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A Contactless CCD Dilatometer for Foil Materials

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A contactless, compact, low-cost dilatometer based on a laser-pulse thermal-conductivity apparatus has been developed to measure the linear thermal expansion of foil materials. The two sample-edge images are projected onto the array of a charge-coupled device (CCD). Changes in sample length are determined from measurements of the corresponding displacements of the sample-edge images focused on the CCD. The dilalometer performance was tested by comparing results of measurements of the thermal expansion for pure copper with published data. The linear thermal expansion of an L-16-type foil of 20-μm thickness, which is a candidate material for thermocontrol layers (in engineering), was measured with the apparatus.

KEY WORDS: CCD; contactless; dilatometer; foil; thermal expansion.

1. INTRODUCTION

Thin films, foils, and coatings used in engineering are required to maintain dimensional stability under varying environmental conditions. There has been an increasing need in recent years for thermal expansion data of these materials. However, because of the problem of buckling and/or twisting of foils and thin films, measurements of their thermal expansion with common dilatometers constructed to measure bulky samples, such as rod-type dilatometers or interferometric dilatometers, are very difficult. A few investigators [1–3] have made special rod-type and interferometric dilatometers suitable for foil samples.

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In order to develop a simple and low-cost dilatometer which allows constraint-free mounting of the foil samples, we examined the feasibility of using a CCD technique [4] with an existing laser-pulse thermal-conductivity apparatus [5] at the Institute of Metal Research, Chinese Academia of Sciences. In this study, the foil sample is mounted vertically between two quartz glass plates in the sample holder to avoid buckling and twisting of the sample, and the displacements of the two sample edges are detected by utilizing two pieces of CCD.

The application of the CCD technique to dilatometry in our study is similar to the photoelectric dilalometer reported by Ruffino et al. [6]. The major difference is that, in our case, only two sample edges are magnified rather than the whole sample. This approach yields a higher magnification (and, in principle, a greater sensitivity), while the optical and mechanical arrangement is compact. This characteristic is particularly important for adapting the dilatometer to the existing laser-pulse thermal-conductivity apparatus.

In the present study, in order to obtain greater accuracy in the length change measurement as well as convenient experimental operation, refinements and improvements were made to most parts of the previous dilatometer, i.e., to the illuminator, optical arrangement, CCD photoelectric system (adopting a high-quality CCD with a high sensitivity), and data acquisition system. The performance of the improved CCD dilatometer was tested by comparing results of measurements of the thermal expansion for pure copper with published data. The linear thermal expansion of an L-16-type foil of 20-μm thickness, which is a candidate material for thermocontrol layers in engineering, was measured with the apparatus.

2. GENERAL CONSIDERATIONS

The paramount consideration when designing the measurement system is that the transformation between the measurement method for thermal diffusivity and that for thermal expansion can be carried out conveniently and easily. The thermal expansion measurement system is mainly the combination of a photoelectric displacement measurement system utilizing a CCD and a chamber in the laser-pulse thermal-conductivity apparatus. A schematic diagram of the instrument is presented in Fig. 1.

Prior to the thermal expansion measurement, the reflector R, the CCD photoelectric system, and the sample holder containing the sample were placed in their respective fixed positions in the laser-pulse thermal-conductivity apparatus. A fine adjustment was then carried out until the optimum condition for optical imaging was achieved. After the thermal expansion measurement, if we need to perform the thermal diffusivity measurement,

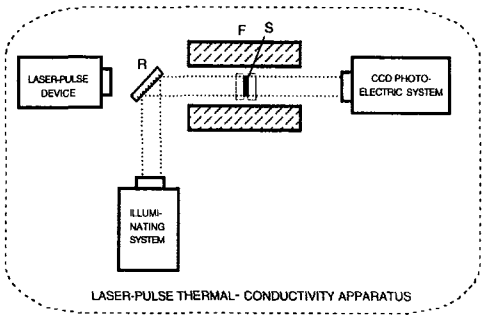

Fig. 1. Schematic diagram of the measurement system. R, reflector; S, sample; F, furnace.

then we remove the reflector R, the sample holder used for the thermal expansion measurement, and the CCD photoelectric system out of the fields.

