HIGH-SPEED INFRARED RADIATION THERMOMETRY FOR MICRO-SCALE THERMOPHYSICAL PROPERTY MEASUREMENTS[1]

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Abstract
A new infrared radiation thermometer having a high temporal response and a high spatial resolution is being developed at the NMIJ to meet the existing demand for measurements of thermophysical properties of thin films, coatings and solids in micro-scale. 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 performances of the thermometer have been well characterized experimentally. Sensing thermal infrared radiation around 10 μm, the thermometer covers the temperature range from –50 ^{o}C up to 150 ^{o}C and has temperature resolution better than 0.3 ^{o}C at –50 ^{o}C. The high spatial resolution has also been checked by using a test pattern (USAF 1951) for rating resolution of optical system. 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 a great potential for measuring thermal diffusivity, thermal conductivity and specific heat capacity of micro-scale substances at low temperatures based on the periodic heating methods and the pulsed laser heating methods.

KEY WORDS: high-speed, infrared thermometer, low temperature, MCT

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INTRODUCTION

Recent developments in material science require the measurement of the thermophysical properties of micro-scale substances, e.g. thin films and coatings, with high accuracy and speed. Laser flash methods and periodic heating methods with laser beams have been investigated actively for measuring thermal diffusivity, thermal conductivity and specific heat capacity of the micro-scale substances[1]. In these methods, it is essential to sense surface temperature change of a specimen heated by a laser beam. In the pulsed heating method, surface temperature of a specimen increases rapidly after the specimen has been irradiated by 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 down to microsecond region for micro-scale substances. 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 right 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. On the other hand, it has two major drawbacks in practice. The lower limit of the temperature range and the upper limit of speed of the response should be extended to meet the demand of advanced thermophysical property measurements.

Recently, the authors have been developing instrumentation and measurement techniques of the infrared radiation thermometry for the thermophysical property measurement[2, 3]. The mid-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 non-linear 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 constructed a multi-element InSb radiation thermometer measuring simultaneously two individual specimens, thermal diffusivity and heat capacity or thermal conductivity of solids can be estimated at a time based on the pulsed laser flash differential scanning calorimetric approach[3]. In these thermometers, which were constructed previously, 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 down to sub-zero level it is essential to measure thermal infrared radiation at longer wavelength. In the present studies, we have constructed two different types of thermal infrared radiation thermometer for the measurement of the physical properties of the micro-scale substances[4]. In this paper, design and characterization of a compact type of the high-speed thermometer is presented. The thermometer consists of a cryogenic MCT photovoltaic detector with maximum spectral response around 10 μm and a compact mirror objective of the Cassegrian type. The thermometer is operated in a DC-mode without a mechanical chopper to realize a high-speed of 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. Performances of the thermometer attains the a temperature range from –50 ^{o}C to above 150 ^{o}C, a speed of response above 100 kHz, and a spatial resolution much better than 1 mm. It has been successfully used to measure temperature change of specimens heated periodically by the laser beam modulated at 100 kHz.

DESIGN OF THE RADIATION THERMOMETER

The cross-sectional view of the infrared radiation thermometer developed in this study is illustrated in Figure 1. In the present study, we intended to develop a new thermometer having the following several features required for the advanced thermomphysical property measurements:

- *Low temperature range down to –50 ^{o}C,*
- *Wide range of temporal response form DC up to 1 MHz*,
- *High temperature resolution < 0.1 ^{o}C at 0 ^{o}C and 0.5^{o}C at –50 ^{o}C,*
- *High spatial resolution < 1 mm* ,
- *Compact and simple to operation*.

In our previous studies, we applied the InSb detectors to our infrared radiation thermometers. The InSb detectors exhibit high performance, though they can cover the wavelength range only below 5.5 μm. To extend the temperature range of the radiation thermometer below 0 ^{o}C, it is essential to observe longer wavelengths around 10 μm band,

