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High-Speed Infrared Radiation Thermometry for Microscale Thermophysical Property Measurements[1]

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A new infrared radiation thermometer having a high temporal response and a high spatial resolution is being developed at NMIJ to meet the existing demand for measurements of thermophysical properties of thin films, coatings, and solids in microscale. The thermometer consists of a photovoltaic (pv)-type of mercury cadmium telluride (MCT) detector and a compact Cassegrain type of mirror optics without a mechanical chopper. The performance of the thermometer has been well characterized experimentally. Sensing infrared radiation around 10 μm of wavelength, the thermometer covers the temperature range from −50 to 150°C and has a temperature resolution better than 0.3°C at −50°C for blackbody radiators. The spatial resolution has also been checked by using a test pattern (USAF 1951) for rating the resolution of optical systems. Temperature changes of specimen surfaces in periodic heating with a laser beam modulated above 100 kHz have been observed successfully with the thermometer. The results shows that the thermometer has great potential for measuring the thermal diffusivity, thermal conductivity, and specific heat capacity of microscale substances at low temperatures based on the periodic heating and pulsed laser heating methods.

KEY WORDS: high-speed infrared thermometer; low temperature; periodic heating; thin film.

1. INTRODUCTION

Recent developments in materials science require the measurement of the thermophysical properties of microscale substances, e.g, thin films and

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coatings, with high resolution and speed. Laser-flash methods and periodic heating methods with laser beams have been actively investigated for measuring the thermal diffusivity, thermal conductivity, and specific heat capacity of microscale substances [1]. In these methods, it is essential to sense radiation temperature change of the surface of a specimen heated by a laser beam. In the pulsed heating method, the surface temperature of a specimen increases rapidly after the specimen has been irradiated by a pulsed laser beam. On the other hand, in the periodic heating method, the surface temperature changes sinusoidally with time in response to the modulated incident laser beam. As the dimensions of a target decrease, the response time of the thermal process decreases rapidly and reaches microsecond levels for substances of the order of micrometers. For these measurements, it is quite effective to apply optical thermometric techniques instead of conventional contact methods using thermocouples or resistance thermometers.

Infrared radiation thermometry is a powerful approach for the measurement of thermophysical properties. It can be applied to any sample, which is opaque at the observed wavelength. No external light source is needed for temperature measurement. It is also possible to measure absolute values of the temperature by using the radiation thermometer calibrated against a blackbody radiator in case the radiant property, i.e. emissivity, of the sample surface is known. On the other hand, it has two major drawbacks in practice. The lower limit of the temperature range and the upper limit of the response speed should be extended to meet the demand of advanced thermophysical property measurements.

Recently, the authors have been developing instrumentation and measurement techniques of infrared radiation thermometry for thermophysical property measurements [2, 3]. The infrared radiation thermometer was applied successfully to the pulsed laser-flash measurement. The temperature change of samples above room temperature in the frequency range from dc up to 10 kHz has been measured. The contribution of the nonlinear temperature dependence of the blackbody radiance near room temperature was also investigated by using the InSb radiation thermometer calibrated against the blackbody radiator [2]. More recently we developed a multi-element InSb radiation thermometer measuring simultaneously two individual specimens; the thermal diffusivity and heat capacity or thermal conductivity of solids can be estimated at a given time based on the pulsed laser-flash differential scanning calorimetric approach [3]. In these thermometers the cryogenic InSb detectors covered the wavelength range up to 5 μm and the frequency region from dc up to 10 kHz.

On the other hand, to extend the temperature range below 0°C, it is essential to measure thermal infrared radiation at a longer wavelength.

In the present study, we have constructed two different types of thermal infrared radiation thermometers for the measurement of the thermophysical properties of microscale substances [4]. In this paper, the design and characterization of a compact type of high-speed thermometer is presented. The thermometer consists of a cryogenic mercury cadmium telluride (MCT) photovoltaic detector with maximum spectral response around 10 μm in the wavelength of radiation and a compact mirror objective of a Cassegrain type. The thermometer is operated in a dc mode without a mechanical chopper to realize a high-speed response. Instead of a mechanical chopper, a cold radiation shield was installed in front of the MCT detector to suppress the background thermal radiation from surroundings. The thermometer performance is characterized by a temperature range from −50°C to above 150°C, a response speed above 100 kHz, and a spatial resolution of about 100 μm. It has been successfully used to measure temperature changes of specimens heated periodically by a laser beam modulated at 100 kHz.