3. MEASUREMENT SYSTEM

The sample is mounted vertically between two quartz glass plates on the support base of a special sample holder, as shown in Fig. 2. Since it is required primarily to measure the thermal expansion of selected foil materials in the temperature range from room temperature to 100°C and in this temperature range Invar alloy has very low thermal expansion, the sample holder and screws were made of Invar alloy to minimize the movement of the sample along the optical axis, which may cause the magnification of the optical system to change. In mounting the foil sample, the two screws are adjusted carefully until the quartz glass plates may shift slightly only along their plane direction if a small force acts on them. In this way, the sample may expand freely and not buckle during the experiment. An alternative way of mounting the sample involves placing three small pieces of foil which are slightly thicker than the sample around the sample between the two holding quartz glass plates and then attaching the two screws tightly (not shown in Fig. 2). The sample temperature is measured with a chromel–alumel thermocouple, which is in close proximity to the sample, through a hole in the rear quartz glass plate. The nominal length of the sample is about 10.2 mm, which is limited by the design of the optical system.

A schematic diagram of the optical system is shown in Fig. 3. The two edges of the sample are illuminated by a halogen condensing illuminator. A combination of lenses (L1), which is used as an optical relay for unit

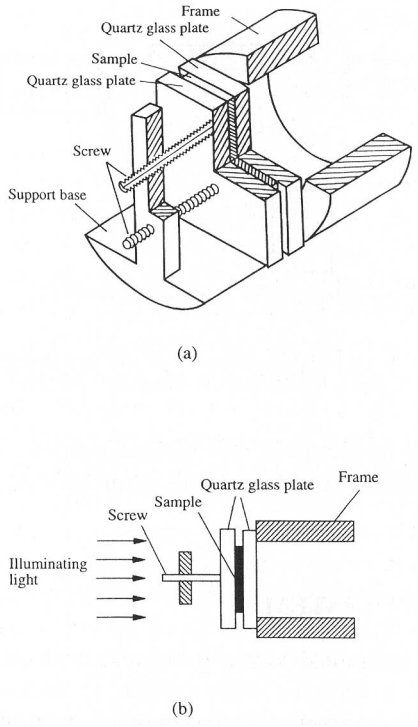

Fig. 2. The sample holder: (a) cut-away diagram; (b) horizontal cross-section diagram.

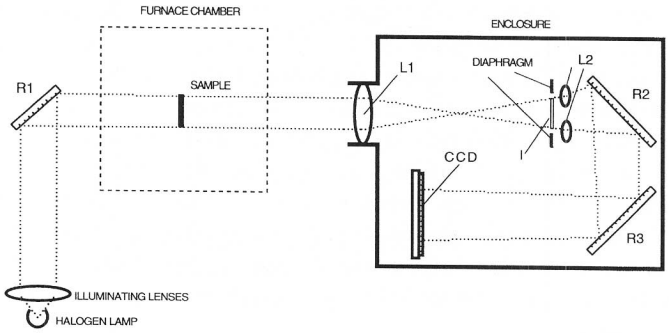

Fig. 3. Basic schematic diagram of the optical system. R1, R2, R3—reflectors; L1—relay lens; I—image of sample for unit magnification; L2—magnifying lenses.

magnification, forms the virtual inverted images (I) of the two sample edges at a conveniently close distance in front of a pair of the combinations of lenses of short focal length (L2). And then the virtual inverted images (I) give rise to the enlarged images of about 40× magnification on the plane consisting of the CCD (C). Reflectors (R1, R2) are inserted into the optical paths in order to make the optical system compact. The CCD adopted in our experiment (TCD 141 C, Toshiba, Japan) consists of 5000 pixels, with an interval length of 7 μm for a detector length of 35 mm. The purpose of placing a diaphragm in the same plane as the sample-edge images of the first stage is to avoid saturation overflow of the CCD video signals, which can be caused by the overlapping of the bright fields of the two sample-edge images. The exposure time of the CCD is controlled from 1 to 125 ms with a drive circuit which is used for scanning each pixel in sequence and acquiring a signal at each pixel. In our case at a magnification of 40×, the exposure time is taken as 68 ms. A data acquisition system performs an 8-bit A/D conversion of the video signal (yielding 256 gray levels) at a frequency of 1 MHz and stores the digitized video images with frame memory access which permits six digital images per sampling command. After the stored digital images are transferred into the computer for data processing, the next series of A/D conversions and store operations begins. Figure 4 illustrates a typical video-image output by the CCD during an experiment.

