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A Mid-IR Pyrometer Calibrated with High-Temperature Fixed Points for Improved Scale Realization to 2,500°C

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Abstract Glass surface temperature can be measured using a radiation thermometer operating at a mid-IR wavelength, typically the 3–5 μm band, where the glass is opaque. For optical fiber preforms, the temperature measurement requirement may exceed 2,200°C. Scale realization at national measurement institutes at these temperatures is usually carried out at short wavelengths, typically less than 1 μm. The mismatch in wavelength can lead to significant uncertainties when calibrating a radiation thermometer working at 3–5 μm. To overcome this, a narrow band 3.95 μm radiation thermometer has been built that is designed to be used from 1,000 to 2,500°C. It is calibrated by measurement of high-temperature metal–carbon eutectic fixed-points. The instrument is based on silicon lenses, with a liquid nitrogen (LN_2)-cooled InSb detector, and narrow-band interference filter. An anti-reflection coated objective lens/aperture stop focuses onto a field stop giving a 1 mm target, then a collimating lens, and glare stop. All parts visible to the detector, other than the target area, are either at LN_2 temperature or are part of a temperature-stabilized housing. A relay-operated shutter that blocks the field stop is used to subtract the background. The size-of-source effect of the instrument has been measured. Gold-point measurements have been made to assess the stability. The device has been calibrated using high-temperature fixed points. A three-parameter fit has been applied and the resultant scale compared to an ITS-90 realization.

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1 Introduction

For dissemination to industry of temperatures above 962°C, the National Physical Laboratory (NPL) maintains a reference radiation thermometer (an IKE-LP2) calibrated in terms of the International Temperature Scale of 1990 (ITS-90) [1]. It is calibrated by characterizing the wavelength-defining interference filters, which are nominally 650 and 900 nm, and using a high emissivity gold fixed-point blackbody [2] as the ITS-90 fixed-point reference value. This radiation thermometer is then used to determine the radiance temperature of a high-temperature blackbody cavity (1,000–3,000°C), which is used as a transfer source to calibrate other radiation thermometers. As part of the uncertainty given to the calibration, the difference in wavelength between the calibrated standard and the test instrument is considered. This takes account of differences in radiance temperature as "seen" by the two thermometers due to the non-ideal emissivity of the cavity.

The uncertainty, taken to be type B and to have a rectangular probability distribution, is, at temperature T, given by

$$u(\Delta\lambda) = \frac{(\lambda_1 - \lambda_2)\, T^2}{c_2} \frac{(1 - \varepsilon)}{\sqrt{3}} \tag{1}$$

where c_2 is the second radiation constant, ε is the emissivity, and λ_1 and λ_2 are the wavelengths of the standard and test thermometers. ε is calculated as 0.999 (with $\varepsilon_{\text{graphite}} = 0.85$) under isothermal conditions, but would be expected to vary when there are furnace temperature gradients. For calibrations, we do not assume the calculated value for ε, we take the difference in temperature of the furnace measured at the two wavelengths, 650 and 900 nm, with the standard instrument. Substituting the measured temperature difference for $u(\Delta\lambda)$ in Eq. 1 (without the $\sqrt{3}$ factor) together with the known standard instrument wavelengths gives a worst-case value for the uncertainty arising from the unknown ε. When an instrument being calibrated has a wavelength similar to the standard, $u(\Delta\lambda)$ is a relatively minor part of the overall uncertainty budget. However, when applied to longer wavelength instruments in the 3–5 micron band, this uncertainty component can exceed 10°C at 2,500°C and dominates all other contributions.

As a general principle, the wavelength used for radiation thermometry should be as short as possible for the temperature range being measured. This is because uncertainty components are frequently proportional to λT^2. Most high-temperature measurements are made with instruments working at 650 nm to 1 μm. On occasion, though, the NPL has to calibrate high-temperature, long-wavelength devices, for example, those used in the glass industry where the material being measured is transparent at shorter wavelengths. To improve the uncertainties in the 3–5 micron band, it was decided to acquire a long-wavelength pyrometer with a small target size which could be calibrated with high-emissivity, high-temperature metal–carbon eutectic fixed-points [3] and then be used to measure the radiance temperature of the high-temperature furnace. No suitable commercially available instrument could be found, so it was decided to construct our own, designated the Mk I. This should allow us to perform calibrations of 3–5 micron instruments at high temperatures with greater confidence and much lower uncertainties.

