid angle of the source and sample when comparing alternative measurement techniques.

5.1.5 The response of the detector-amplifier system must vary linearly with the incident radiant flux, or must be calibrated for linearity, and corrections made for observed deviations from linearity.

6. Apparatus

6.1 *Spectrophotometer*—The spectrophotometer used for the measurement of spectral normal emittance is equipped with a wavelength drive that provides automatic scanning of the spectrum of radiant flux and a slit servomechanism that automatically opens and closes the slits to minimize the variations of radiant flux in the comparison beam. For most materials the wavelength band-pass of the instrument is generally smaller than the width of any absorption or emission band in the spectrum to be measured. Operation of the spectrophotometer at a higher sensitivity level or in a single-beam mode can be used to evaluate band-pass effects. In a prism instrument, several prism compositions can be used to cover the complete wavelength range; however, a sodium chloride prism is typically used to cover the spectral range from 1.0 to 15 μm, and a cesium bromide prism to cover the spectral range from 15 to 35 μm. As a detector, a vacuum thermocouple with a sodium chloride window is used in the spectral range from 1 to 15 μm, and a vacuum thermocouple with a cesium bromide window in the spectral range from 1 to 35 μm. A black polyethylene filter is used to limit stray radiation in the 15 to 35-μm range.

6.1.1 In order to reduce the effects of atmospheric absorption by water vapor and carbon dioxide, especially in the 15 to 35-μm range, the entire length of both the specimen and reference optical paths in the instrument must be enclosed in dry nonabsorbing gas[5] (dew point of less than 223 K) by a nearly gastight enclosure maintained at a slightly positive pressure relative to the surrounding atmosphere.

6.2 *Specimen Furnace*—Fig. 1 is a schematic drawing of the specimen furnace used at the National Institute of Standards and Technology. The high-temperature alumina core surrounding the specimen is wound with 0.8-mm diameter platinum-40 % rhodium wire. The winding is continuous to the edges of the rectangular opening that is cut into the core to permit entrance of the viewing port. A booster winding of the same wire positioned on the outer alumina core, as indicated in Fig. 1, is used to compensate for the large heat losses at the center.

6.2.1 The water-cooled viewing port is machined from copper, and its inner surface is curved to the same radius as the specimen. A shield of platinum foil, 0.05 mm thick, surrounds the outer surfaces of the port, including the edges that face the specimen. This helps to isolate the viewing port thermally from the furnace interior. The inner surfaces of the viewing port and the portion of the platinum shield nearest the specimen are blackened to minimize the possibility of errors from reflected radiation. The opening at the inner end of the port is 3 mm wide by 12.7 mm high.

6.2.2 The alumina support tube (Fig. 1) is surface ground to the same tolerance as given in 7.1 for the test specimen. The spindle is driven by a ⅛-hp motor that is coupled to a gear reducer. With the arrangement used, the rotation of the specimen can be adjusted to any speed in the range from 1 to 300 r/min.

6.2.3 The design of the furnace shell is such that the furnace can be operated in an inert atmosphere, as well as in air. Glass-metal seals are used for power leads and rubber O-ring seals are used for the shell ends, as well as for a sodium chloride viewing window. The thrust bearings are designed to provide a reasonably gastight seal at the point where the spindle shaft enters the shell.

6.2.4 Axial temperature gradients in the specimens are reduced to low values by adjusting the power to the booster coil. The gradients are measured by sighting a micro-optical pyrometer on a rotating specimen through the viewing port. No temperature differences from top to bottom should be observed for any of the specimens at 1200 K. At 1400 K, the top may be

[5] CO_2-free air at a dew point of less than 223 K has been found satisfactory.