Because of the limitation of the resolution of optical lenses, the image of the sample edge is indistinct (Fig. 4). It is impossible to determine precisely which pixel on the CCD array the sample-edge image is located.

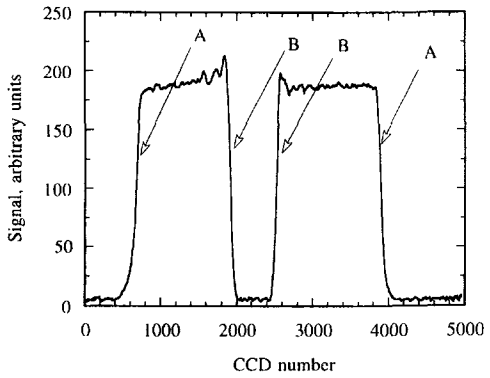

Fig. 4. A typical video-image output by the CCD during an experiment. (A) Illumination distributions for the two sample-edge images. (B) Illumination distributions for the diaphragm image.

However, it is known that the form of illumination distribution of the sample-edge image is identical at any place in the image plane of an ideal optical system [7, 8]. This is sufficient for the thermal expansion measurement, because the relative shift of the sample-edge image rather than its real position is needed.

As the sample is heated, its length l changes by an amount Δl, thereby giving rise to a shift in the video images of the two sample edges on the CCD array.

The equation for calculating Δl is

$$\Delta l = \frac{CN}{M} \tag{1}$$

where $C = 7\ \mu$m (interval of the CCD pixel), N is the total number of pixels counting the shifts of two sample-edge images, and M is the magnification of the optical system.

An expression for the linear thermal expansion of the sample follows from Eq. (1) as

$$\frac{(l - l_0)}{l_0} = \frac{CN}{Ml_0} \tag{2}$$

where l_0 is the reference length of the sample at 293 K. The position where the second derivative of the illumination transition of the sample-edge image is zero may be used as a reference point to calculate the length displacement [6]. In our study a so-called multipoint criterion is adopted for calculating the length displacement in order to reduce the uncertainties from the electrical noise that exists in the video signal. The procedure for calculating the amount of shift of one sample-edge image caused by the temperature change is described as follows. The average values in the top and base regions of the illumination transition for the sample-edge image are determined before changing the temperature, and then the difference between them is divided equally into 64 parts. The coordinates (that is, pixel number of the CCD array) corresponding to 32 equipartition values in the central portion of the illumination transition are calculated by means of pixel linear interpolation and taken as criterion points. By applying the same calculation to the sample-edge image after the temperature change, a second set of 32 criterion points is also obtained. The amounts of shift of the sample-edge image obtained from the 32 pairs of corresponding criterion points are averaged to yield the thermal expansion. A resolution of 0.1 of a pixel (0.7 μm) on the image is achievable. With a lens magnification of 40 $\times$, a length resolution of 0.02 μm is achieved.

4. DILATOMETER PERFORMANCE

The operation of the dilatometer included heating the sample with the furnace and measuring the change in the sample length. The test chamber was evacuated to a pressure of about 10^{-4} Torr, the sample was heated to a series of temperatures in succession, and a thermal steady state was established at each temperature. When the temperature reading did not change by more than 0.2°C in any continuous 5 min interval and the expansion did not change by more than a specified amount, thermal equilibrium was assumed and the program was run to take at least 18 frames of digital images. The average shift of these edge images relative to those at room temperature was then obtained, with a standard error of about 0.3 CCD pixel.

