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Measuring spectral emissivity up to 4000 K†

D. URBAN[1]*, K. ANHALT[1], M. ARDUINI[2], T. STARK[2], J. MANARA[2], P. PICHLER[3], A. EBER[3] AND G. POTTLACHER[3]

[1]*Physikalisch-Technische Bundesanstalt (PTB), Abbestraße 2–12, 10587 Berlin, Germany*
Working Group 7.35 High-Temperature Radiation Thermometry
[2]*Center for Applied Energy Research (CAE), Magdalene-Schoch-Straße 3, 97074 Würzburg, Germany*
[3]*Graz University of Technology (TUG), Rechbauerstraße 12, 8010 Graz, Austria*

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An inter-laboratory comparison involving three participants (PTB, CAE, TU Graz) was conducted on spectral emissivity measurements using experimental setups covering a temperature range from 1250 K to 4000 K and a spectral range between 0.6 µm and 20 µm. The main objective here was to carry out a European-level assessment of the coherency of spectral emissivity measurements at high temperatures. Three refractory materials (molybdenum, tungsten and isotropic graphite) were selected for this comparison. Samples were machined from the same batch of materials and then sandblasted and thermally annealed (PTB, CAE) to achieve uniform and stable surface properties and reduce potential scattering of results between participants. The spectral emissivity of the three materials was then measured by each of the three participants up to the maximum temperature attainable by the devices used. In the overlapping wavelength range from 600 nm to 1100 nm, all measurement methods were in good agreement with regards to their respective uncertainties.

Keywords: Emissivity, high-temperature, radiation thermometry, tungsten, graphite, molybdenum

1 INTRODUCTION

In high-temperature applications (e.g., space aeronautics or fuel-based energy production), material safety, costs and energy use during production, and chemical resistance are critically affected by the thermophysical properties

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*Corresponding author: david.urban@ptb.de

of the materials employed. In particular, optical properties such as spectral emissivity play a key role in radiative energy transfer and temperature measurement via radiation thermometry. Because surface conditions are easily affected by chemical reactions at elevated temperatures and the dominant mode of heat transfer is radiation, the emissivity itself is notoriously difficult to measure at elevated temperatures.

The Joint Research Project EMPIR Hi-TRACE ("Industrial Process Optimization Through Improved Metrology of Thermophysical Properties") [1] aims to establish a metrological infrastructure in order to enable reliable and SI-traceable measurement techniques for thermophysical properties in the high-temperature range up to 3000 K.

For the measurement of the emissivity of solid materials, the measurement setups at the Physikalisch-Technische Bundesanstalt (PTB), the Center for Applied Energy Research (CAE), and the Graz University of Technology (TUG), which are based on radiometric or calorimetric principles as well as reflection polarimetry and use pulse or induction heating, were advanced and validated with respect to the achievable measurement uncertainty and experimentally compared.

2 METHODS

For this inter-laboratory comparison, three unique measurement facilities were developed or improved to measure the spectral normal emissivity of solids (and liquids) up to very high temperatures. The measurement setups of PTB, CAE and TUG rely on different measurement approaches and use distinct sample geometries. This section gives a brief overview of the three fundamentally different measurement methods. Further information on the measurement setups and the uncertainties can be found in the referenced literature.

2.1 PTB – ADeM facility

For measuring spectral normal emissivity in a wavelength range from 500 nm to 1100 nm up to a temperature of around 2500 K, PTB uses a dynamic method known as ADeM that is based on a commercial laser-flash apparatus [2–4].

In this device, a sample is inductively heated inside a water-cooled sample chamber flushed with inert argon gas. Once the sample has reached a temperature equilibrium, a short, high-energy laser pulse (Nd:YAG laser with a pulse length of around 1 ms and energy of roughly 1 J at the sample position) hitting the front side of the sample is used to increase its temperature. The resulting temperature rise of around 1 K to 3 K is measured on the backside of the sample with a radiation thermometer. By using a well characterized optical beamsplitter, a defined reflected portion of each laser pulse is

measured in situ and the laser energy hitting the sample can be determined. The measurement principle itself is based on the definition of the heat capacity (see eq. (3)), with mass m, specific heat c_p, spectral normal emissivity ε_λ, laser energy at the sample position E_L, and temperature rise ΔT.

$$L_{\lambda,\mathrm{S}}(\lambda_0, T_{0,\mathrm{S}}) = \tau_{\mathrm{LP5,w}}\, \varepsilon(\lambda_0, T_0)\, L_{\lambda,\mathrm{S}}(\lambda_0, T_0) \tag{1}$$

$$L_{\lambda,\mathrm{S}}(\lambda_0, (T_0 + \Delta T)_\mathrm{S}) = \tau_{\mathrm{LP5,w}}\, \varepsilon(\lambda_0, T_0)\, L_{\lambda,\mathrm{S}}(\lambda_0, T_0 + \Delta T) \tag{2}$$

$$m\, c_p(T_0) = \varepsilon(\lambda_0, T_0) \frac{E_\mathrm{L}}{\Delta T} \tag{3}$$

