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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

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

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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 <u>www.iso.org/directiveswww.iso.org/directives</u>).

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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 <u>www.iso.org/members.html</u>.

# 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>

#### 3 Terms and definitions

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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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>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 ( $\sigma$ )

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

3.3

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#### Seebeck coefficient

S

intrinsic property which describes the induced voltage (thermal electromotive force, *E*) from a given temperature difference ( $\Delta T$ ) in a material

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

## 3.4 electrical conductivity

 $\sigma$ 

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 (11-):

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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):		Formatted: Pattern: Clear
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<i>E</i> is the induced thermoelectric voltage (thermal electromotive force) between the point H and	44	79.4 pt + 99.25 pt + 119.05 pt + 138.9 pt + 158.75 pt + 178.6 pt + 198.45 pt
point C of the specimen; b8688a403e21/iso-fdis-24687	Y	Field Code Changed
$\Delta T$ is the temperature difference between the point H and point C (= $T_{\rm H} - T_{\rm c}$ ).		Formatted: Font: Not Italic
For Seebeck coefficient measurement, measured temperature is the average temperature of the hot- and	$\square$	Formatted: Font: Not Italic
cold-side thermocouple probes.	Υ	Formatted: Font: Not Italic
By using the measuring system illustrated in Figure 2, electrical conductivity is also measured based on		Formatted: Pattern: Clear
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 4. V4, between the two inner probes.		Formatted: Font: Italic
The electrical resistance, $R_{a}$ is calculated using Ohm's law <u>(following</u> Formula (2 <del>))</del> :	4	Formatted: Pattern: Clear
$\frac{R = V/I}{L} R = V/I $ (2)		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 +
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where <i>V</i> is the voltage;		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
where <i>V</i> is the voltage; <i>I</i> is the current.		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 +
where V is the voltage; I is the current. The resistivity, $\rho_{\star}$ is be calculated by following Formula (3): $\rho = RA/l \rho = RA/l$ (3).		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 +

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2

is the cross-sectional area of the specimen; Α

1

1

3

5

7

9

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.





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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):

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<u>where</u>  $\{zT = S^2 \sigma T / \kappa$ , where *S* is the Seebeck coefficient,  $\sigma$  is the electrical conductivity,  $\kappa$  is the thermal conductivity and *T* is the absolute temperature).

<u>S</u> is the Seebeck coefficient;

 $zT = S^2 \sigma T / \kappa$ 

- $\underline{\sigma}$  is the electrical conductivity;
- <u>κ</u> is the thermal conductivity:
- <u>*T*</u> 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,  $S_{\alpha}^{c}\sigma$ , where *S* is the Seebeck coefficient and  $\sigma$  is the electrical conductivity, can be improved through simultaneous measurement of Seebeck coefficient and electrical conductivity in one run.