The imaging rays irradiated from the sample edge pass through a region of a refractive index gradient resulting from a temperature gradient, therefore, the so-called "mirage effect" occurs and leads to the deflection of the imaging rays. This might result in a false image shift or a defocusing of image and cause a measurement error in the relative change in the sample length. An estimate of the magnitude for the deflection angle of the imaging rays is needed. For simplicity, assuming that the temperature gradient is uniform over the region from the sample at a high temperature to the chamber window in front of the optical system and considering the case of deflection for an imaging ray parallel to the optical axis, the amplitude φ of the ray deflection is given [9] by

$$\varphi = (d/n_{T,p})(dn_{T,p}/dT)(dT/dx) \tag{3}$$

where d is the interaction pathway between the ray and the temperature gradient dT/dx, and $n_{T,p}$ is the refractive index of the gas at temperature T (in °C) and pressure p (in mm Hg). $n_{T,p}$ can be obtained by the following formula [10]:

$$n_{T,p} - 1 = (n_{15,760} - 1)\frac{p(1+\beta_T p)(1+15\alpha)}{760(1+760\beta_{15})(1+\alpha T)} \tag{4}$$

where T is the temperature, p is the pressure, α is 0.00366, $\beta_T = (1.049 - 0.015T) \times 10^{-6}$, $\beta_{15} = 0.813 \times 10^{-6}$, and $n_{15,760} - 1$ has the approximate value of 0.0003. The amplitude φ of the ray deflection can be calculated by combining Eqs. (3) and (4). Typical parameters in our case include $d = 230$ mm, maximum temperature $T_{\max} = 200$°C, and $p = 10^{-4}$ mm Hg. Even if a temperature gradient dT/dx of $100\text{°C} \cdot \text{mm}^{-1}$ (much larger than virtual value) exists, φ is very small (only a few 10^{-9} rad). Thus, the imaging rays can be regarded as straight lines for a practical imaging process, and no significant error can be caused by the "mirage effect."

The uncertainty in the linear thermal expansion arises from errors (random and systematic) in the following parameters: (1) edge image shift, (2) magnification of the optical system, (3) reference length of the sample at 20°C, and (4) sample temperature. In the present study, the error associated with the pixel location on the CCD array is considered to be negligible and is not estimated.

The error in the determination of the edge-image shift on the CCD results from the random electric noise in the video signal, the random mechanical vibration of the system, and the tilt or rotation of the sample during the experiment. The uncertainty combined from the first two sources is estimated to be about 0.3 CCD pixel. To evaluate the effect of a change in the tilt angle of the sample on the edge-image shift measurement, a telescopic collimator which existed in the laser-pulse thermal-conductivity apparatus for the collimating adjustment of the laser pulse path was used to monitor changes in the tilt angle of the sample relative to the base plane of the system during experiments under 200°C. With the collimator, however, no change in the tilt angle was observed. The uncertainty in determining the tilt angle using the collimator is 0.2°, corresponding to a shift amount of 0.4 CCD pixel on the image in the case of a magnification of 40×. If no correlation exists among the various error contributions, the total error in determining the edge-image shift on the CCD is estimated to be 0.5 CCD pixel (square root of the sum of squares of the individual errors).

The actual magnification of the practical optical system may be different from the desired value because of the overall uncertainties in the optical system. The determination of the true magnification was carried out using a translation stage which is driven by a precision micrometer with 0.15-μm differential resolution. A precision-machined plate, 10.2 mm long, with parallel optical knife edges on opposite sides, was mounted on the translation stage, and its two edges were detected by the CCD photoelectric system. If ΔL is the movement of the translation stage measured with the micrometer and $\Delta L'$ is the corresponding edge-image shift measured with CCD pixels, the magnification of the optical system is $\Delta L'/\Delta L$. In this approach, the magnification of the two opposite sample edges were obtained with a difference of 0.3% between them. The correction for different magnifications on the two opposite sample edges was made to the calculation of the displacement. The uncertainty in determining the magnification is estimated to be about 1%.