2. DESIGN OF THE RADIATION THERMOMETER

A cross-sectional view of the infrared radiation thermometer developed in this study is illustrated in Fig. 1. In the present study, we intend to develop a new thermometer having the following features required for advanced thermophysical property measurements:

- Low temperature range down to −50°C,
- Wide range of temporal response from dc up to 1 MHz,
- High temperature resolution; <0.1°C at 0°C and 0.5°C at −50°C,
- High spatial resolution <1 mm, and
- Compact and simple operation.

In our previous studies [2, 3], we applied InSb detectors to infrared radiation thermometers. The InSb detectors exhibited high performance, though they could cover a wavelength range only below 5.5 μm. To extend the temperature range of the radiation thermometer below 0°C, it is essential to observe longer wavelengths around a 10 μm band, for which the Planck curve attains its maximum value near ambient temperature. Un-cooled thermal detectors, thermopiles, and pyroelectrics have been commonly used for thermal infrared radiation thermometers in industry because of the ease and economy of construction, while cryogenic semiconductor infrared detectors, such as an MCT, have great potential to enhance the performance of radiation thermometers for high temperature

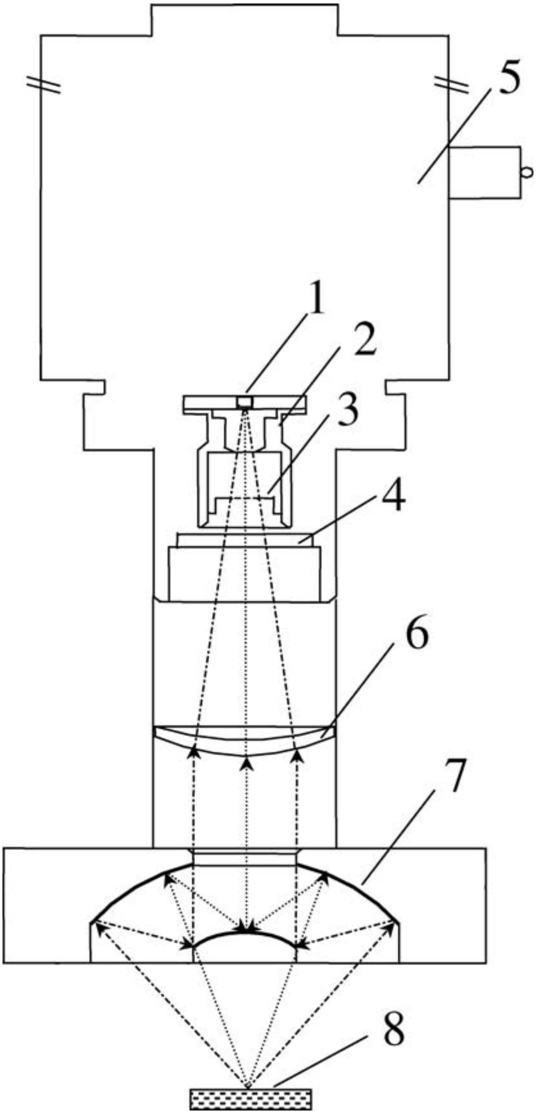

Fig. 1. Optical arrangement of the MCT radiation thermometer. 1: MCT detector, 2: cold radiation shield, 3: aperture, 4: window, 5: liquid nitrogen Dewar, 6: ZnSe lens, 7: objective Cassegrain mirror, 8: sample.

resolution and fast response. Photoconductive (pc-) MCT detectors have been used for thermal infrared instruments. However, a pc-MCT detector has a high 1/f noise and a poor linearity of response, which decrease the performance of the radiation thermometer in the dc-operation mode without a chopper. In this study, we use a photovoltaic MCT detector, which has a low 1/f noise, good linearity, and a high response speed.

