

Expert System Spectropyrometer Results for non-black, non-grey, or Changing Emissivity and Selectively Absorbing Environments

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Abstract. An expert-system multi-wavelength pyrometer based on a grating spectrophotometer has been able to overcome many well-known difficulties of pyrometry. These include unknown, changing, and/or spectral dependence of emissivity as well as environmental absorption of radiation. In addition to a spectrophotometer and the usual optics, the instrument includes a computer which analyzes each measurement and then returns the temperature, the tolerance (a real-time measure of accuracy), and the signal strength (a quantity directly related to the emissivity at a chosen wavelength). The instrument can save the input data, the thermal spectrum, for each temperature measurement. Accuracy to 0.10% is routinely achieved.

INTRODUCTION

Multi-wavelength pyrometers have been proposed with from three to hundreds of wavelengths (1,2,3,4). An analysis of these and similar instruments concludes that accurate temperature determination cannot be made without knowledge of surface emissivity (5). Since emissivity can change with time and wavelength for any number of reasons – composition, surface finish, processing, temperature, etc. - then clearly the only solution is to gain that knowledge while the measurement is being made.

The instrument described here, the Spectropyrometer, differs from other multi-wavelength pyrometers in that it does not rely on prior knowledge. Instead, it examines the data and makes decisions on how to proceed based on the results of tests that it applies. In addition to the temperature, the instrument displays both the tolerance and the signal strength for each measurement. The tolerance is a measure of the accuracy of the temperature determination. The signal strength is defined as the emissivity at one wavelength as modified by the transmission of the optical path and the fill factor of the field of view.

Since the Spectropyrometer's introduction, tens of thousands of thermal spectra from various targets and processes have been analyzed. Two major challenges to pyrometry have been seen: perturbations of the thermal spectra by absorbing gases and spectral dependence of emissivity. Absorptions of extended portions of the visible and near-infrared spectra have been routinely detected in vessels where graphite is exposed to high temperatures. Absorptions at 650 nm, a significant wavelength in pyrometry, appear to be the rule rather than the exception in such furnaces. The monotonic decrease of emissivity with wavelength is often seen with metal targets. Metals for which emissivity decreasing with wavelength has been observed include molybdenum, nickel, silicon, stainless steel, tantalum, titanium, tungsten, and zirconium. The Spectropyrometer detects and removes the absorptions from the thermal spectrum before calculation of temperature; it also detects and corrects for the monotonic decrease of emissivity with wavelength.

The Spectropyrometer is also insensitive to noise: 10% random noise introduces less than 1% uncertainty in the measured temperature. Another novel feature is that a radiation calibration is used because the instrument is based on a spectrophotometer. The device need only be calibrated at one well-known temperature (or with one well-known spectrum, as from a standard lamp). All subsequent temperatures are calculated relative to the known spectrum so the device is accurate throughout its range without extrapolation or interpolation. All temperatures are therefore calculated from first principles. The instrument's narrow bandwidth and wide spectral ranges translate

directly to broad thermal ranges of at least 1700°C for each model. Models using the visible and near-infrared (500 – 1000 nm) have been used from below 800 to above 3600°C; models sensitive in the infrared spectrum (1000 – 2200 nm) have been used down to 300°C. Agreement to within 0.10% with traceable standards is routine.

APPROACH

The mathematics for ratio pyrometry are well known. Solving Planck's law for temperature by dividing the intensities at two different wavelengths and making the usual short-wavelength–low-temperature assumption yields:

$$T_{12} = \frac{c_2(1/\lambda_1 - 1/\lambda_2)}{\ln R - 5 \ln(\lambda_2/\lambda_1)}. \quad (1)$$

Here T is the temperature, c_2 the second radiation constant, λ the wavelength, and R the ratio of intensities at the two wavelengths. A straightforward way of doing multi-wavelength pyrometry would be to calculate all the possible T_{ij} for an ensemble of discrete wavelength/intensity pairs and average them. This works quite well for noise-free data collected on greybody or blackbody sources. To extend the usefulness of the technique to non-ideal targets of practical interest an alternative approach has been adopted.

The spectral data are represented mathematically and selected wavelength/intensity pairs are used to calculate the corresponding temperatures. If the results are single-valued, as determined by the size of the standard deviation of this ensemble of temperatures, it indicates that the sample is a greybody and absorptions, if present, are not significant. The tolerance is defined as the standard deviation of the ensemble.

If the standard deviation of the temperatures calculated is large, it indicates one or more of the following conditions:

- emissivity is a function of wavelength;
- atmospheric absorptions or emissions are present;
- the temperature is not single-valued;
- the optical elements have changed characteristics.

