~~2022-12-08~~

ISO/FDIS 24687:~~2022~~2023(E)

DATE: 2023-xx

ISO TC 206~~/WG 11~~

Secretariat: JISC

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement of Seebeck coefficient and electrical conductivity of bulk-type thermoelectric materials at room and high temperatures

Céramiques techniques — Mesurage du coefficient de Seebeck et de la conductivité électrique de matériaux thermoélectriques de base à températures ambiante et élevée

Style Definition: Heading 1: Indent: Left: 0 pt, First line: 0 pt, Tab stops: Not at 21.6 pt

Style Definition: Heading 2: Font: Bold, Tab stops: Not at 18 pt

Style Definition: Heading 3: Font: Bold

Style Definition: Heading 4: Font: Bold

Style Definition: Heading 5: Font: Bold

Style Definition: Heading 6: Font: Bold

Style Definition: ANNEX

Style Definition: AMEND Terms Heading: Font: Bold

Style Definition: AMEND Heading 1 Unnumbered: Font: Bold

Formatted: Justified

ISO copyright office

Ch. de Blandonnet 8 • CP 401

CH-1214 Vernier, Geneva, Switzerland

Tel. + 41 22 749 01 11

Fax + 41 22 749 09 47

copyright@iso.org

www.iso.org

www.iso.org

Commented [A1]: The reference is to a withdrawn standard which has been replaced

ISO/IEC 2022, Information technology — Character code structure and extension techniques

Formatted: Pattern: Clear

Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see ~~www.iso.org/directives~~www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see ~~www.iso.org/patents~~www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: ~~www.iso.org/iso/foreword.html~~www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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~~www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement of Seebeck coefficient and electrical conductivity of bulk-type thermoelectric materials at room and high temperatures

1 Scope

This document specifies the measurement methods for the electronic transport properties of bulk-type thermoelectric materials at room and elevated temperatures. The measurement methods cover the simultaneous determination of Seebeck coefficient and electrical conductivity of bulk-type thermoelectric materials in a temperature range from 300 K to 1 200 K. The measurement methods are applicable to bulk-type thermoelectric materials used for power generation, energy harvesting, cooling and heating, among other things.

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.

<std>ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 23331, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for total electrical conductivity of conductive fine ceramics*</std>

Formatted: Pattern: Clear

Formatted: Pattern: Clear

Formatted: Pattern: Clear

3 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/obphttps://www.iso.org/obp

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

3.1
thermoelectric figure of merit
zT
dimensionless factor representing the thermoelectric conversion efficiency of a given material

3.2
thermoelectric power factor
$S^2\sigma$
characteristic value of a thermoelectric material given by the product of the square of Seebeck coefficient (S) and electrical conductivity (σ)

Note 1 to entry: The units of the thermoelectric power factor are watts per metre per square kelvin (W/mK^2).

3.3

Seebeck coefficient
S
intrinsic property which describes the induced voltage (thermal electromotive force, *E*) from a given temperature difference (ΔT) in a material

Note 1 to entry: The units of the Seebeck coefficient are microvolts per kelvin (μV/K).

3.4
electrical conductivity
σ
ability of a material to allow the transport of electric charges

Note 1 to entry: The units of electrical conductivity are Siemens per centimetre (S/cm).

4 Principle

This document is for simultaneously measuring the Seebeck coefficient and the electrical conductivity of bulk-type thermoelectric materials using one measurement system. The off-axis four-terminal method can be used to simultaneously measure the Seebeck coefficient and the electrical conductivity of bulk-type thermoelectric material using one measurement system. As shown in Figure 1, the specimen is set between two metal blocks in the heating zone and two thermocouple probes separately contact the surface of the specimen. The measurement of the Seebeck coefficient of a bulk-type thermoelectric material is necessary to measure the temperature difference between two positions (point H and point C) on a specimen and the voltage across the two same positions (Figure 1). Seebeck coefficient can be calculated by following Formula (1~~).~~):

~~$S = E/\Delta T$~~
$$S = E / \Delta T \tag{1}$$

where

E is the induced thermoelectric voltage (thermal electromotive force) between the point H and point C of the specimen;

ΔT is the temperature difference between the point H and point C (= T_H - T_C).

For Seebeck coefficient measurement, measured temperature is the average temperature of the hot- and cold-side thermocouple probes.

By using the measuring system illustrated in Figure 2, electrical conductivity is also measured based on the four-terminal method. This method is conducted by placing four probes. Constant current is applied through the two outmost probes, causing a measurable voltage drop~~(, V)~~, between the two inner probes. The electrical resistance, *R*, is calculated using Ohm's law ~~(~~following Formula (2~~)~~):

~~$R = V/I$~~
$$R = V / I \tag{2}$$

where

V is the voltage;

I is the current.