2 Design

At a wavelength of 650 nm, the radiance at 2,500°C is about 10,000 times larger than the radiance at 1,000°C. At a wavelength of 4 µm, however, the radiance is just six times larger. Therefore, the main priority in designing this instrument is stability, to be able to measure small changes in a large signal. Another requirement is a focal distance and target size suitable for calibration with fixed points of small target size operating at high temperature. A focal distance of 800 mm was selected. It was thought that any shorter working distance would place the front of the instrument uncomfortably close to the furnace. The NPL metal–carbon high-temperature fixed points have a radiating cavity 3 mm in diameter [4], and we would normally want a source to be at least three times larger than the nominal field-of-view of a radiation thermometer. A target size of 1 mm was therefore decided on. A narrow band-pass interference filter with a central wavelength of 3.95 µm was chosen, as this is away from most atmospheric absorption bands. A suitable filter with an FWHM of 80 nm was designed and made by Barr Associates. Silicon was selected as the best lens material because of low internal scatter and low temperature dependence of its optical properties. Optical ray tracing software (Zemax©) was used to determine the optimum lens prescription, within the constraints of using the manufacturer's standard tooling. The 50 mm diameter objective and 31.5 mm secondary lenses, both plano-convex, were manufactured by Davin Optronics. The lenses were anti-reflection coated by the manufacturer to have reflectivity $<0.2\%$ at 4 µm. Following a previous design [5], a glare stop was positioned where the secondary lens images the objective. When correctly matched to an aperture stop at the objective, this can greatly improve the size-of-source effect (SSE) of the instrument [6]. A 4 mm diameter indium-antimonide (InSb) detector was chosen as being the best performer in the 3–5 µm band [7]. A liquid nitrogen (LN)-filled dewar was selected as the best way to cool the detector. Although this is somewhat inconvenient to use, no alternative offers better stability. The interference filter and a field-of-view limiting aperture are held at 77 K along with the detector. The Dewar window is broadband (1–6 µm) anti-reflection coated sapphire. A second (uncooled) short pass filter extends the out-of-band blocking to remove humidity sensitivity caused by a long-wavelength water absorption band that otherwise just falls within the detection range of the system. Invar rods were used as the basis of the component mounts due to its low thermal expansion. Figure 1 shows the layout of the components.

At a wavelength of 4 µm, everything at room temperature is a significant source of radiation to the detector. To minimize fluctuations in the background signal, everything seen by the detector is at a stabilized temperature. A siliconized rubber heater mat is wrapped around the lens tube and held at a nominal 30°C, stable to within 30 mK. A similar arrangement is used to stabilize the amplifier (Vincuulum E690). This is made

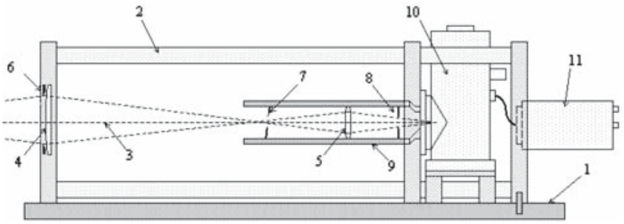

Fig. 1 Layout of the Mk I high-temperature InSb radiation thermometer 1. Anodized Al breadboard 2. Invar rods, 20 mm diameter Optical axis 3. Objective lens, 50.8 mm diameter 4. Focussing lens, 31.5 mm diameter 5. Anodized Al objective stop, 40 mm diameter 6. Anodized Al angled field stop, 400 μm diameter 7. Anodized Al Lyot stop, 5 mm diameter 8. Anodized Al lens tube, temperature controlled 9. Cooled InSb Detector and filter in Dewar (77 K) 10. Transimpedance amplifier, temperature controlled

an integral part of the instrument, to keep connecting wires short. The instrument is fixed to an aluminum breadboard at one place only, to prevent warping by differential expansion. A LabView program controls a relay-operated shutter, which works as a low-speed chopper. Alignment is carried out by having a laser pointer fixed to the breadboard at a known offset to the optical axis. Alignment can be achieved to about 0.3 mm.

3 Characterization

3.1 Warm-up

The temperature control of the lens tube and amplifier takes many hours to stabilize, so these are left permanently on. After filling with LN, it takes approximately 11/2 h for the detector and filter to cool and stabilize to where the background signal becomes constant. The Dewar is rated for 8 h use. If LN_2 is added to extend the time, it takes an hour to stabilize again.