The first two items can be detected and corrections can then be made. Metals typically exhibit emissivity that decreases monotonically with wavelength. It has been shown that a three-wavelength device can resolve temperature if the dependence of emissivity on wavelength is linear (6). With the multitude of wavelengths used by the Spectropyrometer this fact can be exploited and tested. Again, it is deemed successful if the result is single-valued, as judged by the size of the standard deviation.

Absorptions or emissions from process or intervening atmosphere or product offgas can also be detected by comparing the behavior of the data spectrum with that of a thermal spectrum. Once detected, these spectral areas can then be removed from the calculation. Again, the agreement of the temperatures calculated from the remaining data indicates how well the temperature is known.

The last two items, many-valued temperatures and changing optical characteristics must be resolved mechanically. The former requires a smaller sample area (different optics); the latter usually means a process upset has occurred, which must be rectified.

DESIGN AND APPARATUS

The Spectropyrometer consists of an optical input and fiberoptic, a grating spectrophotometer with detector array for wavelength separation and detection, and a computer for analysis and display.

A major requirement for the Spectropyrometer is that the detector array has a linear intensity response; an easy test for this is to compare the results from varying the exposure time while keeping the radiant intensity constant

(7). Several spectrophotometers have been used successfully in this application; detectors have been photodiodes and CCDs. FAR currently uses two spectral ranges: 500 – 1000 nm and 1000 – 1700 nm. The shorter-wavelength range has proved suitable for temperatures from around 800 to at least 4000°C, the longer-wavelength range for temperatures from 300 to 2000°C. Fiberoptic cables of up to 100 meters length have interconnected these detectors to lenses, mirrors, and light pipes of commercial and custom manufacture.

An algorithm comprising an expert system to make the calculations and determinations discussed earlier has been rendered in software suitable for a desktop computer (9,13). The substantial amount of storage readily available on these devices makes it easy to store both raw spectra and processed results.

PERFORMANCE

The instruments described here have been commercially available from FAR Associates since 1997 and in limited use and development since 1987. Consequently, a great deal of data has been collected and analyzed. These analyses have demonstrated accurate performance despite emissivity changes, atmospheric interference, and random noise.

Emissivity Changing with Wavelength

The monotonic decrease of emissivity with wavelength has been detected in several metals (Mo, Ni, Si, Ta, Ti, W, Zr, and stainless steels) and in metal carbide coatings on ceramic substrates. Most metals have been observed to behave similarly (8).

The change of emissivity with wavelength can induce large errors in ratio pyrometers. Figure 1 shows the temperatures that straightforward ratio pyrometry calculations would yield on the data collected from tungsten target by a spectrophotometer. The temperatures calculated from the raw data (the jagged line) and the mathematical representation (the smooth line) are plotted against the average of the two wavelengths used for the ratio calculation. The results are indeterminate within a range of 300 Kelvins. The Spectropyrometer calculates the coefficients of the emissivity's dependence on wavelength using pairs of temperatures and their generating wavelengths from Figure 1. The coefficients thus calculated are inserted into a form of Planck's equation for ratio pyrometry to yield the true temperature (9). Figure 2 shows the results of the Spectropyrometer operating on the data of Figure 1. The indeterminacy of Figure 1 has been replaced with a single value: the average is 3058 Kelvins, with a standard deviation of 4.9K.

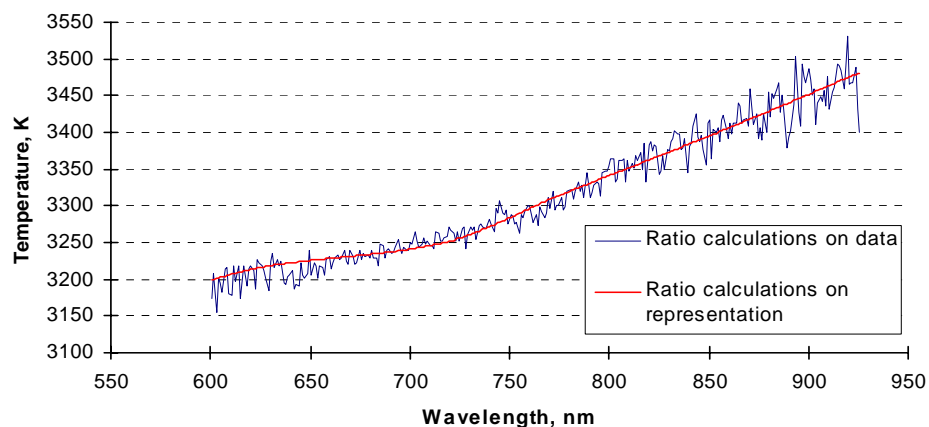


Figure 1. Results of many ratio pyrometry calculations on a tungsten target. Uncertainty is greater than 300K.