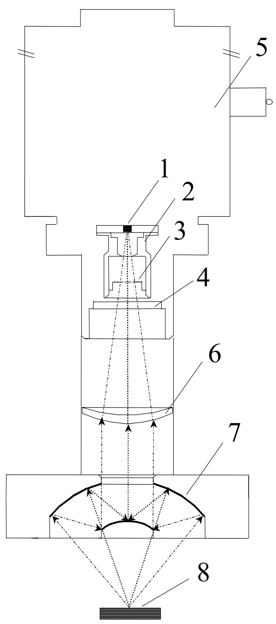

Figure 1. Optical arrangement of the MCT radiation thermometer. 1: MCT detector, 2: cold radiation shield, 3: aperture, 4: window, 5: liquid nitrogen dewar, 6: ZnSe lens, 7: objective Cassegrian mirror, 8: specimen

for which the Planck curve attains its maximum value near ambient temperature. Uncooled thermal detectors, thermopile and pyroelectrics, have been commonly used for thermal infrared radiation thermometers in industry because of ease and economy of construction, while cryogenic semiconductor infrared detectors, MCT, have a great potential to enhance the performance of the radiation thermometer in high temperature resolution, and fast responsivity. Photoconductive (pc-) MCT detectors have been used for the thermal infrared instruments. However, pc-MCT detector has a high 1/f noise and a poor linearity of response, which decreases the performance of the radiation thermometer in the DC-operation mode without a chopper. In this study, we used a photovoltaic MCT detector, which had a low 1/f-noise, and a good linearity and a high speed of the response.

In the infrared spectral range beyond 3 μm, the effect of the thermal emission from the housing and optical components of the infrared instruments, so-called thermal background radiation, is significant. It can cause a considerable increase in detector noise and drift of the output offset with varying 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 use of a mechanical chopper restricts speed of response of the radiation thermometer. To realize the wide range of temporal response from DC up to 1 MHz, it is necessary to operate the infrared thermometer in DC-mode without a mechanical chopper. To achieve high sensitivity, good short-term stability and high speed of response at the low temperature, a liquid nitrogen cooled photovoltaic (pv-) MCT detector of 0.5 mm diameter (Fermionics) with a specially designed radiation shield was used. The spectral response of the pv-MCT detector has a maximum in the wavelength from 8 μm to 10 μm. The detector was installed in a bottom view type of vacuum dewar. The radiation shield[5] was made of copper and treated with a diffusive low reflectance black coating. The inner wall of the radiation shield was grooved to reduce inter-reflection of stray light. Inside the radiation shield, a small aperture was inserted. A full angle of view of about 15 degree was defined for the detector by the cold aperture. The radiation shield was fixed to the same cold metal base-plate as used with the MCT detector element and cooled down to –196 ^{o}C in a vacuum dewar. An antireflection-coated silicon window was used on the vacuum dewar to prevent visible light from reaching the MCT detector, and causing the dark current to increase.

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 diameter of the primary and secondary mirrors are 48 mm and 16 mm, respectively. The simple mirror optics were adopted to get a compact and robust instrument. The collected radiation is directed onto the detector through an antireflection-coated ZnSe lens of 63.5 mm in focal length and 28 mm in diameter. For focusing, the thermometer was mounted on a translation stage in vertical direction. The photocurrent signal of the MCT detector operated in a zero-bias current mode was led into to a home-made I-V converter circuit, which consists of a high-speed operational amplifier and a feedback resistor of 1MΩ. The voltage signal is a range from 0 V to 5 V and measured with a DVM, oscilloscope or lock-in-amplifier.

PERFORMANCE TEST

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

Calibration

The results of the calibration against a blackbody in the temperature range between 0 °C and 50 °C are shown in Figure 2. The cylindrical cone blackbody cavity is fully immersed vertically in the temperature-controlled stirred fluid bath. The size of opening aperture of the cavity is 20 mm diameter and the calculated effective emissivity of the cavity is higher than 0.999. Temperature of the blackbody-cavity was measured by a calibrated platinum resistance thermometer. The radiation thermometer was focused on the entrance aperture of the blackbody cavity. The signal output level was 1.45 V and 2.2 V for a blackbody of 0 °C 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 1V, which corresponded to the output offset of the thermometer.

The solid curve in Figure 2 represents a fitted curve to relate the output to the target temperature in the range from 0 °C to 50 °C based on the Planck's equation,

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

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 the least-square fitting. It is seen from this figure that the output of the thermometer agrees well with theory. The estimated value of b was 8.3 μm, which was expected for the MCT detector and the value of d parameter was 1.04 V, which agreed well with the signal output for the liquid nitrogen. Consequently, the temperature range from –50 °C up to 150 °C can be covered by the thermometer. The temperature resolution expressed in terms of the noise-equivalent temperature differences (NEDT) were evaluated 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 use of thermophysical property measurements.