The resistivity, *ρ*, is be calculated ~~by~~ following Formula (3~~).~~):

~~$\rho = RA/l$~~
$$\rho = RA / l \tag{3}$$

where

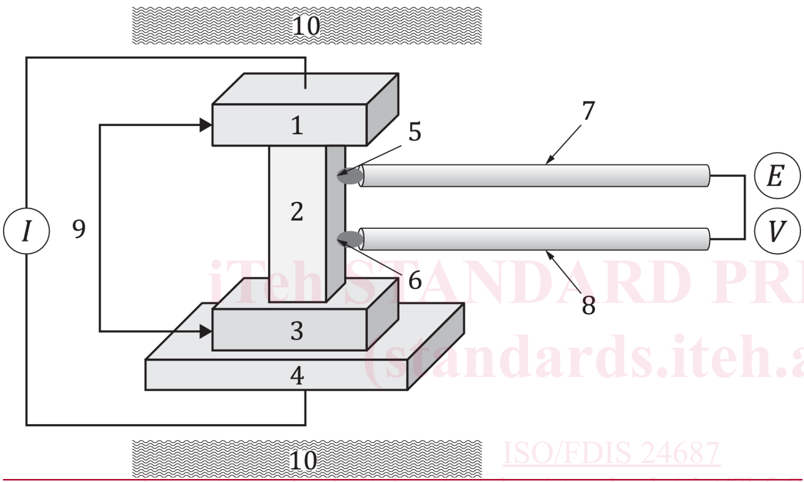
Formatted: Pattern: Clear
Formatted: Pattern: Clear
Formatted: Pattern: Clear
Formatted: Adjust space between Latin and Asian text, Adjust space between Asian text and numbers, Tab stops: Not at 19.85 pt + 39.7 pt + 59.55 pt + 79.4 pt + 99.25 pt + 119.05 pt + 138.9 pt + 158.75 pt + 178.6 pt + 198.45 pt
Field Code Changed
Formatted: Font: Not Italic
Formatted: Font: Not Italic
Formatted: Font: Not Italic
Formatted: Pattern: Clear
Formatted: Font: Italic
Formatted: Pattern: Clear
Formatted: Adjust space between Latin and Asian text, Adjust space between Asian text and numbers, Tab stops: Not at 19.85 pt + 39.7 pt + 59.55 pt + 79.4 pt + 99.25 pt + 119.05 pt + 138.9 pt + 158.75 pt + 178.6 pt + 198.45 pt
Field Code Changed
Formatted: Pattern: Clear
Formatted: Adjust space between Latin and Asian text, Adjust space between Asian text and numbers, Tab stops: Not at 19.85 pt + 39.7 pt + 59.55 pt + 79.4 pt + 99.25 pt + 119.05 pt + 138.9 pt + 158.75 pt + 178.6 pt + 198.45 pt
Field Code Changed

A is the cross-sectional area of the specimen;

l is the separation between the two inner probes.

: $R = V/I$, where V is voltage and I is current as shown in Figure 2. The resistivity can be calculated as follows: $\rho = RA/l$, where A is the cross-sectional area of the specimen, and l is the separation between the two inner probes. The electrical conductivity is the reciprocal of the resistivity. For electrical conductivity measurement, measured temperature is the actual temperature of the specimen, which generally can be measured by furnace temperature.

24687_ed1fig1.EPS

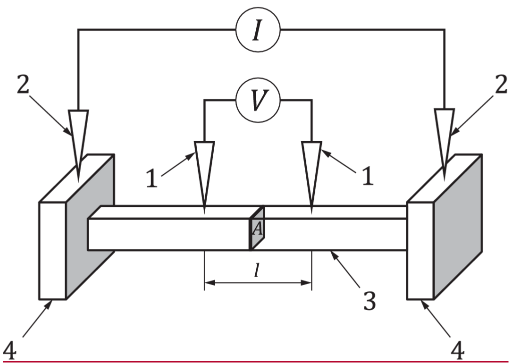


Key

1 upper metal block
2 specimen
3 lower metal block
4 heater
5 point C
6 point H
7 upper thermocouple probe (cold side)
8 lower thermocouple probe (hot side)
9 current electrode
10 heating furnace

Figure 1 — Schematic diagram of off-axis four-terminal method for simultaneous measurement of Seebeck coefficient and electrical conductivity

24687_ed1fig2.EPS



Key

1	inner probes	2	outer probes
3	specimen	4	electrodes

Figure 2 — Schematic diagram of four-terminal method to measure the electrical resistivity

The results of an interlaboratory test are given in Annex A.