In the infrared spectral range beyond 3 μm, the effect of thermal emission from the housing and optical components of the infrared instruments, the so-called thermal background radiation, is significant. It can cause a considerable increase in detector noise and drift of the output offset with a variation in the ambient temperature. Most of the conventional thermal infrared radiation thermometers have a mechanical chopping system to modulate an incident beam. The mechanical chopper is usually used as a reference surface to compensate for the background radiation change caused by the ambient temperature changes. However, the use of a mechanical chopper restricts the response speed of radiation thermometers. To realize a wide range of temporal response from dc up to 1 MHz, it is necessary to operate the infrared thermometer in a dc mode without a mechanical chopper. To achieve high sensitivity, good short-term stability, and high response speed at low temperatures, a liquid-nitrogen-cooled photovoltaic (pv) MCT detector of 0.5 mm in diameter (Fermionics) with a specially designed radiation shield is used. The spectral response of the pv-MCT detector has a maximum in the wavelength from 8 to 10 μm. The detector is installed in a bottom-view type of vacuum Dewar. The radiation shield [5] is made of copper and treated with a diffusive low reflectance black coating. The inner wall of the radiation shield is grooved to reduce inter-reflection of stray light. Inside the radiation shield, a small aperture is inserted. A full angle of view of about 15° is defined for the detector by a cold aperture. The radiation shield is fixed to a cold metal base plate with the MCT detector element and cooled by liquid nitrogen to just above its boiling point of −196°C in the vacuum Dewar. An anti-reflection-coated silicon window is used on the Dewar to prevent visible light from causing an increase in the dark current of the MCT detector.

A fraction of the radiation emitted by a sample surface under investigation is collected by a gold-coated Cassegrain type mirror with a focal length of 25 mm, which is attached to the incident port of the detector. The effective diameters of the primary and secondary mirrors are 48 and 16 mm, respectively. A simple mirror optics is adopted to provide a compact and robust instrument. The collected radiation is directed onto the detector through an anti-reflection-coated ZnSe lens of 63.5 mm in focal length and 28 mm in diameter. For focusing, the thermometer is mounted on a translation stage in the vertical direction. The photocurrent signal of the MCT detector operated in a zero-bias current mode was fed into a homemade current–voltage (I–V) converter circuit, which consists of a high-speed operational amplifier and a feedback resistor of 1 MΩ. The voltage signal ranges from 0 to 5 V and is measured with a digital voltmeter, an oscilloscope, or a lock-in amplifier.

3. PERFORMANCE TESTS

Several tests were performed to check characteristics of the infrared thermometer.

3.1. Calibration

The results of the calibration against a blackbody in the temperature range between 0 and 50°C are shown in Fig. 2. A cylindrical cone blackbody cavity is fully immersed vertically in the temperature-controlled stirred fluid bath. The size of the opening aperture of the cavity is 20 mm in diameter, and the calculated apparent emissivity of the cavity is higher than 0.999. The temperature of the blackbody cavity was measured with a calibrated platinum resistance thermometer. The radiation thermometer was focused on the entrance aperture of the blackbody cavity. The signal output levels were 1.45 and 2.2 V for a blackbody of 0 and 50°C, respectively. Additionally, we also measured the signal output of the thermometer for liquid nitrogen in the vacuum flask. The signal output was about 1 V, which corresponded to the output offset of the thermometer.

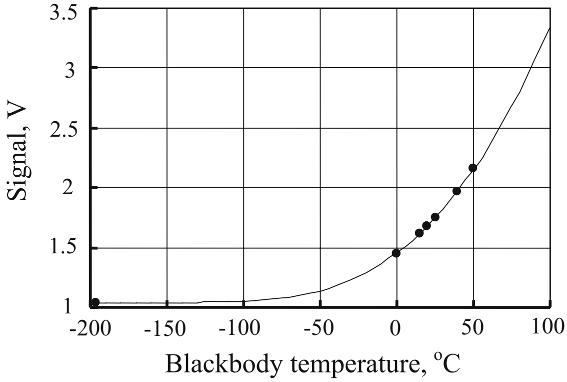

Fig. 2. Calibration curve of the radiation thermometer.