While equation (3) describes the calorimetric part of the measurement, there are two additional equations to describe the radiometric part. Equations (1) and (2) describe the measured spectral radiances of the radiation thermometer using Planck's law and the spectral transmissivity of the output window $\tau_{\mathrm{LP5,w}}$ at λ_0. The temperatures $T_{0,\mathrm{S}}$ and $(T_0 + \Delta T)_\mathrm{S}$ represent the adiabatic temperature rise using an emissivity of 1. They are iteratively determined using a mathematical model based on the heat transfer equations to analyze the temperature rise at the back side of the sample measured with the radiation thermometer. The model accounts, among others, for radial heat losses and the temporal as well as spacial distribution of the laser pulse. Kirchhoff's law for opaque materials is applied, which states that the spectral emissivity is equal to the spectral absorption at the laser wavelength, which is the same as the central wavelength of the radiation thermometer ($\lambda_0 = 1064$ nm).

In this case, the only direct measurands are the laser energy hitting the sample and the temperature rise of the sample. The central wavelength, transmissivity of the output window, and mass of the sample are measured separately and are only parameters in the equations above. For determining the spectral normal emissivity, it is necessary to know the specific heat, either from external measurements or from literature data. For the measurement results presented in this work, the specific heat values used were likewise determined in the Hi-Trace project by LNE, VINCA and PTB [5–7]. The spectral normal emissivity $\varepsilon(\lambda_0, T_0)$, the true base temperature T_0 of the sample, and the temperature rise ΔT can be derived by numerically solving the system of equations (1–3).

The measurement range for the spectral normal emissivity is extended by means of a well characterized array spectrometer operating at wavelengths between 500 nm and 1100 nm. First the spectral normal emissivity is measured at λ_0 using the fast radiation thermometer and the temperature of the sample is determined. In the next step, the spectral radiance detected by the array spectrometer is compared to the calculated spectral radiance of a blackbody at the same temperature to determine the spectral normal emissivity of the sample in a broader spectral range.

2.2 CAE – EMMA facility

The Emissivity Measurement Apparatus (EMMA) was developed at CAE specifically for determining the emissivity of opaque samples at high temperatures [8] in the wavelength range of 1 μm to 20 μm. It relies on a comparison of the spectral radiance of a sample and of a reference blackbody at equal temperature to determine the spectral emissivity.

A graphite cylinder (diameter of 20 mm, length of 100 mm and emissivity > 0.99) serves as a blackbody reference and can be inductively heated inside a vacuum chamber. The spectral intensity is then measured by an FTIR spectrometer (Bruker Vertex 70v) and a mirror arm to allow angularly resolved measurements. In order to simplify the measurement procedure, the system was calibrated before the start of each measurement campaign by performing measurements at several temperatures of the blackbody radiator and deriving a calibration function as described in [8]. This allows the measurements to be carried out at different temperatures of the sample, which may be different from the temperatures of the black body radiator. Since the background radiation coming from space immediately surrounding the cylinder needs to be considered, the walls of the vacuum vessel are coated with a high-emissivity paint (Nextel Velvet Coating 811–21) and their temperature is controlled independently. For a second measurement, the graphite cylinder is closed off by the sample, which, depending on its material composition, can be heated either directly by induction or indirectly via the hot graphite cylinder.

In order to determine the surface temperature of the investigated samples, the temperature gradient inside the samples was derived by measuring the temperatures at two positions at different distances from the surface. For this purpose, two small holes were prepared in the samples and the temperatures were measured using radiation thermometers focused on the cavities as mentioned in [8].

The measured spectral directional radiative intensity $L_{\lambda,\mathrm{meas}}$ (see eq. (4)) is a combination of the intensity emitted by the sample itself $L_{\lambda,\mathrm{bb}}(T_{\mathrm{sp}})$ at the sample temperature T_{sp}, dependent on the spectral directional emissivity $\varepsilon_{\mathrm{d},\lambda}$, and the reflected intensity $L_{\lambda,\mathrm{bb}}(T_{\mathrm{amb}})$ of the hemispherical surrounding space at ambient temperature T_{amb} linked with the spectral hemispherical-directional reflectivity $\rho_{\mathrm{hd},\lambda}(\theta,T_{\mathrm{sp}})$ of the sample.

$$L_{\lambda,\mathrm{meas}}(\theta,T_{\mathrm{sp}},T_{\mathrm{amb}}) = \varepsilon_{\mathrm{d},\lambda}(\theta,T_{\mathrm{sp}})\cdot L_{\lambda,\mathrm{bb}}(T_{\mathrm{sp}}) + \rho_{\mathrm{hd},\lambda}(\theta,T_{\mathrm{sp}})\cdot L_{\lambda,\mathrm{bb}}(T_{\mathrm{amb}}) \quad (4)$$