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<ul> <li>6 Apparatus</li> <li>6.4.5_1_Current source</li> <li>7.4.5_1_Current source</li> <li>7.4.5_1_Current source should be accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 10.5 % on ranges of _1-A to +1-A used in the measurement of Accurate to 2.4 duit space between Latin and Asim for Adjust space between Adain text and numbers.</li> <li>7.4.5.4.5.4.5.4.5.5.5.5.5.5.5.5.5.5.5.5.</li></ul>	ISO/FDIS 24687: <mark>2022<u>2023</u>(E)</mark>			
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64: 9_2, treetronic volunteer         The determine volunteer should be at least capable of measuring potential differences from 10° V to AGUS with a resolution below 10° V.         63: 6.3	The current source should be accurate to ±0,5 % on ranges of1-A to +1-A used in the measurement,	->		Formatted: Font: Bold
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6.3       6.3       Conducting metal blocks,         6.4       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 ungsten. 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 ar ole as the lectrodes for applying the urrent 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. Thermocouple probes should have a resolution of at least 0.01 K or better. Thermocouple probes should have a resolution of at least 0.01 K or better. 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 termocouple wires without additional better to a measurement of 200 K to 1 200 K. Thermocouple wires without additional better to a sould be explained for working from 300 K to 1 200 K. Thermocouple probes should be contacted 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 hall be calable for test requirement. The test chamber shall be calable for test requirement. The test chamber should be executed 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 pressive	The electronic voltmeter should be, at least capable of measuring potential differences from $10^{-7}$ V t	<b>0</b> •		text, Adjust space between Asian text and numbers
<ul> <li>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 tungsto. 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 trut, dyiust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt</li> <li>Formatted: p2, Adjust space between tain and Asian text, and numbers, Tab stops: Not at 20 pt<td></td><td>•</td><td></td><td></td></li></ul>		•		
NOTE       Ptor Pt - Pt 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 integrating electrical probes for measuring the voltage and thermal probes for measuring the test, Adjust space between Asian text and numbers, Tab stops: Not at 20 pt         The diameter of thermocouple probes should have a resolution of at least 0,01 K or better. Thermocouple probes should be checked periodically as their output may drift with usage or contamination.         NDTE       In some equipment, the voltage can be measured only with thermocouple wires without additional electrical probes.         6.5       6.5         Test chamber shall be capable of heating both the specimen and the conducting metal blocks up to at heast 1200 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 the same sample to be measured. For the measurement of oxides, oxygen partial pressure should be caccurate to at least 0,01 mm in accordance with 150 3611.         6.7 Aberiodic check of apparatus 6,7       Apparatus and equipment	The contact surface of conducting metal blocks shall be sufficiently large compared to a measuremen specimen. A specimen shall be placed between two conducting metal blocks, such as platinum o tungsten. One end of the specimen is heated while the other acts as a heat sink, dispersing heat, thu cooling that side. In addition, the conducting metal blocks play a role as the electrodes for applying th	r s		text, Adjust space between Asian text and numbers,
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 text, Adjust space between Asian text and numbers, tab stops: Not at 20 pt the text and numbers. The text chamber shall be capable of heating both the specimen and the conducting metal blocks up to at least 0,01 K or not the text chamber shall be capable of heating both the specimen and the conducting metal blocks up to at least 0,01 K at a variety of gases such as helium, argon, nitrogen and oxygen or a mixture of backfilled with a variety of gases such as helium, argon, nitrogen and oxygen or a mixture of backfilled gas is required by Seebeck coefficient measurement of oxides, oxygen partial pressure should be oraided to avoid the reduction or oxidation of the samples. <b>6.6 G.Dimension-measuring device 1.5 choinersion-measuring device 1.5 choinersion-measuri</b>				
<ul> <li>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.</li> <li>NOTE In some equipment, the voltage can be measured only with thermocouple wires without additional electrical probes.</li> <li>6-5 6.5 Test chamber, BAG884403e211/so-fdis-24687</li> <li>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 evaluated 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.</li> <li>6-6-6.0 Dimension-measuring device</li> <li>such as a Vernier-calliper or other devices used for measuring the dimensions of the specimen shoul a cordance with ISO 3611</li> <li>6.7 Periodic check of apparatus6.7 Apparatus and equipment</li> </ul>	6.4 <u>6.4</u> Thermocouple probes.	<b>←</b>	R	text, Adjust space between Asian text and numbers,
electrical probes.       Istom 115:24687/         6.5       6.5       Test chamber,       b8688a403e21/iso-fdis-24687/         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 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.       Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers.         6.6-6.6_0 Dimension-measuring device       Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Latin and Asian text, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers.         6.7-Periodic check of apparatus 6.7       Apparatus and equipment	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			
<ul> <li>6.5</li></ul>	electrical probes.	al		
<ul> <li>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.</li> <li>6.6.6.6.0 Dimension-measuring device         <ul> <li>such as a Vernier-calliper or other devices used for measuring the dimensions of the specimen-should*</li> <li>Formatted: p2, Adjust space between Latin and Asian text, Adjust space between Asian text and numbers</li> <li>6.7 Periodic check of apparatus 6.7 Apparatus and equipment</li> </ul> </li> </ul>	6.5 <u>6.5</u> Test chamber <u>.</u> b8688a403e21/iso-fdis-24687	<u>c</u> 4	4-4	text, Adjust space between Asian text and numbers,
<ul> <li><u>such as a</u> Vernier-calliper or other devices used for measuring the dimensions of the specimen-should</li> <li><u>be</u> accurate to at least 0,01 mm in accordance with ISO 3611</li> <li><u>6.7 Periodic check of apparatus</u> 6.7 <u>Apparatus</u> and equipment</li> </ul>	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			
be, accurate to at least 0,01 mm in accordance with ISO 3611       text, Adjust space between Asian text and numbers         6.7 Periodic check of apparatus       Apparatus       and equipment	6.6-6.6 Dimension-measuring device			
6.7 Periodic check of apparatus 6.7 Apparatus and equipment		d•		
	6.7 Periodic check of apparatus 6.7 Apparatus and equipment			
The apparatus (or equipment) should be, checked periodically through measuring a certified reference formatted: p2, Adjust space between Latin and Asian material or a reference material to ensure if they are working properly (Refer tosee Annex B).		e◀		

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