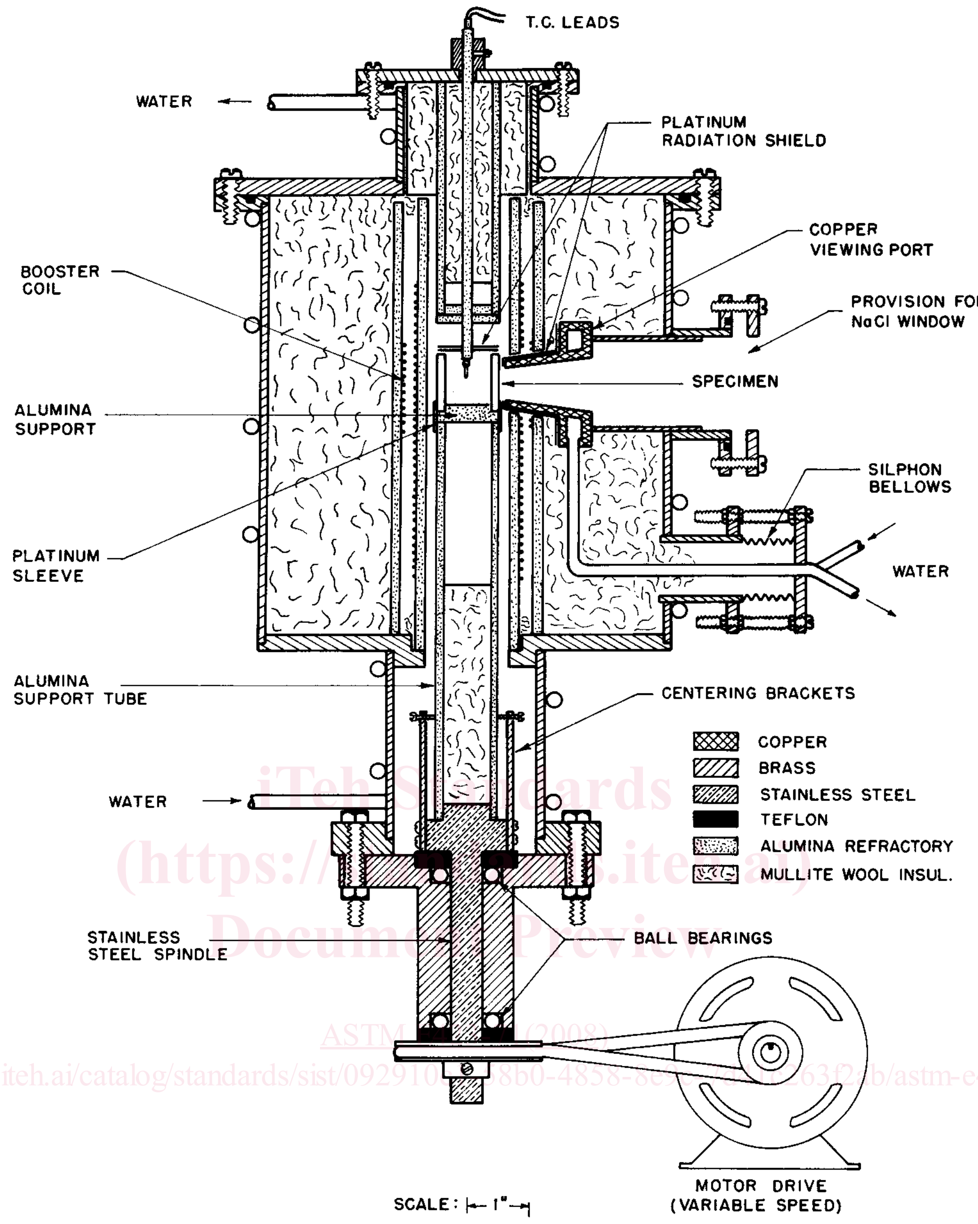

FIG. 1 Specimen Furnace

iTeh Standards (https://standards.iteh.ai) Document Preview ASTM E423-71(2008) https://standards.iteh.ai/catalog/standards/sist/09291088-8b0-4858-8e...263f2fb/astm-e423-712008

as much as 2 K higher than the bottom, while at 1600 K, the top may be as much as 6 K higher. The precision to which the pyrometer can be read is about 2 K. The specimen thermocouple is located in the center of the specimen cavity and is shielded from furnace radiation as shown in Fig. 1.

6.3 *Blackbody Furnaces*—Fig. 2 is a schematic of the blackbody furnace used at the National Institute of Standards and Technology. Two of these are required in the measurement system and are made to be as nearly identical as possible. The inner cavity of the furnaces is formed of fused alumina bonded with 20 weight % of a calcium aluminate cement. This mixture, which sets hydraulically, is mixed with 17.5 weight % of water and vibrated into a greased plaster mold. The mold is made by using a grooved alumina tube as a pattern; a brass mandrel is positioned at the center of the mold to form the outline of the cavity. After allowing 20 h for curing, the core is removed from the mold, dried in an oven for 24 h, and then heated to 1925 K for 1 h. It is later wound with 0.8-mm diameter platinum-40 % rhodium resistance wire.

6.3.1 An alumina disk with a 12.7 by 3-mm center slit is placed over the front end of the cavity to reduce heat losses by radiation. The viewing port is formed with mullite wool, over which is placed a thin layer of alumina cement. The shaping of the port is facilitated by use of a tapered aluminum mandrel machined especially for the purpose.

6.3.2 All furnace thermocouples (including the one for the specimen furnace) are made of 0.5-mm diameter platinum—platinum-10 % rhodium wire that has been calibrated. Two-hole alumina tubing is used to insulate the leads. This tubing is inserted into an alumina sleeve that is cemented into the core