Equation (4) can be solved for the spectral directional emissivity with respect to the law of reciprocity ($\rho_{\mathrm{dh}} = \rho_{\mathrm{hd}}$), conservation of energy for non-transparent samples ($\alpha_{\mathrm{d},\lambda} + \rho_{\mathrm{dh}} = 1$), and Kirchhoff's law, which describes the identity of spectral directional absorptivity and emissivity ($\alpha_{\mathrm{d},\lambda} = \varepsilon_{\mathrm{d},\lambda}$):

$$\varepsilon_{\mathrm{d},\lambda}(\theta,T_{\mathrm{sp}}) = \frac{L_{\lambda,\mathrm{meas}}(\theta,T_{\mathrm{sp}},T_{\mathrm{amb}}) - L_{\lambda,\mathrm{bb}}(T_{\mathrm{amb}})}{L_{\lambda,\mathrm{bb}}(T_{\mathrm{sp}}) - L_{\lambda,\mathrm{bb}}(T_{\mathrm{amb}})} \quad (5)$$

The spectral directional emissivity can therefore be determined by measuring the intensities of the reference blackbody and the sample while carefully controlling the ambient temperature of the surrounding space.

2.3 TUG – Pulse-heating facility

At TUG the spectral normal emissivity of solids and liquids at a wavelength of 684.5 nm [9] is measured by a combination of an ohmic pulse-heating apparatus [10] and a microsecond division of amplitude polarimeter [11].

In the ohmic pulse-heating apparatus, a large current pulse (> 1000 A) is distributed through a thin wire-shaped sample inside an argon-filled chamber. The sample heats up via joule heating, melts and, upon reaching the material's boiling point, explodes. The whole heating process, including the two phase transitions, only takes around 30 µs to 50 µs. This short experiment time prevents chemical reactions with any surroundings and further ensures that the liquid wire can be observed without collapsing due to gravitational forces once the sample has melted. Sample temperature measurement is performed with a pyrometer specially designed for very fast time responses and operating at a wavelength of 649.7 nm with 10 nm FWHM bandwidth. The radiance temperature can later be used to calculate the true temperature once the spectral normal emissivity of the sample is known.

For the measurement of spectral normal emissivity, a microsecond division of amplitude polarimeter (µs-DOAP) is used. Here, a linearly polarized laser beam with a wavelength of $\lambda_0 = 684.5$ nm is focused on the surface of the wire-shaped sample. The reflection of the laser beam changes its polarization state and can be analyzed by the µs-DOAP, which uses the Stokes formalism for polarized light. By splitting the detected signal into four intensities, the complex refractive index n and the extinction coefficient κ for the sample material are calculated. These quantities can then be used to determine the normal spectral reflectivity ρ at the laser wavelength λ_0

$$\rho(\lambda_0) = \frac{(n-n_0)^2 + \kappa}{(n+n_0)^2 + \kappa}\,, \tag{6}$$

where n_0 is the refractive index of the ambient medium. Applying Kirchoff's law, the spectral normal emissivity $\varepsilon(\lambda_0)$ can finally be determined for opaque materials:

$$\varepsilon(\lambda_0) = 1 - \rho(\lambda_0) \tag{7}$$

3 SAMPLE MATERIALS

Because of their high melting points and high purity, three solid, homogeneous materials were identified as possible reference materials for spectral normal emissivity at very high temperatures:

- Tungsten 99.95% from Goodfellow with a melting point of approx. 3683 K [12]
- Molybdenum 99.9% from Goodfellow with a melting point of approx. 2896 K [13]
- Graphite IG210 from Toyo Tanso with a melting point of approx. 4773 K

To reduce potential inhomogeneity effects, the samples of PTB and CAE were machined for the inter-laboratory comparison measurements from the same blocks of tungsten, molybdenum, and graphite by the Laboratoire National de Métrologie et d'Essais (LNE). The wire samples for TUG were received from Goodfellow. Before the experiment the TUG samples were treated with abrasive paper grade 1000 and cleaned afterwards with acetone. A detailed description and summary of the chemical composition can be found in [14]. The samples were sent to PTB for further distribution.

Both the PTB and CAE measurement setups use disc-shaped samples of different dimensions. The pulse-heating apparatus of TUG requires thin, wire-shaped samples, which are cut to size from a single 3 m long wire. The sample geometries are listed in table 1.

TABLE 1
Sample geometries

Institute	Sample shape	Dimensions
PTB	disc	Ø: 10 mm, thickness: (2–3) mm
CAE	disc	Ø: 20 mm, thickness: 4 mm
TUG	wire	Ø: 0.5 mm, length: 70 mm

Sample geometries for the measurement setups of PTB, CAE and TUG.

When the disc-shaped samples arrived at PTB, the metallic samples appeared to have non-homogeneous surfaces. This may have been due to surface oxidation during the actual machining; saw marks, for example, remained after the sample rods were cut to the desired thicknesses. Since spectral emissivity is dependent not only on the sample material, temperature, and wavelength, but also on surface conditions of the sample, it was decided to sandblast the disc-shaped tungsten and molybdenum samples at PTB with white fused alumina (EKW 360, average grain size between 21 µm and 25 µm) to ensure good homogeneity, reproducible surfaces, and better sample-to-sample variability.