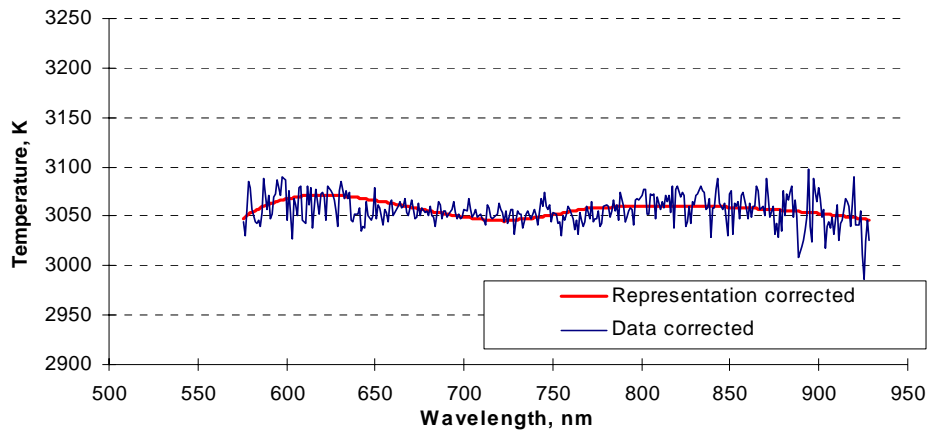


Figure 2. Spectroscopy results on the tungsten target of Figure 1. Uncertainty is 5K.

The spectral emissivity can now be calculated from the raw data (the thermal spectrum) and the measured temperature. The results of such a calculation are shown in Figure 3, along with published data for tungsten (10).

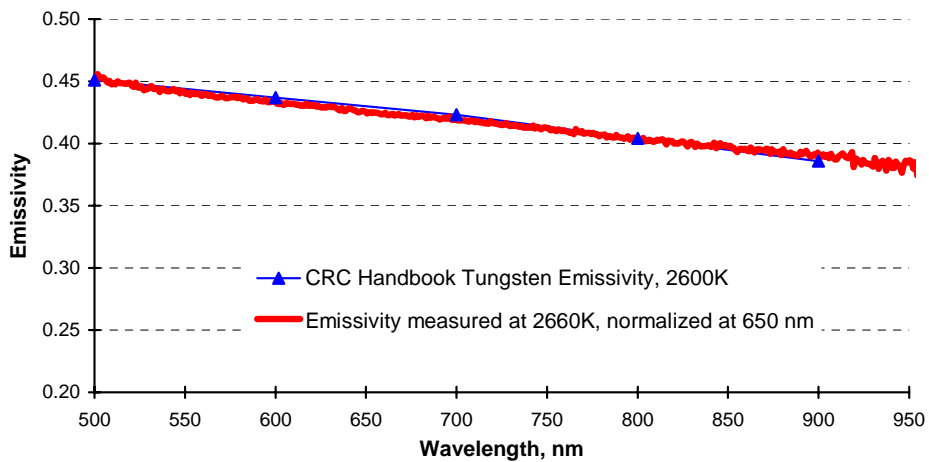


Figure 3. Emissivity calculated as described above, plotted with and normalized to data taken from the CRC Handbook.

The monotonic decrease of emissivity with wavelength in metals, while common, is not universal. Surprisingly small changes in composition have been seen to affect the emissivity greatly. Figure 4 shows the results of measurements made on rods of pressed tantalum powder sintered in vacuum. The proprietary compositions differed by only a few atomic percent of alloying materials. Nonetheless, one behaved as a greybody, the other as a metal with emissivity being a function of wavelength.

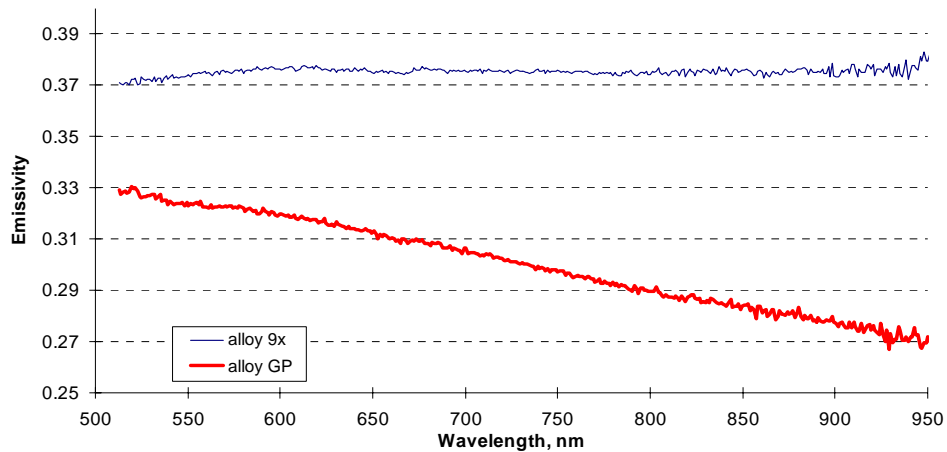


Figure 4. Tantalum metal exhibiting both greybody (alloy 9x) and colorful behavior (alloy GP).