3.1.1 Size-of-Source Effect

Size-of-source effect (SSE) measurements were made using water-cooled apertures in front of a sodium heat-pipe blackbody source. Although thermal radiation from the apertures is still detectable at 4 μm, the contribution to the signal is insignificant compared to the heat-pipe at over 900°C. The measurements showed that the addition of the glare stop significantly improves the SSE of the instrument (Fig. 2). The SSE ratio, with the glare stop, is 0.9926 at 3 mm (the size of the fixed-point radiating cavity) relative to 25 mm (the diameter of the blackbody calibration furnace).

3.1.2 Gold Point

Measurements were made of the NPL gold point [2] in a three-zone furnace. A typical melt and freeze cycle is shown in Fig. 3. The repeatability at 1,064°C was approximately 70 mK ($k = 1$).

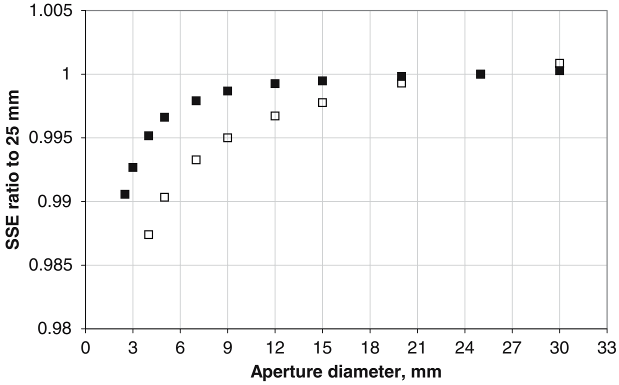

Fig. 2 SSE curves of the Mk I with (■) an without (□) glare stop. SSE is the ratio of the signal at aperture diameter *x* mm to aperture diameter 25 mm

The pyrometer output is stable over the short term. There is no sign of cycling with the room air-conditioning that would indicate humidity sensitivity. The offset between the melt and freeze plateaux could be due to the repeatability of the pyrometer or the size-of-source effect.

4 Calibration

The Mk I radiation thermometer was calibrated using blackbody fixed points of copper (1084.62°C), and metal–carbon eutectic fixed points of cobalt–carbon ($\sim$ 1,324°C), platinum–carbon ($\sim$ 1,738°C), and rhenium–carbon ($\sim$ 2,474°C) [8]. These were used as transfer standards, with the LP2 (at 900 nm wavelength) used to assign melt and freeze temperatures. The fixed points were realized in the Thermogage graphite tube furnace. This has no window but instead relies on a steady purge of argon to stop oxidation of the furnace and its contents. The fixed points have relatively high emissivity

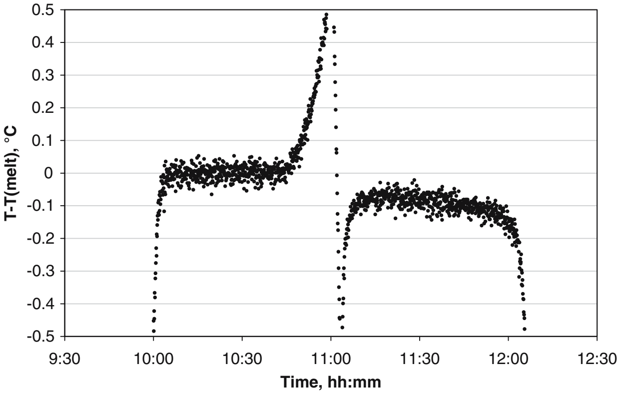

Fig. 3 Typical melt and freeze of the NPL gold-point reference standard with the Mk I

(0.99965 calculated) and the radiating cavity of each is isothermal during the melts and freezes. Therefore, the LP2 standard and the Mk I should see almost the same radiance temperature, despite the difference in wavelength. There is a small difference, which is accounted for in the uncertainty budget. A least-squares algorithm was used to fit the data using a Sakuma-Hattori function [9];

$$V\,(T) = \frac{C}{e^{c_2/AT+B} - 1} \tag{2}$$

where A, B, and C are fit parameters, $V(T)$ is the pyrometer signal at temperature T, and c_2 is the second radiation constant.