An optical impression before and after the sandblasting can be seen in figure 1. The sandblasting process removed the yellow oxidation layer of the tungsten as well as the dark spots on the molybdenum sample and created a reproducible surface roughness of around 1 µm, which was estimated using microscopy. These improvements to the surface and roughness uniformity were also confirmed by optical microscopy at PTB.

The sandblasted surface structure, however, was not stable with temperature as can be seen in figure 2. A tungsten sample was analyzed using a

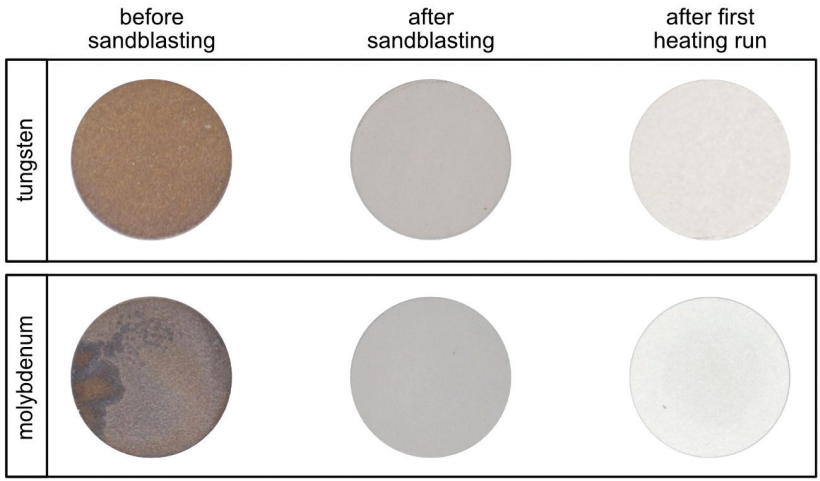

FIGURE 1
Images of the tungsten and molybdenum samples at each preparation step. After sandblasting, the surfaces appear optically homogeneous. After the first heating cycle, however, the surface changes again to a temperature stable structure.

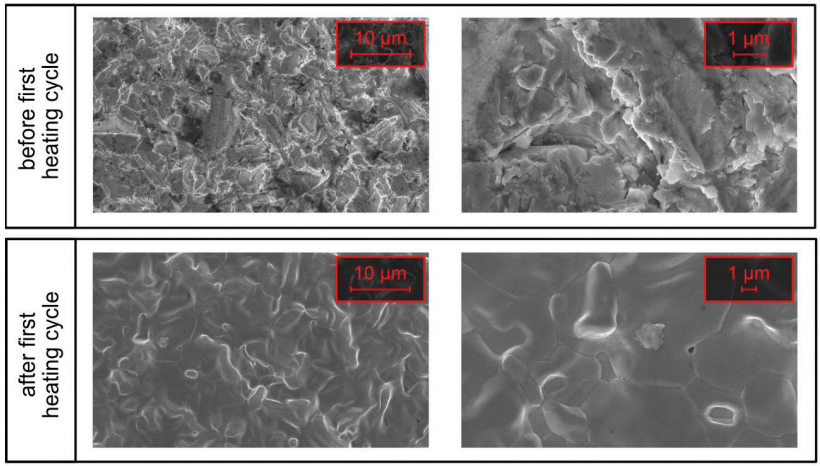

FIGURE 2
Scanning electron microscopy images of a tungsten sample before and after the first heating cycle. Sharp edges are rounded off and the surface structure is fused together after the tempering.

scanning electron microscope once before and once after the first heating cycle up to the maximum temperature of 2250 K. This showed that the microstructure of the surface had undergone a transformation, with all sharp edges rounded off and deeper crevices fused and blended together.

This was further supported by an emissivity measurement of a sandblasted tungsten sample by PTB during the first heating cycle. Figure 3 shows the spectral normal emissivity at 1064 nm as a function of temperature, with numbers indicating the order of the subsequent measurements. Starting at a temperature of 1450 K the emissivity decreases almost linearly from 0.65 to 0.43 with increasing measurement temperature up to the 2250 K maximum. In the cooling phase back down to 1550 K, the emissivity value remains constant with respect to the uncertainty. A similar behavior can be observed for the sandblasted molybdenum samples.