A material’s emissivity behavior is known to depend on the surface morphology. A molybdenum target with two surface modifications was spatially scanned using the Spectropyrometer. The modified surfaces consisted of a light surface knurling and a square-bottomed channel, 1.2 times as deep as its width. The scan used a spot size half the width of the modifications. The scan made several measurements down the length of the modifications, and included a length of unmodified material. Both modified surfaces were more efficient radiators than the unmodified material, but all exhibited the monotonic decrease in emissivity with wavelength associated with metals. The geometry is shown in Figure 5.

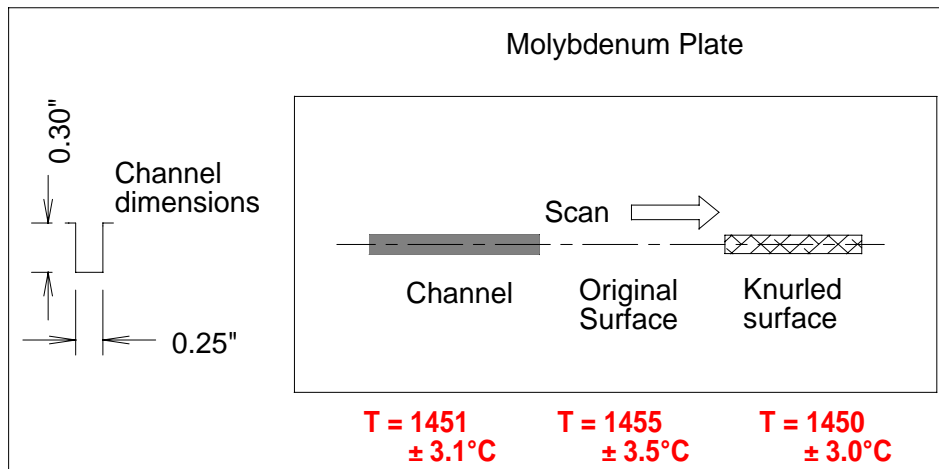


Figure 5. A molybdenum plate with two surface modifications; the scanning spot was 0.12” in diameter.

Despite the emissivity for the three surfaces varying from 12% to 25% over the wavelength range of the Spectropyrometer, the temperatures returned for the three regions agreed very well: 1451 ± 3.1°C in the channel, 1455 ± 3.5°C for the knurled surface, and 1450 ± 3.0°C for the unmodified surface. The emissivities for the three regions are shown in Figure 6.

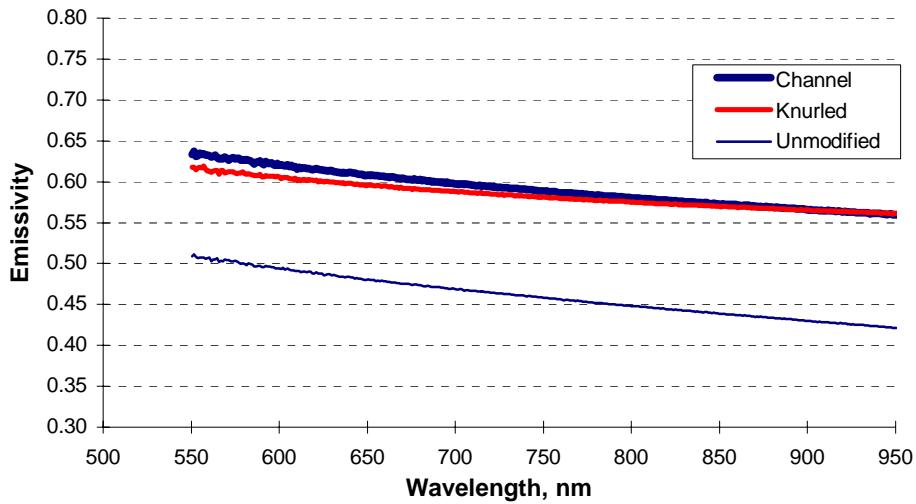


Figure 6. Emissivity of the three scanned molybdenum surfaces of Figure 5.

Silicon metal is known to be a difficult target for non-contact temperature measurement. Consequently, measurements were made on a radiantly-heated silicon target using both the long and short wavelength Spectropyrometers. The data for both is shown in Figure 7; note the Vis/NIR pyrometer had to contend with the added emission of a helium-neon laser. The two Spectropyrometers shared the same optics, thus measuring the silicon target at different points in the process cycle. Despite the differences, the signal strengths as measured by each Spectropyrometer were in good agreement. The target area of the pyrometer optics was 10 times the heated area of the sample, thus the signal strength is multiplied by 10 to correct the values measured to the actual emissivity. The corrected values correspond well with the reported values for silicon metal (11), even showing the bandgap discontinuity at 1100 nanometers.

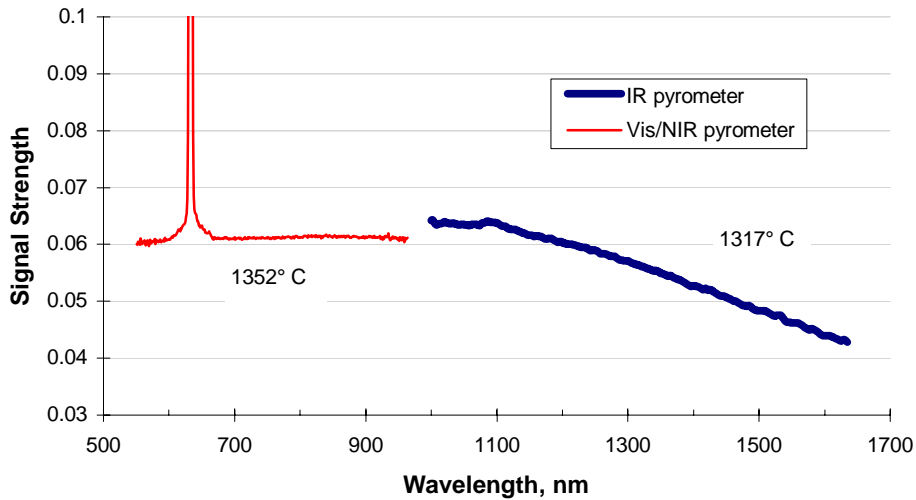


Figure 7. Signal strengths for a silicon target as measured by both the short and long wavelength Spectropyrometers. Emissivity is achieved by multiplying these values by 10, the ratio of the target area to the heated area. The strong emission at 632 nm is an aiming laser left on during processing.

Materials with ceramic coatings are of great technological interest, so a metal carbide-coated graphite sample was the target for measurement of emissivity at elevated temperatures. The sample was placed in a horizontal tube furnace, open on the end that faced the sample coating, and purged with argon from the closed end. A sliding sight tube, also purged, was rigidly attached to a lens assembly which was connected to the Spectropyrometer by a fiberoptic. The optical input of the sight tube was placed just inside the open end of the tube furnace while the furnace was heated to the desired setpoint. The furnace sensor and the Spectropyrometer agreed during this period;

the Spectropyrometer determined radiation from this arrangement was greybody. When the setpoint was reached, the sight tube was plunged into the furnace to within 1 millimeter of the target surface to eliminate sidewall radiation. The probe was allowed to remain in close proximity with the sample for a few seconds, and then withdrawn. Figure 8 shows the temperature history of this sequence.

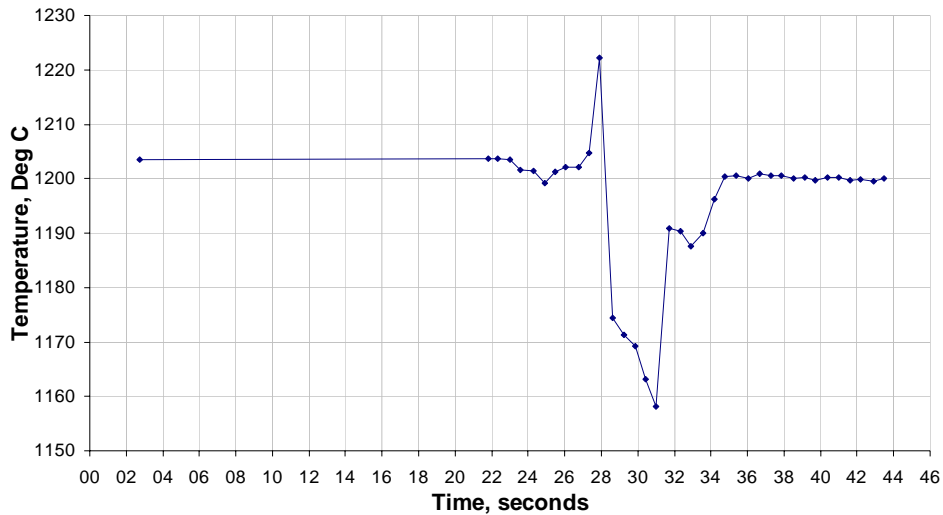


Figure 8. Temperature history for metal carbide emissivity determinations. The probe was stationary from 0 to 23 seconds; inserted from 24 to 28 seconds, at the measuring location from 28 to 31 seconds, removed from 31 to 35 seconds, and stationary until the end of data. The 1220° data point at 27.9 seconds is spurious: as a result of the probe’s motion blackbody and sample radiation were mixed in one measurement. The furnace setpoint was 1200°C for this series.

In contrast to the results during heat-up, data from the close approach to the sample indicated that the emissivity had a substantial spectral dependence, as shown in Figure 9. The temperatures detected at the target surface were consistent with the temperatures returned by the Spectropyrometer before and after the plunge, showing the rapid cooling expected from a small heated body in close proximity with a large cold one. Of interest here is the automatic detection of the change of emissivity from grey to wavelength-dependent as the sidewall radiation was eliminated, and the agreement of the temperatures for both cases.

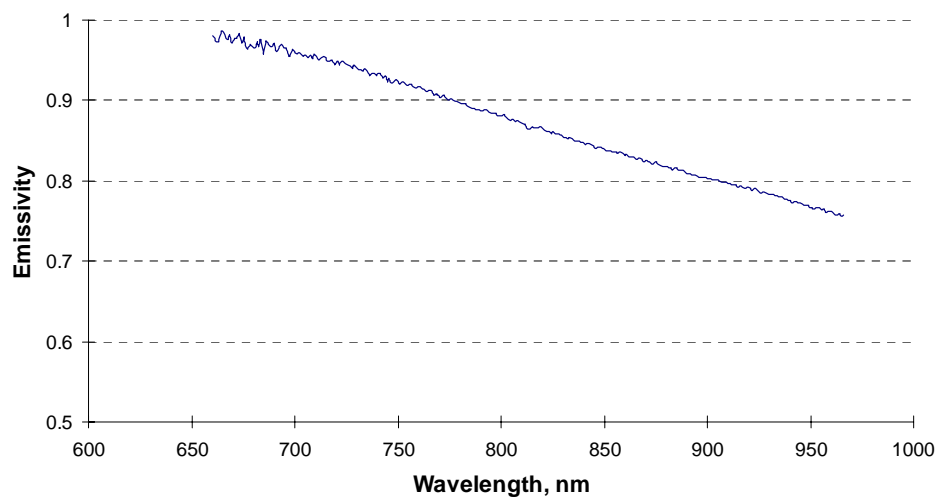


Figure 9. Emissivity of a metal carbide coating at 1175°C; these data were acquired at time = 28.5 seconds of the history recorded in Figure 8.

Atmospheric Absorption and Emission

Substantial gaseous absorptions have been observed in many applications, especially those with graphite as either workpiece or refractory. Figure 10 is a graph of both the spectrum of a carbonaceous workpiece undergoing graphitization and Planck's curve for the detected temperature.

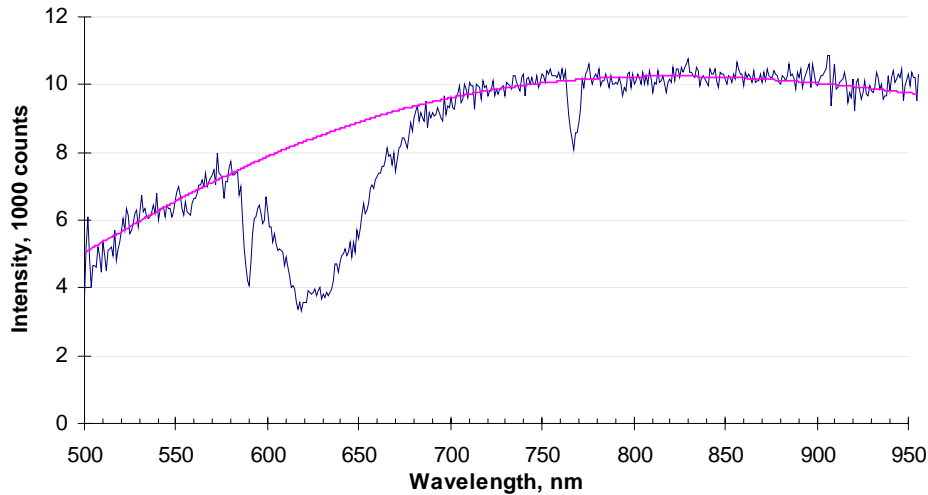


Figure 10. Thermal spectrum with Planck's curve for the measured temperature, 3253°C, superimposed.

Immediately after this measurement, the target area's inert gas purge was increased. Figure 11 shows the corresponding spectra. Note that the "before" and "after" temperatures are within 7°C of each other, with the temperature measured during increased purging being cooler, as expected.