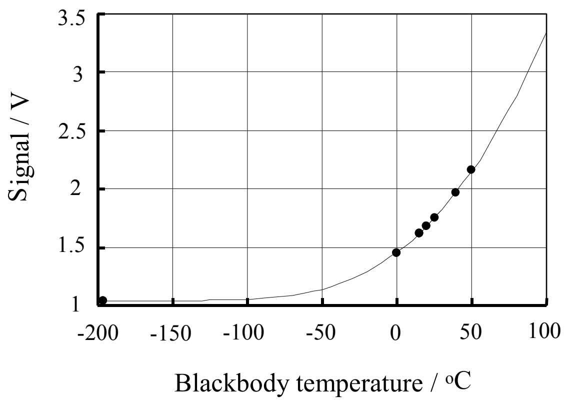

Figure 2. Calibration curve of the radiation thermometer

Time response

Time response is one of the most important characteristics of the thermometer for measuring the thermophysical properties in micro-scale. Firstly, 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 (λ = 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 with shorter wavelength than 10 μm, the detector still responds, though slightly, to visible beam. In addition it was assumed that time response of the MCT detector is independent on the wavelength of the incident radiation. The modulated visible laser beam was split into two beams, one of which was directed onto the MCT detector in the vacuum dewar with ZnSe window, which transmitted the visible beam. The other beam was led 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 measurement indicate that the MCT detector operated with the I-V converter circuit respond to the modulated beam in the frequency range from DC up to 3 MHz.

Secondly, we measured the response of the radiation thermometer to the modulated thermal emission under the same conditions as for the periodical heating measurement. In this experiment, the window of the vacuum dewar of the MCT detector was replaced by the 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 Cassegrian mirror objective and heated by the modulated laser beam, which entered from the front side of the specimen though an optical fiber of 0.1 mm in diameter.

Figure 3 shows a result for a single crystal silicon specimen of 1 mm thickness. The surface of the silicon specimen was blackened to increase the emissivity around 10 μm. The surface was heated directly by the laser beam, λ = 830 nm, mean power = 250 mW, which was modulated at 100 kHz. The signal output of the radiation thermometer was fed

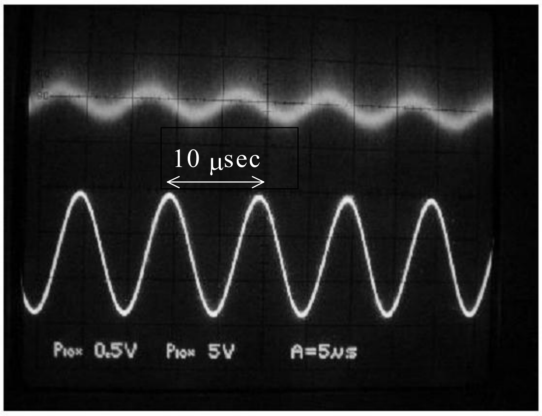

Figure 3. Thermal radiation signal of Si crystal heated periodically by the laser beam (λ = 830 nm) modulated at 100 kHz. upper trace is signal output of the radiation thermometer, lower trace is modulation signal of the laser beam

directly into an oscilloscope. The lower trace in figure 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 enables us to measure the temperature change in the sub-microsecond region without a phase sensitive detection. This demonstrates a potential application of the thermometer to not only periodic thermal phenomena but also transient phenomena in sub-microsecond region. In this study, we also measured the similar modulation signals for thin specimens of glassy-carbon, Pyrex-glass, aluminum, and phosphor bronze.

Spatial resolution

The spatial resolution is also an important characteristic of radiation thermometers aimed at measuring micro-scale samples or mapping anisotropic materials. For conventional radiation thermometers, the variable aperture method with a large area blackbody is used for characterization of the effective target-size[6]. In this study we constructed a miniature apparatus for evaluation of the effective target-size of the thermometer. A vertical blackbody-cavity with an aperture of 10 mm diameter was used as a 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 mm and 5 mm in diameter. The results show that the effective target-size of the thermometer is about 0.3 mm in diameter, which is enough to our intended use.