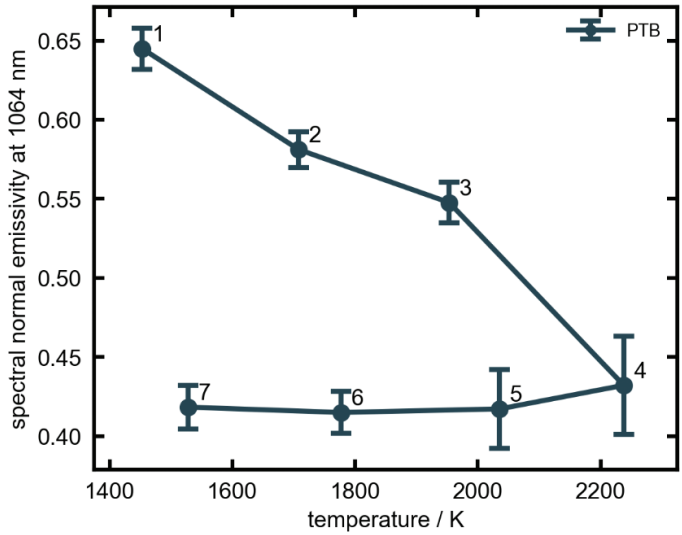

FIGURE 3
Spectral normal emissivity of a sandblasted tungsten sample measured by PTB during the first heating cycle. The numbering indicates the measurement sequence. After a decrease of the emissivity from 0.65 to 0.43 during the heating phase, it remains constant in the subsequent cooling phase.

To minimize the effect of structural changes due to the tempering of the samples, we decided to heat the sample to a temperature at least 50 K above the maximum measurement temperature prior to the actual emissivity measurements.

4 RESULTS

The following section presents the results for the spectral normal emissivity of tungsten, isotropic graphite, and molybdenum in order to compare and validate the different approaches followed by PTB, CAE and TUG. From the measurement data taken by each participant, certain wavelength and temperature intervals were selected to allow a direct comparison between the partners.

PTB measured the spectral normal emissivity in a wavelength range from 500 nm to 1100 nm, CAE provided measurement data over a broader measurement range, from 1 µm to 20 µm, and TUG investigated the spectral emissivity at 684.5 nm in the solid and liquid phases up to 4000 K. While PTB and CAE measured the emissivity at specific temperatures (the temperatures differ slightly because of the different techniques for adjusting the measurement temperatures), TUG offered a linear interpolation for the entire measurement temperature range. The temperature used by PTB was chosen as the reference temperature for comparing the different methods.

4.1 Isotropic graphite

An overview of the measurement results from PTB and CAE for the spectral normal emissivity of IG210 graphite in the temperature range between 1476 K and 2081 K as a function of wavelength is presented in figure 4 on the left. Three temperatures are presented in red, blue and yellow, with continuous lines showing the PTB results and dashed lines those of CAE.

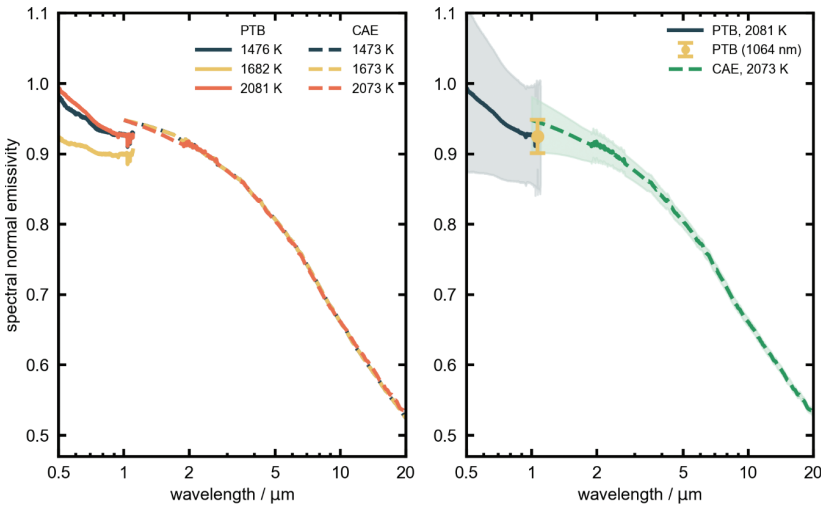

FIGURE 4
Spectral normal emissivity of IG210 graphite. Left: Overview of the spectral emissivity at different temperatures. Right: Visualization of the uncertainties ($k = 1$) of the PTB and CAE emissivity values at the highest measurement temperature of around 2075 K.

In the visible spectral range, the spectral normal emissivity ranges from near 1 to 0.9, with a slight decrease at higher wavelengths. This trend continues up to wavelengths of around 4 µm, after which the emissivity drops further to a minimum of < 0.55 at 20 µm. While the CAE emissivity values nearly fully coincide over the entire temperature range for a given wavelength in the IR range, PTB's measurement shows a slight disconnect in the VIS spectral range at a temperature of 1682 K.

The right side of figure 4 shows, as an example, the realized ($k = 1$) uncertainties of PTB and CAE at the highest measured temperature of around 2075 K. PTB values for the spectral emissivity are depicted as a blue continuous line (with array spectrometer) and in yellow (dynamically at 1064 nm), while CAE values are shown as a dashed green line. Overall, the relative uncertainties range from 2.6% to 11.8% for PTB and from 0.8% to 4.9% for CAE depending on the wavelength and the measurement temperature. A more detailed overview is given in table 2.

TABLE 2
Relative uncertainty for the spectral emissivity of IG210 graphite

	Rel. uncertainty ($k = 1$) for spectral emissivity of IG210 graphite / %		
T / K	**PTB 1064 nm**	**PTB (500 … 1000) nm**	**CAE (1 … 20) µm**
~ 1476	2.6	8.0 …6.3	4.9 … 0.9
~ 1682	2.7	8.3 …6.5	4.3 … 0.8
~ 2081	5.1	11.8 …7.6	3.5 … 0.8

Relative uncertainties of PTB and CAE measurements of the spectral emissivity of IG210 graphite. The relative uncertainties generally decrease with increasing wavelengths for the spectrally resolved measurements of PTB and CAE.

The results for the spectral normal emissivity of PTB and CAE were in good agreement across the entire temperature range as seen by the overlapping of the uncertainty intervals in the wavelength range from 1000 nm to 1100 nm. The full dataset is published as a data repository on Zenodo [15].

4.2 Tungsten

An overview of the measurement results of PTB, CAE and TUG for the spectral emissivity of sandblasted (PTB, CAE) and burnished (TUG) tungsten in the temperature range between 1373 K and 2378 K as a function of wavelength is presented in figure 5 on the left. Three temperatures are presented in red, blue and yellow, with continuous lines showing the PTB results, dashed lines representing those of CAE, and dots the results from TUG.

For all three temperatures, the spectral emissivity decreases with increasing wavelength from a starting value of around 0.55 at 500 nm to around 0.12 at 20 µm. Tungsten shows a typical infrared-optical behavior of metallic surfaces as the emissivity for different temperatures intersect at a so-called crossover point, in this case at a wavelength of $\lambda_X = 1.47$ µm. For wavelengths below λ_X, the spectral emissivity decreases with temperature, while the opposite is true for wavelengths above λ_X.

The right side of figure 5 shows, as an example, the realized ($k = 1$) uncertainties of PTB and CAE at the highest measured temperature of around 2375 K. PTB values for the spectral emissivity are depicted as a blue continuous line (with array spectrometer) and in yellow (dynamically at 1064 nm), CAE values are shown as a dashed green line, and TUG data as a red dot at 684.5 nm.

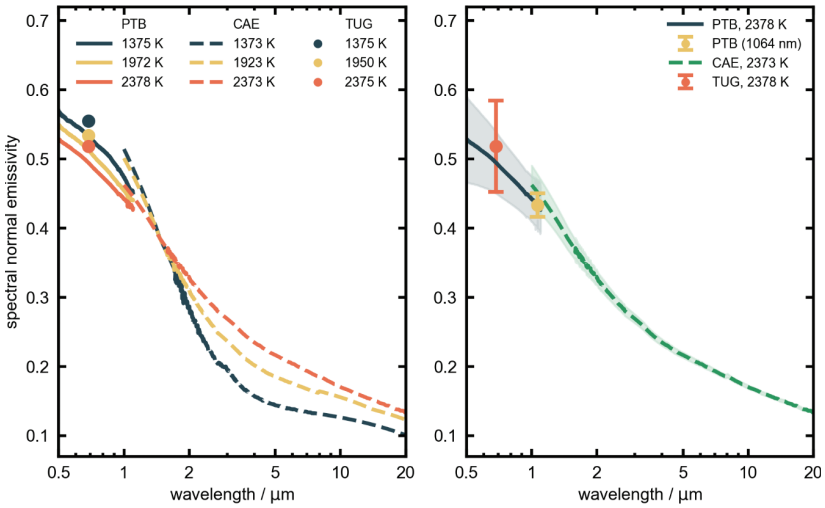

FIGURE 5
Spectral normal emissivity of sandblasted (PTB, CAE) and burnished (TUG) tungsten. Left: Overview of the spectral emissivity at different temperatures. Right: Visualization of the uncertainties ($k = 1$) of the PTB, CAE and TUG emissivity values at the highest measurement temperature of around 2375 K.

Overall, the relative uncertainties range from 3.5% to 11.6% for PTB, 1.3% to 10.5% for CAE, depending on the wavelength and measurement temperature, and roughly 12.5% for TUG. A more detailed overview is given in table 3.

TABLE 3
Relative uncertainty for the spectral emissivity of sandblasted (PTB, CAE) and burnished (TUG) tungsten

	Rel. uncertainty ($k = 1$) for spectral emissivity of tungsten / %				
T / K	**PTB 1064 nm**	**PTB (500 … 1000) nm**	**CAE (1 … 20) µm**	**TUG, solid 684.5 nm**	**TUG, liquid 684.5 nm**
~ 1375	3.8	9.9 … 6.8	10.5 … 1.6		
~ 1974	3.5	9.0 … 6.7	7.5 … 1.4	12.1	
~ 2381	5.0	11.6 …7.5	6.1 … 1.3	12.7	
~ 3800					40.6

Relative uncertainties of PTB, CAE and TUG measurements of the spectral emissivity of sandblasted (PTB, CAE) and burnished (TUG) tungsten. The relative uncertainties generally decrease with increasing wavelengths for the spectrally resolved measurements of PTB and CAE.

Figure 6 depicts the results for the spectral normal emissivity at TUG's measurement wavelength of 684.5 nm as a function of temperature. For the solid phase, PTB values are shown as blue dots and TUG as a red line. Once again, the metallic behavior of the emissivity is confirmed, with emissivity lower

with increasing temperature for wavelengths below λ_X. TUG also measured the emissivity in the liquid phase up to temperatures of 4200 K with increased relative uncertainties of roughly 40.6%.

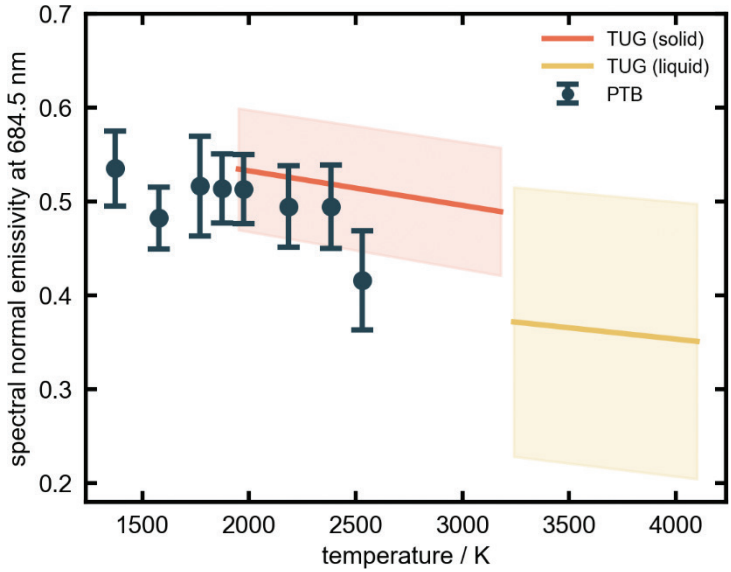

FIGURE 6
Spectral normal emissivity of sandblasted (PTB) and burnished (TUG) tungsten at 684.5 nm as a function of temperature for the solid (PTB, TUG) and liquid phase (TUG). Uncertainties are shown at the $k = 1$ level.

The results obtained by PTB, CAE and TUG for the spectral normal emissivity are in good agreement across the whole temperature range as seen by the overlapping uncertainty intervals in the wavelength range from 684.5 nm to 1100 nm. The full dataset is published as a data repository on Zenodo [16].

4.3 Molybdenum

An overview of the measurement results of PTB, CAE and TUG for the spectral emissivity of sandblasted (PTB, CAE) and burnished (TUG) molybdenum in the temperature range between 1373 K and 2073 K as a function of wavelength is presented in figure 7 on the left. The representation is analogous to figure 5.

The spectral emissivity behaves similarly to the spectral emissivity of sandblasted tungsten with respect to the wavelength and temperature dependence. Here again, a crossover point confirms typical metallic behavior.

The right side of figure 7 shows the realized ($k = 1$) uncertainties of PTB and CAE at the highest measured temperature of around 2375 K, with representation analogous to figure 5. Overall, the uncertainties range from 3.8%

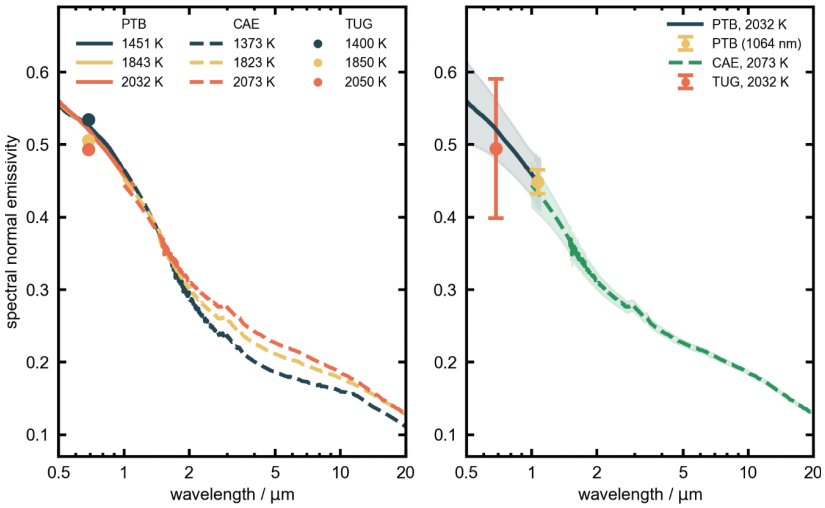

FIGURE 7
Spectral normal emissivity of sandblasted (PTB, CAE) and burnished (TUG) molybdenum. Left: Overview of the spectral emissivity at different temperatures. Right: Visualization of the uncertainties ($k = 1$) of the PTB, CAE and TUG emissivity values at the highest measurement temperature of around 2050 K.

to 10.1% for PTB, 1.4% to 10.5% for CAE, depending on the wavelength and measurement temperature, and around 19.0% for TUG. A more detailed overview is given in table 4.

TABLE 4
Relative uncertainty for the spectral emissivity of sandblasted (PTB, CAE) and burnished (TUG) molybdenum

	Rel. uncertainty ($k = 1$) for spectral emissivity of molybdenum / %				
T / K	**PTB 1064 nm**	**PTB (500 … 1000) nm**	**CAE (1 … 20) µm**	**TUG, solid 684.5 nm**	**TUG, liquid 684.5 nm**
~ 1375	3.8	15.2 … 8.7	10.5 …1.7		
~ 1825	6.7	10.7 … 7.2	7.9 …1.4	18.1	
~ 2073	4.1	10.0 … 7.0	7.0 …1.4	19.9	
~ 3000					16.7

Relative uncertainty of PTB, CAE and TUG measurements of the spectral emissivity of sandblasted (PTB, CAE) and burnished (TUG) molybdenum. The relative uncertainties generally decrease with increasing wavelengths for the spectrally resolved measurements of PTB and CAE.

Figure 8 depicts the spectral emissivity at TUG's measurement wavelength of 684.5 nm as a function of temperature, with representation analogous to figure 6. Again, the spectral emissivity shows a trend similar to that of the

tungsten sample. The emissivity of the liquid phase, however, rises after a sharp drop from 0.25 at 2550 K to 0.31 at the maximum temperature of 3200 K (measured by TUG with slightly lower relative uncertainties of roughly 16.7%).

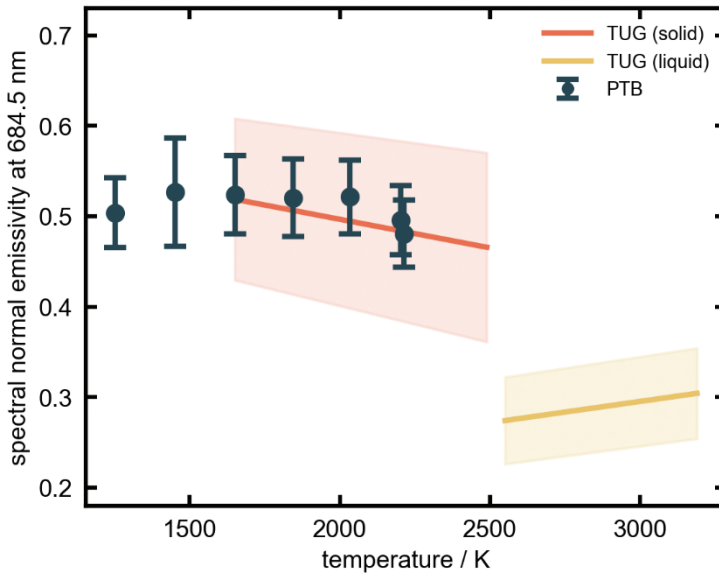

FIGURE 8
Spectral normal emissivity of sandblasted (PTB) and burnished (TUG) molybdenum at 684.5 nm as a function of temperature for the solid (PTB, TUG) and liquid phase (TUG). Uncertainties are shown at the $k = 1$ level.

The results obtained by PTB, CAE and TUG for the spectral normal emissivity are in good agreement across the entire temperature range as seen by the overlapping uncertainty intervals in the wavelength range from 684.5 nm to 1100 nm. The full dataset is published as a data repository on Zenodo [17].

5 DISCUSSION

The inter-laboratory comparison investigated the agreement in high-temperature spectral emissivity measurements of solid materials performed on a European level by three participating institutions, namely the Physikalisch-Technische Bundesanstalt (PTB), the Center for Applied Energy Research (CAE), and the Graz University of Technology (TUG). The experimental setups used to measure the spectral emissivity differed with regard to the radiometric, calorimetric and polarimetric principles applied and to the employed heating methods (pulse or induction heating). The described comparison

allowed the experimental validation of the different setups with respect to achievable measurement uncertainty.

Within the framework of the Hi-Trace project, other thermophysical properties, e.g., the thermal diffusivity [18–20] and the heat capacity [5–7] for the three refractory materials (samples from the same batch and source), were also measured and the results recently published in a Zenodo open access repository in the community "Metrology of high temperature thermophysical properties" [21].

In the absence of reference materials for spectral emissivity at temperatures above 1000°C (with the exception of blackbody cavity radiators with emissivity near unity), the three refractory materials used in this comparison – combined with the spectrally resolved emissivity data presented – could be considered as potential reference materials in high-temperature applications, radiation thermometry and radiometry. The surface roughness of the metal sampes however would need to be carefully measured using a standard technique and matched to the surface conditions of the reported samples.

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DATA AVAILABILITY

Datasets of spectral emissivity values versus temperature are available in a Zenodo open access repository in the community "Metrology of high temperature thermophysical properties".

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