The solid curve in Fig. 2 represents a reference function fitted to correlate the output to the target temperature in a range from 0 to 50°C based on Planck's equation,

$$V = \frac{c_1}{a\left[\exp\left(\frac{c_2}{bT}\right) - 1\right]} + d, \tag{1}$$

where V is the output signal, T is the absolute temperature, c_1 and c_2 are the first and second radiation constants, and a, b, and d are parameters determined by least-squares fitting. a, b, and d correspond to gain, effective wavelength, and output offset of the thermometer, respectively. It is seen from this figure that the output of the thermometer agrees well with theory. The estimated value of b is 8.3 μm, which is expected for the MCT detector, and the value of the d parameter is 1.04 V. Consequently, a temperature range from −50 up to 150°C can be covered by the thermometer. The temperature resolution expressed in terms of the noise-equivalent temperature difference (NETD) is evaluated to be better than 0.3°C for a target temperature of −50°C and better than 0.1°C for temperatures above 0°C, which were satisfactory for our intended thermophysical property measurements.

3.2. Time Response

The time response is one of the most important characteristics of the thermometer for measuring the thermophysical properties in microscale. First, we evaluated the time response of the detector unit, which consisted of the MCT detector and the I–V converter. In this measurement, a visible laser diode ($\lambda = 635$ nm, 0.9 mW) operated in the current modulation mode was used as a source. Although the spectral responsivity of the MCT detector gradually decreases at wavelengths less than 10 μm, the detector still responds to a visible beam. In addition, it is assumed that the time response of the MCT detector is independent of the wavelength of the incident radiation because of a quantum detector. The modulated visible laser beam was split into two beams, one of which was directed onto the MCT detector in the vacuum Dewar with a ZnSe window, which transmitted the visible beam. The other beam was fed into a high-speed silicon *p-i-n* photodiode detector. By changing the modulation frequency of the laser diode from dc to 5 MHz, the signal output of the detector unit was compared with one of the fast silicon detector. The results of the measurements indicate that the MCT detector operated with the I–V converter circuit responds to the modulated beam in the frequency range from dc up to 3 MHz.

Second, we measured the response of the radiation thermometer to the modulated thermal emission under the same conditions as those for the periodic heating measurement. In this experiment, the window of the vacuum Dewar of the MCT detector was replaced by a silicon window, which was opaque in the visible region, and the objective optics was attached to the detector unit. The specimen was placed at the focal plane of the Cassegrain mirror objective and heated by the modulated laser beam, which was irradiated on the front surface of the specimen through an optical fiber of 0.1 mm in diameter.

Figure 3 shows a result for a single crystal silicon specimen of 1 mm in thickness. The surface of the silicon specimen was blackened to increase the emissivity around $\lambda = 10\,\mu\mathrm{m}$. The surface was heated by the incident laser beam, $\lambda = 830\,\mathrm{nm}$ and mean power = 250 mW, which was modulated at 100 kHz. The signal output of the radiation thermometer was fed into an oscilloscope. The lower trace in Fig. 3 represents the modulation signal of the heating laser, and the upper trace represents the signal of the infrared radiation thermometer. This result indicates the radiation thermometer can be used to measure the temperature change in the sub-microsecond region. This demonstrates a potential application of the thermometer to not only periodic thermal phenomena but also transient phenomena in a sub-microsecond region.

3.3. Spatial Resolution

The spatial resolution is also an important characteristic of radiation thermometers for measuring temperatures of microscale surfaces or mapping anisotropic materials. For conventional radiation thermometers, the variable aperture methods with a large area blackbody cavity are used for characterization of the effective field-of-view [6]. In this study we constructed a miniature apparatus for evaluation of the effective field-of-view of the thermometer. A vertical blackbody cavity with an aperture of 10 mm diameter was used as a infrared radiation source. In front of the aperture, movable plates of various pinhole sizes were positioned automatically at the focus position to vary the effective size of the source between 0.1 and 5 mm in diameter. The results show that the effective field-of-view of the thermometer is about 0.3 mm in diameter, which is sufficient for our intended use.