Subsequently, we used a standard pattern of "USAF 1951 1X" to test the resolving power optical system in order to characterize the effective spatial resolution of the thermometer in scanning operations. Figure 4 represents the picture of the USAF 1951 1X pattern. The test pattern was placed on the focal plane of the thermometer at a room temperature and scanned by using a two-dimensional translation stage.

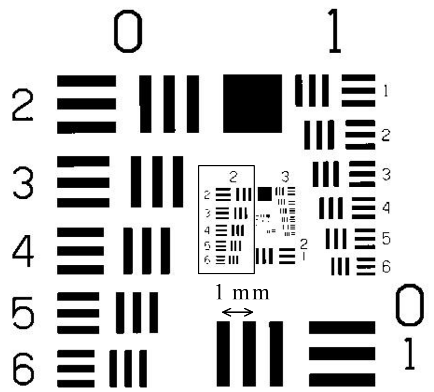

Figure 4. Image of USAF 1951 1X chart. 2G - 2L pattern is inside boxed line

Figure 5 shows signal outputs in measuring across a pattern of 2G-2L, second lines on second group of chart, on the USAF 1951 1X, which lying in a uniform array at a regular center interval of 223 μm. This result indicates that the radiation thermometer can distinguish clearly between adjacent patterns. In Figure 5, signal of the thermometer is very small for the background and increased at the positions of the pattern, in which the signal approached that of a blackbody radiance at a room temperature. The test pattern is a negative one, in which the pattern of quarts glass substrate is formed on a background deposited with chrome. The chrome 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 specular reflective surface of the chrome background. On the other hand, the detector senses the blackbody radiation near ambient temperature from the pattern of the quartz glass substrate having a high emissivity in the thermal infrared region.

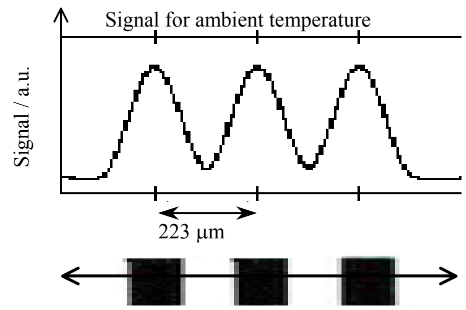

Figure 5. Radiance signal in measuring the 2G-2L pattern (4.49 pair / mm) on the test chart, USAF 1951 1X

Figure 6 illustrates the counter map for the test pattern, which was measured in two-dimensional scanning. On the left side, signal maps for the shape of figures representing the 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 below 1 mm.

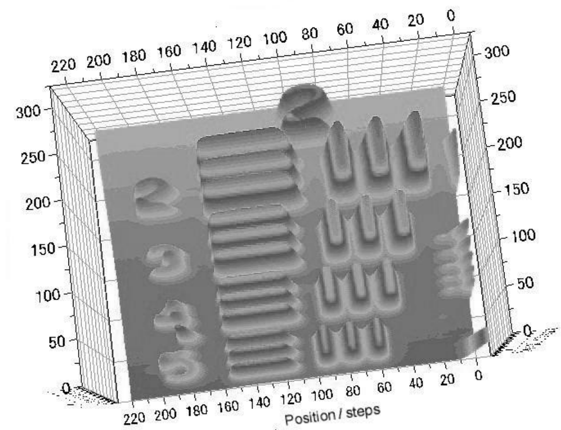

Figure 6. Counter map of radiant signal on a part of 2G chart of the USAF 1951 1X. line pitch of patterns : 223 μm for 2L, 198 μm for 3L, 177 μm for 4L, and 157 μm for 5L pattern

CONCLUSIONS

We developed a thermal infrared radiation thermometer for measuring the thermophysical properties of micro-scale substances at low temperatures based on the periodic heating methods and the pulsed heating methods. The thermometer which consists of the photovoltaic MCT detector with maximum spectral response around 10 μm and simple mirror optics is a compact and robust instrument. Temperatures down to –50 ^{o}C can be measured with 0.3 ^{o}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 much better than 1 mm, which was sufficient to investigate anisotropic materials, coatings, and films.

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