The SSE of the LP2 is small and included in the scale realization uncertainty. The radiance distribution in the furnace during fixed-point measurements was measured and combined with the Mk I SSE results to correct the measured fixed-point values to a uniform 25 mm source, the geometry used in the comparison described below. The corrected and uncorrected values were used to fit Eq. 2. The difference in the two resulting scales (less than 0.5°C from 1,000 to 2,500°C) was included in the uncertainty budget. The corrected fit parameters were used as the basis of the scale comparison.

In future, it is expected that the M–C fixed-point values will be specified and there will be no need to use a calibrated pyrometer to assign a melt temperature. In that case, it is likely that only the melt temperature will be defined. For the measurements here, the fixed points were used as transfer devices so both the melt and freeze were used, and averaged to provide a reference value.

5 Comparison

5.1 Measurement

The Mk I was compared to the LP2 reference standard, at 900 nm wavelength, from 1,000 to 2,500°C in 250°C steps with two repeat points, using the NPL Thermogage blackbody calibration source. This has been slightly modified from standard; the furnace tube length has been shortened to reduce the chance of clipping a pyrometer's field-of-view, and additional insulation is used above what the manufacturer specifies. This reduces temperature gradients (as determined by scanning across the back-wall of the cavity) but makes the response time of the furnace slower.

Each instrument was aligned on the center of the back-wall of the radiating cavity. Checks were made that the instruments were not clipping the furnace by scanning horizontally and vertically across the front of the source. At each temperature, the LP2 was used to check that the furnace was stable before measurements were made—typically 15 min was needed. At each calibration temperature, the Mk I was displaced by 1 mm to either side, and the radiance change noted, to allow for any mismatch between the LP2 and Mk I alignment. At 2,250°C, the Mk I was left exposed to the radiating cavity for 30 min (after the furnace had stabilized) to determine any drifting occurrence as the optics was heated, a problem found with some high temperature radiation thermometers. No effect on the output was discernable. Two sets of readings were made at each calibration point, separated by about a 2-min interval. The inverse of Eq. 2 was used to determine the temperature using the fit parameters from the calibration.

5.2 Uncertainties

The following uncertainties were included: the scale realization of the LP2, the size-of-source effect, the alignment of the Mk I on the back wall of the radiating cavity, the repeatability of the measurements, the stability of the furnace, and the residuals of the fit to Eq. 2.

6 Discussion

Figure 4 shows that Mk I gives a low radiance temperature compared to LP2 when used with the Thermogage as a calibration device. The non-isothermal conditions seem to have a significant impact on the emissivity. The effective emissivity may be lower than calculated because of temperature gradients, or may be different at different wavelengths. The gradients may change, giving rise to a temperature dependence. The data match within the uncertainties what would be expected for an effective emissivity of about 0.995. It looks as if concerns about calibrating long-wavelength instruments with a short-wavelength standard are justified. These results will allow a much better assessment of uncertainties when calibrating long-wavelength devices. From Eq. 1, the uncertainty contribution from the mismatch of wavelengths will be about 1.5°C at 2,500°C for a 5 μm instrument and an effective emissivity of 0.995, an improvement of a factor of about four.

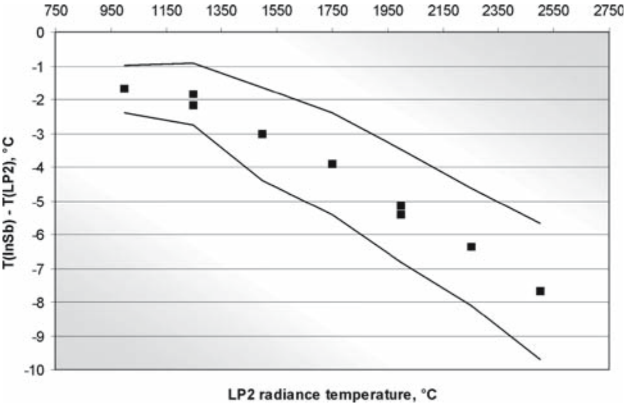

Fig. 4 Difference in measured temperatures of the Thermogage furnace as seen by the LP2 and Mk I InSb (☐). The $k = 2$ uncertainties are shown (—)

Before Mk I can be used as part of an accredited calibration service, the long-term stability will need to be evaluated. Until then, the results here can be used to apply a realistic (if large) uncertainty when calibrating with the short-wavelength standard.

7 Conclusion

A 3.95-μm narrow-band InSb radiation thermometer, with good SSE, has been constructed and calibrated using high-temperature metal–carbon eutectic fixed points. This instrument will allow improved calibration of high-temperature mid-IR pyrometers.

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