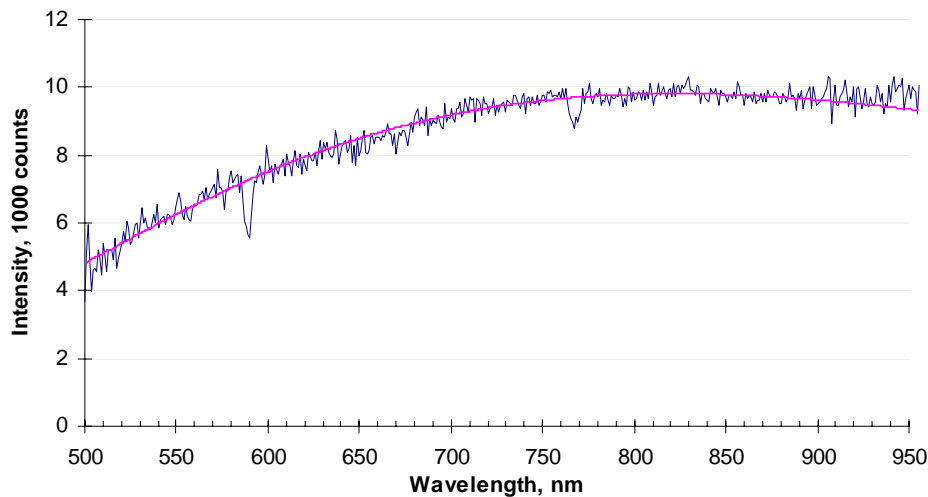


Figure 11. Thermal spectrum of same conditions as Figure 10, but with a greater purge rate; temperature is 3246°C.

Prior to the increased purge, under the conditions of Figure 10, measurements were made with a vanishing filament pyrometer; it returned 2800°C, a difference of 450°C. Obviously, a conventional pyrometer with sensitivity anywhere in the affected area of the spectrum would also have been incorrect.

Emissions have also been detected, particularly in vessels with exposed electrical conductors. Figure 12 shows an absorption/emission spectrum from the hot isostatic pressing of a ceramic material. It was calculated by subtracting the data spectrum from the normalized Planck's law curve for the temperature returned by the Spectropyrometer. The spectral area from 600 to about 740 nm shows the emission, which is attributed to gaseous species glowing due to a parasitic electrical path through the process atmosphere. Increasing process gas flow

while maintaining the same pressure in the vessel reduced the gas's conductivity and flipped the emissions back to absorptions.

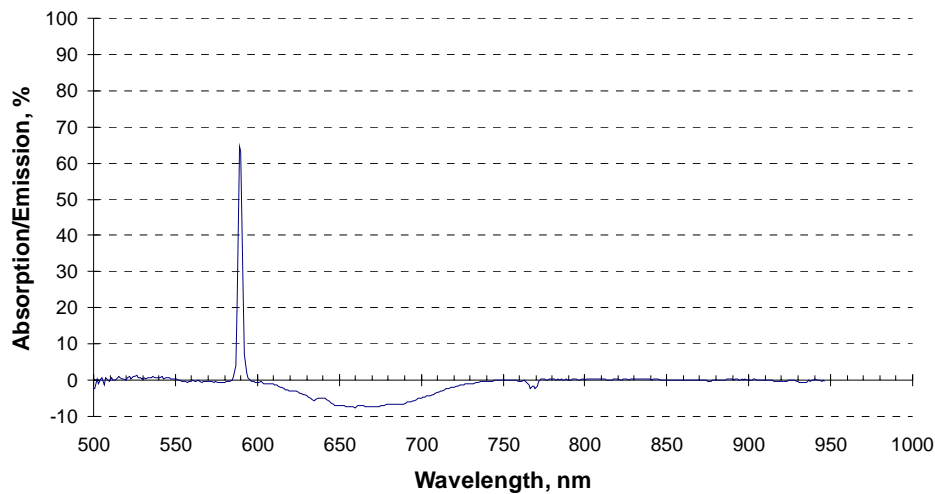


Figure 12. Difference between data spectrum and normalized Planck's law temperature spectrum; negative values indicate emissions.

Graphs such as Figure 12 have been used to identify the species present in the process atmosphere.

Random and Systematic Error

Others (12) have reported 5% uncertainty in temperature with 1% random error (noise) in the thermal spectrum. The Spectropyrometer shows very different results because the entire spectrum contributes to generating the best thermal curve possible. To illustrate, theoretical thermal spectra with 5% and 10% noise added were generated for a wide range of temperatures. These were then processed by the Spectropyrometer as if they were data the instrument had collected. The results given in Table 2 show a maximum difference of less than 0.6% from the true temperature and an average difference of 0.07% and 0.24%, respectively.

TABLE 2. Spectropyrometer results on data with added noise; tolerance defines 100% confidence interval.

Temperature, °C	Output with 5% noise, °C	Output with 10% noise, °C
1000	1001.6 ± 8.1	1001.8 ± 12.7
1400	1400.4 ± 6.6	1402.9 ± 6.7
1800	1799.1 ± 6.7	1801.8 ± 13.3
2200	2201.6 ± 2.8	2194.8 ± 17.2
2600	2600.6 ± 5.6	2607.3 ± 24.2
3000	3004.5 ± 8.4	2980.5 ± 31.6
Average error	0.07%	0.24%

A thermal spectrum from an industrial process, already fairly noisy in appearance, was modified with 10% noise and is shown as Figure 13. The temperature returned from the noisy data differed by 13°C (0.4%) from the original. The tolerance, the standard deviation of the temperature calculation, increased by 13°C, giving an immediate indication of the increased uncertainty.