Formatted: Pattern: Clear

5 Significance and use

This document gives guidance for simultaneously measuring the high-accuracy and low-error Seebeck coefficient and electrical conductivity of thermoelectric materials. Therefore, this standard is intended to be used for the development, characterization and quality control of thermoelectric materials, data acquisition for high-efficiency thermoelectric system design, etc.

Thermoelectric materials show Seebeck effect, Peltier effect and Thomson effect. The Seebeck effect is the direct conversion of heat into electricity. The conversion efficiency of a thermoelectric material is determined by the dimensionless thermoelectric figure of merit, zT, calculated following Formula (4):

$$zT = S^2 \sigma T / \kappa \qquad (4)$$

Field Code Changed

where ~~($zT = S^2\sigma T/\kappa$, where S is the Seebeck coefficient, σ is the electrical conductivity, κ is the thermal conductivity and T is the absolute temperature).~~

- S is the Seebeck coefficient;
- σ is the electrical conductivity;
- κ is the thermal conductivity;
- T is the absolute temperature.

Thermoelectric materials show a trade-off relation between Seebeck coefficient and electrical conductivity according to carrier concentration. Therefore, the accuracy of the power factor, $S^2\sigma$, where S is the Seebeck coefficient and σ is the electrical conductivity, can be improved through simultaneous measurement of Seebeck coefficient and electrical conductivity in one run.

Formatted: Font: Not Italic

6 Apparatus

~~6.1~~ 6.1 Current source

~~The current source should be~~, accurate to ±0,5 % on ranges of –1 A to +1 A used in the measurement.

~~6.2~~ 6.2 Electronic voltmeter

~~The electronic voltmeter should be~~, at least capable of measuring potential differences from 10^{-7} V to 0,05 V with a resolution below 10^{-7} V.

~~6.3~~ 6.3 Conducting metal blocks.

The contact surface of conducting metal blocks shall be sufficiently large compared to a measurement specimen. A specimen shall be placed between two conducting metal blocks, such as platinum or tungsten. One end of the specimen is heated while the other acts as a heat sink, dispersing heat, thus cooling that side. In addition, the conducting metal blocks play a role as the electrodes for applying the current when measuring the electrical conductivity.

NOTE Pt or Pt – Pd alloy is the best electrode material due to high measuring temperatures.

~~6.4~~ 6.4 Thermocouple probes.

The diameter of thermocouple probes shall be 0,5 mm or less to obtain reproducible Seebeck coefficient value. Thermocouples should have a resolution of at least 0,01 K or better. Thermocouple probes integrating electrical probes for measuring the voltage and thermal probes for measuring the temperature should be designed for working from 300 K to 1 200 K. Thermocouple probes should be checked periodically as their output may drift with usage or contamination.

NOTE In some equipment, the voltage can be measured only with thermocouple wires without additional electrical probes.

~~6.5~~ 6.5 Test chamber.

The test chamber shall be capable of heating both the specimen and the conducting metal blocks up to at least 1 200 K as well as maintaining the test temperature within ±1 K during the test, by which vacuum environment shall be available for test requirement. The test chamber should be evacuated below 3 Pa and can be backfilled with a variety of gases such as helium, argon, nitrogen and oxygen or a mixture of these. Low-pressure helium can be used to improve the thermal contact between the probe and the sample. However, low pressure may affect the measured Seebeck coefficient. Determination of optimum pressure of backfilled gas is required by Seebeck coefficient measurement according to gas pressure for the same sample to be measured. For the measurement of oxides, oxygen partial pressure should be controlled and monitored to avoid the reduction or oxidation of the samples.

~~6.6~~ 6.6 Dimension-measuring device

, such as a Vernier-calliper or other devices used for measuring the dimensions of the specimen ~~should be~~, accurate to at least 0,01 mm in accordance with ISO 3611.

~~6.7 Periodic check of apparatus~~ 6.7 Apparatus and equipment

~~The apparatus (or equipment) should be~~, checked periodically through measuring a certified reference material or a reference material to ensure if they are working properly (~~Refer to~~see Annex B).

Formatted: Font: Bold

Formatted: Font: Bold

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers, Tab stops: Not at 20 pt

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers, Tab stops: Not at 20 pt

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers, Tab stops: Not at 20 pt

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers

Formatted: Font: Bold

Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers