Contents lists available at ScienceDirect



International Communications in Heat and Mass Transfer

journal homepage: www.elsevier.com/locate/ichmt



# Development of a micro-infrared thermometry method for surface temperature distribution and variation of millimeter particle during high-temperature reaction

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## ARTICLE INFO

Keywords: Single particle Infrared thermometry High-temperature reaction Surface temperature distribution

# ABSTRACT

Temperature strongly affects the conversion efficiency, local reactivity, and particle stabilization of the reactions, particularly at high temperature. This study integrated infrared thermography with a high-temperature hot stage reactor to develop a methodology of continuous temperature measurement for a single particle reaction at high temperatures. The external optical transmission transmittance and temperature field core were corrected, and the relationship between the measured temperature ( $T_t$ ) and true temperature ( $T_0$ ) was obtained by adjusting the sample emissivity. Sample temperature with known emissivity at elevated temperature was measured with 1% deviation. The instantaneous temperature measurement for the gasification process was carried out and the overall emissivities of coal particles were corrected. Temperature distribution and variation on the coal particle surface during the gasification process were further obtained and consistent with the previous research results. The proposed online temperature measurement method provides a new approach for gaining insights into particle reaction states.

# 1. Introduction

As a non-contact temperature measurement method, the temperature measurement technology of infrared thermal imaging has been widely used in the fields of military, chemicals, electric power, and metallurgy [1,2], In the field of energy and chemical engineering, for gas-solid reactions occurring at high temperatures, accurate temperature measurement is of paramount importance for enhancing thermochemical conversion efficiency and reaction rate and elucidating the reaction mechanism [3,4].

The conversion of gas-solid reactions exhibits a strong dependence on particle temperature [5–7]. The variability of particle surface reactivity is influenced by factors such as the inhomogeneous distribution of ash and volatiles, as well as differences in particle surface structure [8]. For reactions with significant thermal effects, such as gasification or combustion, particle surface temperature differences are a visual reflection of the influence of those factors on the reaction process, while temperature differences also change the local reaction properties. Furthermore, the non-uniform temperature distribution on the particle surface results in the appearance of the additional forces acting on the particle surface, which was named the reaction-induced force, affecting the fluctuation and rotation of the particles during high-temperature reactions, further influencing the dispersion of the particles within the fluidized bed [9–11]. However, the investigation of reaction-induced forces resulting from variations in temperature distribution had predominantly relied on numerical simulations, with limited literature available focusing on experimental-level analysis of temperature distribution of single-particle surfaces [12,13].

Local temperature differences of the particle are usually not taken into account in the previous study, resulting in deviations from the

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https://doi.org/10.1016/j.icheatmasstransfer.2024.107695

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actual temperature [14,15]. However, for the gas-solid reaction with significant thermal effects, such as the combustion of pulverized coal. Coal quality characteristics affect the combustion temperature. Higher-order coal has higher maturity and lower volatile and ash content, and the flame temperature of higher-order coal is 300 K higher than that of lower-order coal at 1400 K, and there is also a significant difference in the temperature of the particle surface [10]. Hence, it is necessary to experimentally investigate the particle temperature distribution to provide basic data support for further measuring the effect of temperature inhomogeneity on the reactivity and motion of particles.

Obtaining the accurate surface temperature of a single particle during reactions through direct contact measurements is highly challenging due to factors such as particle size and reaction rate [16,17]. Infrared thermometry has been widely used in the field of temperature measurement under the assumption of a gray body. Whereas, for reactions at high temperatures (>1000 K), the temperature measurement accuracy was affected by the particle surface emissivity and ambient thermal radiation, resulting in primary deviations in the temperature measurement results [18,19]. Therefore, it is necessary to develop a method for measuring the temperature distribution phenomena during singleparticle gas-solid reactions.

Non-invasive optical thermometry is a fundamental experimental method for the study of reactions. Currently, radiometric thermometry is the prominent method to obtain the average particle temperature, and this method encompasses various techniques, including monochromatic, bicolor, trichromatic, and full-band methods [20-22]. The monochromatic method faced limitations due to constraints in temperature measurement equipment and reactor size, posing challenges in directly measuring the surface temperature distribution of micrometersized particles. Brix et al. [23] determined the temperature difference between particles during the combustion of millimeter-sized singleparticle pulverized coal up to 700 K using the monochromatic method. The bicolor method is generally based on the ash-body assumption to reduce the effect of emissivity and has high accuracy [24]. Schneider et al. [25] examined the evolution of coal particles during combustion in an oxygen-rich atmosphere using the bicolor pyrometer, and the results indicated that the mean particle temperature was about 1800 K with a variance of 100 K. The trichromatic method offers superior temperature measurement accuracy while poor dynamic temperature measurement range. Khatami et al. [20] developed a specific trichromatic pyrometer to obtain surface temperatures under the assumption that the emissivity was either wavelength-independent or a linear function within the range of 60-1000 nm, and the temperature difference of the two above assumptions as reported was about 40 K. Full-band thermometry is generalizable and easy to operate but has a large measurement error for non-gray bodies. Full-band thermometry is a versatile and direct method but has large measurement errors for non-gray bodies. From those studies based on thermal radiation sensors, it was clear that the accuracy of temperature measurements depended on the assumptions made about the spectral emissivity function. Notably, current studies were mostly focused on flame thermometry or combustion of particle clusters in the entrained bed, and limited studies focused on the temperature distribution of single-particle gasification processes at the milli or micron scale.

The accuracy of temperature field measurements based on infrared thermal imaging systems is related to a variety of factors. However, based on the results of the above studies, the emissivity measurement of the target sample is the key parameter affecting the infrared temperature measurement accuracy [26,27]. While the radiometric calibration technique employed in infrared thermal imaging cameras can be used to establish a mathematical model for the emissivity and radiometric temperature measurements, the technique is only effective for static objects under specific conditions. Factors such as particle structure, composition, and temperature variations affect the sample surface emissivity, and experimental determination of the sample emissivity remains necessary [28]. Kan et al. [29] calculated the temperature and

emissivity of samples in the temperature range of 913-1255 K based on multi-band near-infrared radiation spectroscopy using least-squares fitting with a relative measurement error of <6%. Wen et al. [30] employed radiation spectroscopy to measure the emissivity of stainless steel within the temperature range of 700-900 K and evaluated linear and logarithmic emissivity models. The spectral emissivity of stainless steel at the tested temperatures conformed to a quadratic polynomial model. Yan et al. [17] measured the emissivity and temperature of a type R thermocouple simultaneously using a spectrometer and a high-speed camera, and the thermocouple emissivity was similar to pure platinum and conformed to the gray-body assumption under the conditions of 1450–1530 K. In conclusion, it is feasible to establish a mathematical model of emissivity and radiometric temperature measurement by adopting radiometric calibration. However, the effect of spatial inhomogeneity of emissivity on the accuracy of the thermometry results needs to be further considered.

This study aimed to develop a non-invasive and online method for measuring the surface temperatures of particles within the millimeter or micron range during high-temperature reactions. In conventional reactors, capturing the individual particle reaction process using imaging equipment is challenging due to the influence of airflow, with the low spatial resolution. In this study, an infrared camera was combined with a high-temperature hot stage to enable the measurement of temperature distribution during the gasification process of individual particles. To achieve this, an infrared camera was calibrated using calibrated thermocouples, and external optical transmission and emissivity were corrected for the radiant temperature measurement process. Afterward, the feasibility of the method was verified using sample particles with known emissivity, and the distribution characteristics of particle temperatures during coal gasification were measured.

## 2. Materials

In this study, a variety of particles with different particle sizes were selected to verify the feasibility and accuracy of the online temperature measurement system at high temperatures. The detailed characteristics and parameters of the samples are shown in Table 1 and Fig. 1. The accuracy of temperature measurements obtained from a thermal infrared camera is impacted by thermal radiation diffraction. Typically, for regular objects at the micrometer or nanometer scale, the impact of thermal radiation diffraction effects on temperature measurements is relatively minimal owing to their relatively uniform radiation characteristics [17,24]. Consequently, in the quasi-static temperature measurement validation experiments, thus. Samples of various micron scale materials were used and fashioned into thin slices with a 1 mm diameter using molds to mitigate the diffraction effect on the measurement results. For example, the surface morphology, 3D reconstruction, and surface height fluctuation of the CaO sample are shown in Fig.  $2(a_1)$ (a<sub>3</sub>), respectively. The particle surface was observed to be flat and smooth, with the thickness of the pressed particle flake measuring at 210 µm. Analysis of the relative height data indicated that the particle surface height fluctuation was within 10  $\mu$ m, which was regarded as a homogeneously flat surface for millimeter-scale flakes. These samples were positioned at the central notch of the high-temperature hot stage, and each experiment set was conducted five times to guarantee the precision of the test results.

The emissivity of a wide range of materials is a function of temperature. According to the previous studies [31,32], those are expressed as least squares polynomials fitted to experimental data as shown in Eq. (1). Notably, the set temperature ( $T_a$ ) inside the high-temperature hot stage was taken as the temperature in Eq. (1) in this study, and the emissivity results of the materials used in this study are shown in Fig. 1.

$$\varepsilon_0(T) = a + 10^{-5}bT + 10^{-8}cT^2 \tag{1}$$

where *a*, *b*, and *c* are fitted coefficients.

#### Table 1

Characteristics and emissivity parameters of the materials used in this study.

Material	Purity (%)	Granularity (µm)	ε	Emissivity	а	b	c
Si	99.5	40	0.69–0.70		0.71	-4.32	2.72
SiO <sub>2</sub>	99.9	2	0.60 - 0.37		0.61	25.8	-31.4
CaO	98.0	30	0.89-0.83		1.28	-64.0	22.7
$Al_2O_3$	99.0	60	0.59-0.45		0.98	-53.0	10.2
Fe <sub>2</sub> O <sub>3</sub>	99.0	30	0.75-0.85		-0.01	161	-75.0



**Fig. 1.** Emissivity variation of the materials used in this study at different set temperatures  $(T_a)$ .

Besides, a typical gas-solid reaction with significant thermal effects, in-situ gasification of coal particles, was selected for the experiment, while the temperature variations during the reaction process were measured online. The feedstock chosen for the gasification experiments was Yili coal (Xinjiang, China) with particle sizes ranging from 0.8 to 1.2 mm. The coal sample was dried in an oven at 105  $^{\circ}$ C. The proximate analysis and ultimate analysis are shown in Table 2. Proximate analysis refers to the determination of the moisture, volatile matter, fixed carbon, and ash content of a sample, while ultimate analysis refers to the determination of the elemental composition of the organic materials in the sample, typically including carbon, hydrogen, nitrogen, sulfur, and oxygen content.

Before instantaneous temperature measurement, the infrared test surface of the coal char particle was smoothed through grinding and polishing to ensure consistency in the surface structure and avoid the impact of surface structural inhomogeneity and thermal radiation diffraction on the temperature distribution. The optical morphology, 3D reconstruction height map, and surface height fluctuation are shown in Fig.  $2(b_1) \sim (b_3)$ , respectively. The surface morphology demonstrated the smooth particle surface with distinct contours. A minimal height fluctuation was observed in the particle surface, and the relative height

#### Table 2

Composition and reactivity of coal samples used in this study.

data in Fig. 2(b<sub>3</sub>) shows the fluctuations within 30  $\mu$ m in the X and Y directions. The peripheral region exhibited more variation. Notably, the fluctuation of the surface height rapidly decreased to <10  $\mu$ m within the range of the relative distance of 0.1–0.6 mm, confirming the homogeneity of the surface structure. (See Fig. 2).

Meanwhile, the particle weight loss was used during coal particle gasification experiments to calculate the rate of thermochemical conversion. The conversion curve was taken as a reference for the temperature measurement experiment. Further, the gasification reactivity at 90% conversion ( $\tau_{0.9}$ ) was calculated to evaluate the overall reactivity [33]. As shown in Table 2, the temperature had a significant effect on the gasification reactivity of coal particles, and the gasification reactivity at 1173 K, 1273 K, and 1373 K are  $0.0014 \text{ s}^{-1}$ ,  $0.0044 \text{ s}^{-1}$ , and  $0.0094 \text{ s}^{-1}$ , respectively. Besides, coal particles with different conversions were prepared by controlling the reaction time.

During the gasification process, the carbon in the coal was gradually consumed, forming ash particle. Due to the complex composition and structure of coal and ash, sample emissivity is difficult to accurately characterize in the literature and needs to be corrected using radiation temperature. The variation of sample surface emissivity during gasification could be obtained by fitting the emissivity of coal particles for different reaction stages. In this experiment, the true temperature of the coal can be regarded as corresponding to the temperature in the center of the hot stage. The measured radiant temperature was recorded by the FLIR A615 infrared camera with an assumed emissivity value of one. Hence, after determining the relationship between the measured and true temperature in the following section, the emissivity of coal particles with different conversions could further be calculated combined with Eq. (8).

#### 3. Methodology

#### 3.1. Online temperature measurement system

To facilitate the observation and recording of the reaction process, an infrared temperature measurement device (FLIR A615, America) combined with a hot stage (Linkman, Britain) was constructed, as illustrated in Fig. 3. The hot stage temperature was measured by a thermocouple placed inside the heated crucible, which was calibrated before its initial use. The heating process and gasification experiments were conducted within the high-temperature hot stage, where the sample was brought to a set temperature at a ramp rate of 25 K/min, while the infrared camera was utilized to record the temperature distribution of particles during the gasification process through the visualization window located on the top of the hot stage. To avoid the influence of the difference in the spatial

	Proximate analysis (ad, wt%)					Ultimate analysis (ad, wt%)			
Components	М	v	FC	А	С	Н	S	Ν	0*
	6.23	29.96	54.18	9.63	63.64	4.03	1.22	1.04	15.13
Reactivity		1173 K			1273 K			1373 K	
$\tau_{0.9} \ (s^{-1})$		0.0014			0.0044			0.0094	

The oxygen content was obtained by difference.



Fig. 2. The optical surface morphology, 3D reconstructed height map, and surface height fluctuation of CaO flake  $(a_1 \sim a_3)$  and coal char test surface  $(b_1 \sim b_3)$ , respectively.



Fig. 3. Schematic diagram of infrared thermography system.

position of the particles on the accuracy and reproducibility, the position of the thermal imaging camera and the high-temperature hot stage has been fixed in the pre-calibration work, i.e., the focal point of the infrared camera and the center of the hot plate in the z-direction are coincident. A 1.2 mm diameter groove was cut in the center of the heating crucible to fix the position of the sample.

The FLIR A615 infrared camera is equipped with an uncooled vanadium oxide detector, which generates a high thermal image of  $640 \times 480$  pixels in the long infrared wavelength band (7.5 µm–14 µm). Depending on the test object, the temperature measurement range can be adjusted to +573–2273 K, with a temperature measurement accuracy of  $\pm 2$  K. Before the experiment, pre-measurement calibration and optimization were conducted, including the amorphous silicon layer macro lens and the use of a standard heat source to calibrate the thermal camera, aiming to reduce the influence of thermal radiation diffraction effect. Additionally, the atmospheric radiation can be automatically corrected according to the camera measurement distance, atmospheric

temperature, and relative humidity; the non-uniformity response drift of the detector through the built-in calibration program is automatically corrected; reflective apparent temperature correction is based on the input reflection temperature. To observe millimeter-sized particles within the hot stage chamber, a  $1.5 \times$  macro lens with a field of view (FOV) of 7°x5.3° was used in this study. In this experiment, the distance between the object and the camera lens was fixed at 0.13 m. The actual area of each pixel is to be 24.9 µm × 25.0 µm based on the trigonometric relationship in Eq. (2).

$$d_i = \frac{2 \cdot \tan\left(\frac{\theta_i}{2}\right) \cdot h}{n_i} \tag{2}$$

where  $\theta$  is the field of view, *h* is the working distance between the object and the camera, *n* is the pixel resolution, and the subscript *i* represents the orientation of each pixel point, including the horizontal direction *x* and the vertical direction *y*.

## 3.2. IR camera calibration

#### 3.2.1. Temperature calculation principle

When measuring the particle temperature inside the hot stage, the infrared radiation entering the infrared thermal camera contained two components, the real radiation of the object and the reflection of the environmental infrared radiation. Thus, the total radiation intensity can be expressed as Eq. (3).

$$L = \tau_{\rm b} \cdot \varepsilon \cdot L(T_0) + \tau_{\rm b} \cdot (1 - \varepsilon) \cdot L(T_{\rm e})$$
(3)

where *L* is the total radiation intensity received by the infrared thermal camera;  $\tau_{\rm b}$  is the optical transmission transmittance;  $\varepsilon$  is the sample emissivity;  $T_{\rm e}$  is the environmental temperature;  $T_0$  is the true temperature.

According to the previous study [34], in the 7.5–14 mm band, based on Planck's law, the radiated powder can be written approximately as Eq. (4).

$$L_{R}(T_{0}) = \int_{\Lambda_{\lambda}} L_{\lambda}(T_{0}) d\lambda \approx C T_{0}^{1/n}$$
(4)

where  $\Delta \lambda$  is the wavelength range of infrared radiation received by the camera, *C* and *n* are the parameters related to the spectral bands received by the infrared thermal camera, which can be obtained by fitting and is independent of the sample to be measured.

The deviation between both measured and true temperatures relied on the radiation intensity. As shown in Fig. 3, in this experiment conducted in the hot stage, the sample was placed in the center of a heated crucible with an inner diameter of 5 mm, and the overall environmental temperature except for the heated crucible could still be approximated as consistent with room temperature (298 K). When the millimeter particles were heated to a high temperature (>1000 K), the atmospheric radiation on the temperature measurement results was negligible, as shown by the Stephen-Boltzmann law. The true radiation received from the sample is much higher than the atmospheric radiation by two orders of magnitude. Hence, the effect of atmospheric radiation on the temperature measurement results is neglected here. Further, the relation equation between the measured temperature ( $T_r$ ) and the true temperature ( $T_0$ ) can be described as Eq. (5) after considering the external optical transmittance ( $\tau_b$ ).

$$T_{\rm r} = \varepsilon_{\lambda}^n \cdot \tau_b^n \cdot T_0 \tag{5}$$

The above equation was corrected to Eq. (6) after considering the effect of external transmission efficiency and transmittance.

$$T_{\rm r} = \varepsilon_{\lambda}^{\mu} \cdot \tau_b^{\mu} \cdot T_0 \tag{6}$$

where m is the radiative correction parameter.

## 3.2.2. External optical transmission

The external optical transmission is the parameter assigned to the infrared camera that supports its internal measurement function and generates the temperature value. Under experimental conditions (1173 K–1373 K), to determine the percentage transmittance of the calcium fluoride window, the average temperature of the heated chamber (sapphire sheet) inside the hot stage was first recorded under conditions without the calcium fluoride window, and the true temperature ( $T_0$ ) of the sapphire sheet is the set temperature ( $T_a$ ) inside the hot stage core. Transmittance is a state function of ambient conditions and is independent of the characteristics of the sample, hence, sample emissivity and external transmittance could be specified as 100% initially. After that, the window was installed and the external optical transmission parameters were modified until the average temperature of the surface was equal to the temperature measured without the window.

Fig. 4 shows the results of temperature measurement and window transmission correction. The results showed that as the temperature



**Fig. 4.** Radiant temperature measurements  $(T_r)$  of sapphire sheet at different set temperatures and external optical transfer correction curve.

increased, the external optical transmission efficiency decreased slightly, but the variation exhibited minimal fluctuation (<0.01). As a result, the external transmission efficiency could be deemed stable in the range of 1173–1373 K, and the infrared transmittance of the calcium fluoride glass in the 7.5–14 mm band was measured to be 0.91 with a m of 1.93.

#### 3.2.3. Temperature field measurement

In this experiment, the true surface temperature of the samples was regarded as equal to the temperature field where this sample was placed in the hot stage. The inner surface of the core of the high-temperature hot stage consists of a smooth and flat sapphire sheet, which has a fixed emissivity of 0.65 and behaves as an approximate gray body. The distribution of the temperature field inside the hot stage is calculated directly from the infrared camera with input parameters including sample emissivity, optical transmission, and set temperature. Meanwhile, to assess the reliability of the temperature field measurement obtained from the infrared camera, two calibrated thermocouples were introduced to test temperatures. The first thermocouple was embedded on the outer side of the hot stage core, which was named TC1. While the other, named TC2, was positioned in the center of the core, where the particle was placed. These thermocouples measured the temperatures at the edge and center of the hot stage core, respectively, serving as means to validate the accuracy of the temperature field determined from the



Fig. 5. Schematic diagram of the high-temperature hot stage reactor core structure.

infrared camera measurements, and the schematic diagram is shown in Fig. 5. The thermocouple temperatures were recorded via a data acquisition module, and it is worth noting that only the tip ( $\sim 2$  mm) measurement portion of the TC2 was placed at the center of the table core. The thermocouple temperature was used as the true value for the temperature measurement, and the emissivity was fine-adjusted until the IR temperature at that specific location matched with the thermocouple results.

The temperature distribution field in the sample heating area is shown in Fig. 6. The temperature measured zone is the center of the crucible inner surface, with a region of 1.5\*1.5 mm, as shown in Fig. 5. Compared with the set temperature  $(T_a)$ , the temperature in the center area was slightly lower, approximately 20 K. Due to the distribution of the heating resistance wire, the background temperature was unevenly distributed on both sides, with a temperature difference of up to 10 K. Meanwhile, the background temperature difference increased slightly with the increase in set temperature. Previous studies have typically used the thermocouple temperature as the average temperature of the heating field [35-37]. However, this approach inevitably increased measurement errors. Therefore, the temperature distribution of the actual heating field needed to be further considered when measuring the temperature distribution on the particle surface and the average of the temperature field was taken as the true temperature of the sample particle at that specific location.

## 3.2.4. Parameter calibration

The infrared camera provided the measured radiation temperature  $(T_r)$  of the sample at the set temperature  $(T_a)$ , and further, based on Eq.

(6), the true temperature  $(T_0)$  of the sample could be calculated. To determine the relationship between the measured temperature and emissivity, the emissivity parameter of the infrared camera was varied and the variation of the measured radiant temperature was recorded. The measured temperature was directly obtained from the camera, while the true temperature of the sample was the temperature at the center point of the hot table, where the sample was placed. Therefore, the parameter *m* was determined by fitting Eq. (7).

$$ln\left(\frac{T_r}{T_0}\right) = n \cdot ln(\varepsilon_r) + m \cdot ln(\tau_b)$$
<sup>(7)</sup>

As shown in Fig. 7, the radiative correction parameter n is 1.91. Hence, for a sample with a known emissivity, the true temperature could be obtained through Eq. (8).

$$T_0 = \frac{T_{\rm r}}{\varepsilon_{\lambda}^n \cdot \varepsilon_b^m} \tag{8}$$

where m is 1.93, and n is 1.91 in this study.

#### 4. Results and discussion

#### 4.1. Quasi-static temperature measurement

The temperature measurement results of the materials used in the experiment are shown in Fig. 8. The emissivity of the sample has been determined and is a fixed value at a specific temperature, which is also listed in Fig. 1. The corrected center temperature of the hot stage was regarded as the true temperature ( $T_0$ ) of the samples. According to



Fig. 6. Temperature field distribution in the center of the high-temperature hot stage at different set temperatures.



Fig. 7. Fitted calibration curves for the parameters of the equations.

Fig. 6, it was lower than the set point by about 10–20 K, and this difference increased as the surrounding temperature increased. Fig. 8(a) shows the deviation of particle temperature measurement results at different temperatures. The method had favorable measurement results with high repeatability for samples with known emissivity, while the total temperature measurement error increases slightly with increasing temperature. The deviation of results had high repeatability within 10 K for different temperatures, which proved the generalizability of this method.

In Fig. 8(b), the horizontal coordinate represented the ratio of the measured temperature results ( $T_{exp}$ ) to the true temperature ( $T_0$ ), which aimed to visualize the measurement error of the sample. Fig. 8(b) demonstrated that the error between the experimental measurements and the true temperature values fell within the range of ±1%, proving

the accuracy of this method. The experimental results for various pure particle materials exhibited a distribution on both sides of the true value. This distribution confirms that, within the acceptable error tolerance, the method had reliable test results for materials with known emissivity. Compared with the measurement results of different materials, Si and  $Al_2O_3$  have better results than the other materials because of the stable emissivity.

## 4.2. Sample emissivity correction in reaction

The emissivity of the samples at different gasification stages was determined based on the sample temperatures obtained from infrared thermography measurements, and the result is shown in Fig. 9. The emissivity measurement results of different coal particle samples under the same reaction conditions revealed that the coal surface emissivity variability was within 5% confirming surface composition homogeneity. Therefore, as the reaction proceeded, the overall distribution of the emissivity to the particle surface is experimentally assumed to be homogeneous While this assumption does result in localized measurement deviations, it is noteworthy that the maximum difference in emissivity between pure ash and pure carbon is approximately 0.07, which can cause a temperature deviation of several of degrees at high temperatures above 1000 K. Nonetheless, for reactions with distinct thermal effects, these errors do not compromise our assessment of reaction conditions and progress.

For coal and ash particles, the sample emissivity was negatively correlated with the reaction temperature in the range of 1173 K to 1373 K, as shown in Fig. 9(a). However, the reaction temperature had a limited effect on the emissivity at 1173 K and 1373 K, with maximum differences of 0.01 and 0.006, respectively. Consequently, in the subsequent investigation, the sample emissivity can be assumed to remain constant with temperature, and the sample emissivity under the



Fig. 8. (a) Temperature measurement results of experimental materials, and (b) temperature measurement deviation.



Fig. 9. Coal and ash sample emissivity correction curve, (a) at different ambient temperatures, (b) for coal with different conversion.

condition of 1273 K was selected as the mean value of emissivity. The emissivity results of coal samples at different reaction stages are shown in Fig. 9(b). With the increase of conversion, the sample emissivity increased nonlinearly. Based on the previous study [17,32,38], an emissivity model was employed to fit the emissivity curve. The results are shown below.

$$\varepsilon = 0.738 - 0.009 \cdot x + 0.072 \cdot x^2 \tag{9}$$

The coal emissivity of 0.738 and the coal ash emissivity of 0.802 are slightly lower than the results reported in the literature [32], the reason for which may be due to the estimation of the ambient temperature.

# 4.3. Instantaneous temperature measurement in gasification

The experiment provided data on the surface temperature distribution of each particle under different test conditions, The coal char emissivity is manually assigned according to the test results in Fig. 9. Fig. 10 illustrates the temperature variation on the particle surface at the set temperature of 1273 K. In the test area of Fig. 10, the difference of the surface temperatures before the reaction was within 19 K. This measurement bias is acceptable for the ambient environment at 1273 K, as it is much smaller than the temperature difference due to the thermal effect. Consequently, for samples of the same scale and location, this method is primarily focused on studying the reaction process, specifically the overall temperature variation in the sample during the reaction and the temperature difference at the same location from the initial state.

During the gasification process, affected by the differences in the surface structure and ash distribution, the surface gasification characteristics of coal were different. It was manifested in the non-uniformity of the temperature distribution on the coal surface, where the coal at the low-temperature point had a fast gasification rate and high gasification activity. The temperature difference at the same time in the gasification process can be >50 K. Nonetheless, the general trend revealed a low center temperature and high surrounding temperature distribution on the particle surface, which was related to the convection and radiation heat transfer between the particle and gas medium. Meanwhile, as shown in Fig. 10, with the gasification, the particles shrank slightly, and the minimum and average temperatures in the middle of the sample surface show a trend of decreasing and then increasing, which correlated with the change in the reaction rate.

In the test results, the temperature distribution within a  $500*500 \ \mu m$  rectangular area in the center of the particle was selected. The results of the pixel points near the edge of the particle were discarded because these test results can be greatly affected by ambient radiation [9]. Additionally, to establish the relationship between the average temperature of the particle surface and the gasification process, we



Fig. 10. Particle surface temperature distribution during the gasification process at 1273 K.

conducted a series of experiments three times, and all the results were obtained by taking the average of the measured data. The variation of the absolute temperature difference  $(\Delta T_t)$  between the initial and t moment during the coal gasification process was shown in Fig. 11. Meanwhile, the mean and deviation of particle temperatures for each group of conditions and the maximum temperature difference on the surface of the same particles were also presented in Table 3.

As shown in Fig. 11, during the gasification process, the variation of the absolute temperature difference shows a peaked shape, proving that the particle surface temperature decreased and increased as the reaction progressed, correlating with the decreased reaction rate. Compared with the reaction time, there was a slight delay in returning to set temperature. Temperature played a crucial role in the gasification process, and the thermal effect became more prominent as the temperature difference  $(\Delta \overline{T}_{0 max})$  of the whole reaction process increased. Additionally, as shown in Table 3, noticeable temperature variations were observed among different particles, and the standard deviation ( $\sigma$ ) of the measured temperature ranged from 7.99 K, 5.02 K, to 4.10 K as the set temperature increased from 1173 K to 1373 K. Low temperature led to a higher reaction time differentiation, leading to higher temperature standard deviations. For the same particle, significant fluctuations in surface temperature were observed, and the maximum temperature difference  $(\Delta T_{surf,max})$  on the surface increased with rising reaction temperature, and the high value was up to 59.28 K, which reflected the variability of the reaction activity on the sample surface.

# 4.4. Comparison and validation

In this study, gasification experiments of single particles were carried out under a pure CO2 atmosphere at 1173-1373 K Since there was no direct literature data for comparison, we only compared the maximum temperature difference ( $\Delta \overline{T}_{0,max}$ ) under different environments, and the results are shown in Fig. 12. Under experimental conditions, measurements with a maximum temperature measurement error of no >10 K are acceptable. The result showed that our experimental results were similar to the temperature results obtained by Kuster et al. [9] for the lignite coal using the colorimetric method of direct measurement. As reported [39], the gasification activity of Anthracite is approximately one order of magnitude lower than that of Lignite, hence, resulting in a lower temperature variation. Considering the particle size effect, the results of Prabhakar et al. [5] are higher about 8 K. Shen et al. [40] obtained the particle temperature based on the experimental results of heat conservation analysis of the gasification process, so there is a certain gap with the results of the present study. Nevertheless, considering the inherent variability in heat transfer and reaction among individual particles, it is reasonable to expect such discrepancies. In more depth, Fong et al. [41] calculated the temperature difference between the internal core region of the particles and the external environment taking the presence of pores and cracks into account. We did not present their results in Fig. 12 due to the great variability of the experimental conditions with our experiments. However, considering the heat transfer impediments between the ash layers of individual particles, it is anticipated that the temperature within the internal core reaction area would be even lower, and further calculations are needed to obtain the true gasification temperature of the sample in future work.

#### 5. Conclusion

This study presented a method for measuring the surface temperature distribution of micro/mm particles using an infrared camera combined with a high-temperature hot stage. Corrections for external optical transmission and temperature field of the hot stage core were realized in combination with the calibrated thermocouple temperatures. The relationship equation between the measured temperature and the true temperature was established. A series of sample particles with known



Fig. 11. Variation of the temperature difference between the initial and t moment during the coal gasification process.

Table 3	
Volatility analysis of particle mean temperature measurements.*	

$T_{\rm a}/{\rm K}$	$\Delta \overline{T}_{0,max}/\mathrm{K}$	$\sigma/K$	$\Delta T_{surf,max}/\mathrm{K}$
1173	17.7	7.99	21.26
1273	28.9	5.02	49.60
1373	56.2	4.10	59.28

 $\sigma$ : Standard deviation of maximum temperature difference among different particles;

 $\Delta T_{surf,max}$  : Maximum difference in the particle surface temperature at the same time.

 $\Delta \overline{T}_{0,max}$ : The maximum temperature difference during gasification process;



Fig. 12. Measured particle maximum temperature difference during gasification compared to the literature data.

emissivity at elevated temperatures were measured and the temperature measurement deviation was within 1%, validating the feasibility of the method. Besides, constant temperature gasification experiments were carried out, and the emissivity of the samples at different reaction stages was measured. The results displayed the distribution and variation of the particle surface temperature during the reaction process, which is an important factor in the gasification process in the particle dispersion and inhomogeneity movement. The online temperature measurement system provides a new approach for in situ observation of high-temperature reactions.

## CRediT authorship contribution statement

Haigang Zhang: Data curation, Formal analysis, Investigation, Methodology, Resources, Software, Visualization, Writing – original draft, Writing – review & editing. Zhongjie Shen: Conceptualization, Formal analysis, Funding acquisition, Methodology, Project administration, Software, Supervision, Writing – review & editing. Jianliang Xu: Formal analysis, Investigation, Resources. Qinfeng Liang: Conceptualization, Investigation, Methodology, Resources, Validation. Zhenghua Dai: Data curation, Methodology, Writing – review & editing. Jianhong Gong: Formal analysis, Methodology. Haifeng Liu: Conceptualization, Formal analysis, Methodology, Supervision, Validation.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

The authors are unable or have chosen not to specify which data has been used.

## Acknowledgments

This study is supported by the National Natural Science Foundation of China (22378130), National Key R&D Program of China (2022YFC3902502-04), Key R&D Program of Xinjiang Uygur Autonomous Region (2022B03026-1), the Fundamental Research Funds of the Central Universities (2022ZFJH004).

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