We also used a standard pattern of “USAF 1951 1X” for testing the optical resolving power in order to characterize the effective spatial resolution of the radiation thermometer in scanning operations. Figure 4 represents the picture of the USAF 1951 1X pattern. The test pattern was

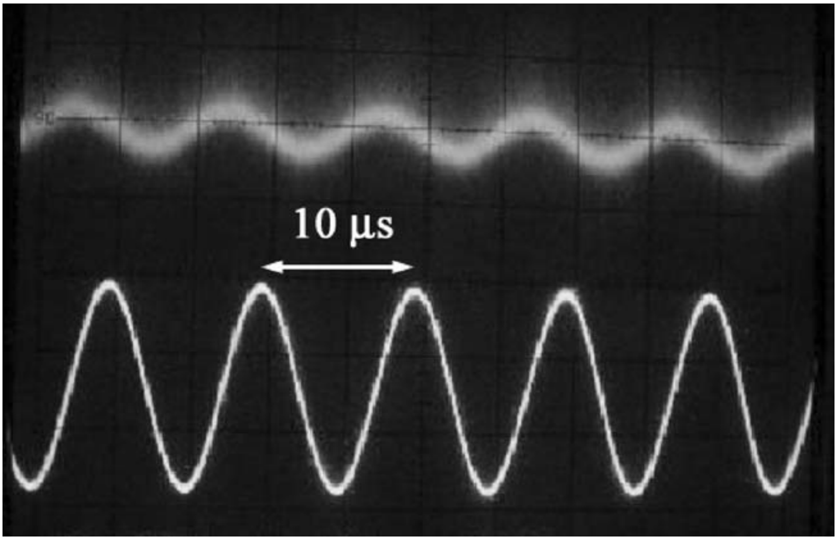

Fig. 3. Thermal radiation signal of Si crystal heated periodically by the laser beam ($\lambda =$ 830 nm) modulated at 100 kHz. Upper trace is signal output of the radiation thermometer; lower trace is modulation signal of the heating laser beam.

placed on the focal plane of the thermometer at room temperature and scanned by using a two-dimensional translation stage.

Figure 5 shows output signals in measuring across a pattern of 2G-2L, second lines on a second group of chart, on the USAF 1951 1X, which is lying in a uniform array at a regular center interval of 223 μm. This result indicates that the radiation thermometer can distinguish clearly the adjacent patterns. In Fig. 5, the signal of the thermometer is weak for the background and increases at the positions of the pattern, in which the signal approaches that of blackbody radiation at room temperature. The test pattern is a negative one, in which the pattern on a quartz glass substrate is formed on a background deposited with chromium. The chromium background has an optical property of highly specular reflection. In such a case, the detector operated in the cryogenic Dewar observes its own cold image reflected on the specularly reflective surface of the chromimu background. On the other hand, the detector senses blackbody radiation at near ambient temperature from the pattern of the quartz glass substrate having a high emissivity in the thermal infrared region.

Figure 6 illustrates the contour map for the test pattern, which was measured by the infrared radiation thermometer in two-dimensional scanning. On the left side, signal maps for the shape of figures representing the

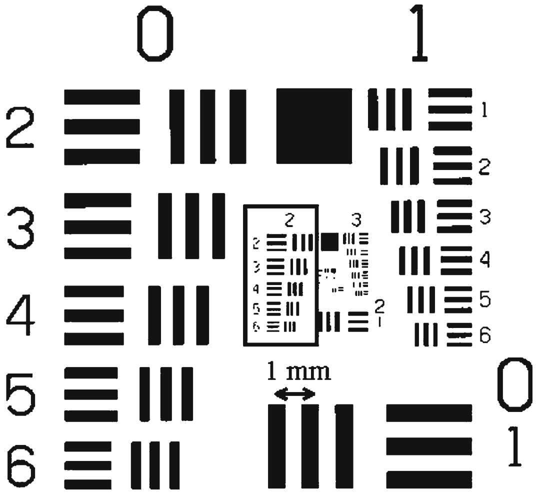

Fig. 4. Image of USAF 1951 1X chart: 2G-2L pattern is inside boxed line.