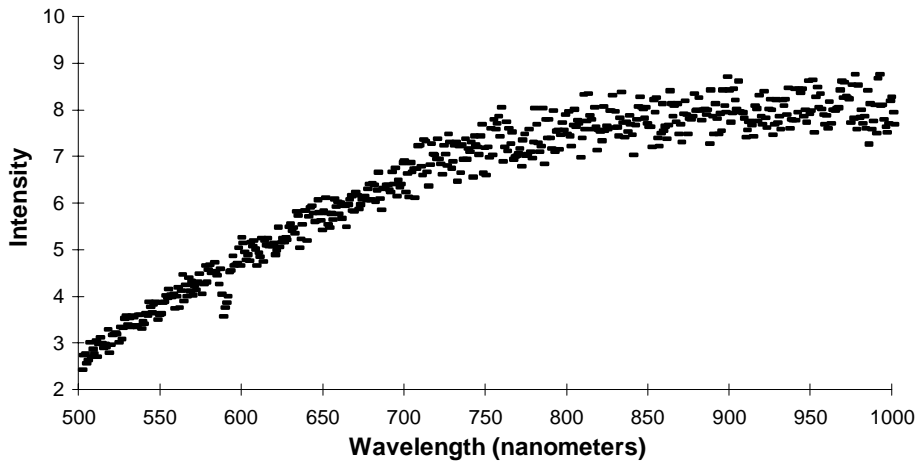


Figure 13. Data collected in an industrial environment with 10% noise added. Before the addition, temperature was returned as $2887.0 \pm 10.3^\circ\text{C}$, after the addition the value was $2899.7 \pm 23.2^\circ\text{C}$. The change in tolerance exactly predicts the increased uncertainty in temperature.

These extreme examples show that noise is not a problem with the Spectropyrometer.

Systematic error is not a concern. If it were limited to pixels scattered through the spectrum it would be eliminated in the same fashion as gaseous absorptions; if it involved the entire spectrum, (e.g., stray light) it would be removed by the algorithm that removes the background (dark current) before the temperature calculation.

CALIBRATION

Calibration of the Spectropyrometer is straightforward, requiring only a single, well-known spectrum from a blackbody or a standard lamp. The known spectrum must be such that all the elements of the detector array are illuminated sufficiently above the background level. Table 3 shows a typical calibration. Average uncertainty is seen to be $\pm 0.043\%$.

TABLE 3. Typical calibration results; source was an Electro Optical Industries BWS156 blackbody.

NIST-traceable standard, $^\circ\text{C}$	Spectropyro-meter, $^\circ\text{C}$	$\Delta T/T$ $\pm\%$ absolute
995.5	996.0	0.020
1198.1	1200.9	0.095
1492.1	1493.5	0.040
1982.6	1983.5	0.020
2184.0	2182.0	0.041

The calibration itself is the same as the intensity calibration for any spectrophotometer: wavelength-by-wavelength division of the known spectrum of the source by the spectrum returned by the instrument generates the correction array. Disagreement between source and instrument calibrated in this manner has regularly been seen to be smaller than that expected from the error expression of ratio pyrometry:

$$\frac{dT}{T} = \frac{T}{c_2} \left(\frac{\lambda_1 \times \lambda_2}{\lambda_1 - \lambda_2} \right) \frac{dR}{R} \quad (2)$$

where T is the absolute temperature, R the ratio, c_2 the second radiation constant, and λ_1 and λ_2 are taken as the extremes of the Spectropyrometer's useful wavelength range. This equation also points out the advantage of a

radiation calibration. The quantity modifying dR/R is substantially less than unity for much of the Spectropyrometer's range; for example, a radiation calibration with a 1% uncertainty corresponds to a temperature calibration with an uncertainty of 0.25% at 2000°C for a silicon instrument.

CONCLUSIONS

An expert system multi-wavelength pyrometer, the Spectropyrometer, has been successful in many industrial processes. These include metal, ceramic, and engineered-materials manufacture and processing. The Spectropyrometer has returned consistent temperatures despite changing emissivity and atmospheric absorptions and emissions. It requires no prior knowledge of the target or the environment. In addition to the temperature, the instrument returns the tolerance, a real-time measure of the temperature determination's accuracy. Further, thermal spectra are stored and can be analyzed for more information, including species present in the process atmosphere. For targets with changing emissivity and/or substantial noise, the maximum uncertainty is typically below 0.75%. Where the temperature is well known and the source well behaved (greybody sources), the Spectropyrometer achieves a maximum uncertainty of less than 0.1%.

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