The sample length at room temperature (20°C) was measured using an optical comparator obtained commercially. The comparator consists of a traveling microscope [11] with a magnification of 40× and a system of micrometer screws having 0.1-μm resolution. The sample was held by two

glass plates and placed horizontally on the support table of the comparator so that it was flat due to the weight of the upper glass plate. The microscope has a micrometer eyepiece in which a pair of fixed parallel wires can be used to align the sample edge. The sample length is therefore the transverse displacement of the microscope from the position for aligning one sample edge to that for aligning the opposite sample edge. The major source of error in the reference dimension of the sample at 20°C arises from uncertainties associated with the sample-edge condition and the mounting condition of the sample in the holder before the experiment. Careful preparation of the sample could limit the divergence of opposite edges to within 2 μm/mm. When mounting the sample in the sample holder, we adjusted the rotation of the sample holder about the principal axis of the system until the tilt angle of the sample to the base plane of the system monitored by the telescopic collimator reached a maximum of close to 90°. The maximum possible error in the reference sample length at room temperature from the tilt of the sample mounted in its holder is less than 1 μm. Thus, it is estimated that the total uncertainty in the value of the sample length at room temperature contributes an error in $(l-l_0)/l_0$ not greater than 0.03%.

For most materials, the temperature measurement uncertainty is the dominant contributor to the thermal expansion uncertainty. After thermocouple calibration and correction for thermocouple-to-sample gradients, an uncertainty of 0.5°C remains.

For a sample with a thermal expansion coefficient of 10^{-5} K^{-1} and a length of 10 mm, the estimated overall uncertainty in linear thermal expansion at 100°C is not greater than 3%. A sample of high-purity (99.99%) copper with a thickness of 80 μm was used to evaluate the overall dilatometer performance; results for three runs are indicated and compared

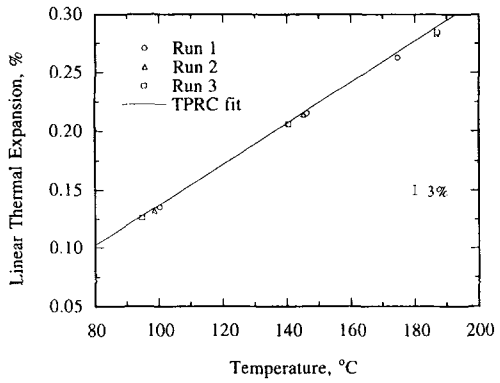

Fig. 5. Test measurements on pure copper.

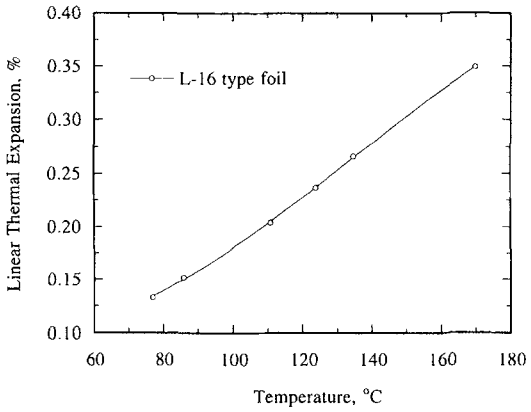

Fig. 6. Linear thermal expansion of a type L-16 foil thermocontrol layer measured with the apparatus.

with the recommended value by TPRC [12] in Fig. 5. Our data are systematically lower than the TPRC fit, but within 3%, demonstrating the validity of the method and apparatus. The linear thermal expansion for a type L-16 sample of a thermocontrol layer with a thickness of 20 μm was measured with this apparatus and is illustrated in Fig. 6. The maximum estimated uncertainty at 100°C is less than 3%.

5. CONCLUSION

A contactless, low-cost, and compact dilatometer based on a laser-pulse thermal-conductivity apparatus has been developed and tested. Results show that the dilatometer is capable of measuring the linear thermal expansion of foil materials over the range from room temperature to 100°C and thus meets the requirement for the evaluation of some types of thermocontrol foil layers.

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