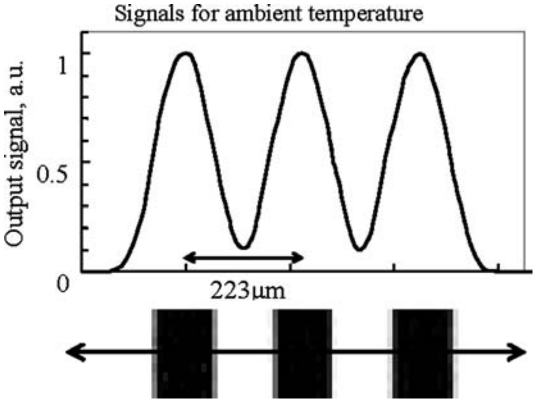

Fig. 5. Radiation signal in measuring the 2G-2L pattern (4.49 pair/mm) on the test chart, USAF 1951 1X.

line numbers of the pattern can be found. On the lowest line, 5L, the patterns lying in an array at a regular center interval of 157 µm can be clearly distinguished. From these results, it is concluded that the radiation thermometer allows us to perform scanning of thermal targets with a spatial resolution of the order of 100 µm.

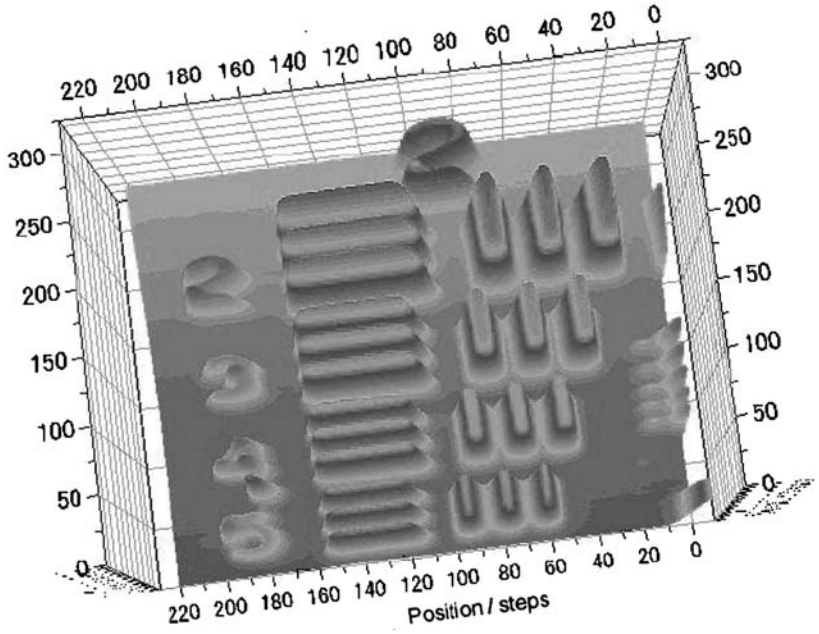

Fig. 6. Contour map of radiation signal on a part of 2G chart of the USAF 1951 1X: line pitch of patterns: 223 μm for 2L, 198 μm for 3L, 177 μm for 4L, and 157 μm for 5L pattern.

4. CONCLUSIONS

We have developed a thermal infrared radiation thermometer for measuring the thermophysical properties of microscale substances at low temperatures based on periodic heating and pulsed heating methods. The thermometer, which consists of a photovoltaic MCT detector with a maximum spectral response around 10 μm and simple mirror optics, is a compact and robust instrument. Temperatures down to −50°C can be measured with 0.3°C temperature resolution. The temperature change of the sample surface heated by the laser beam modulated at 100 kHz was monitored by the thermometer. The effective spatial resolution of the thermometer in the scanning mode was around 100 μm, which was sufficient to investigate anisotropic materials, coatings, and films.

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