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New Opto-Electronic Dilatometer[1]

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A new type of dilatometer has been designed, which uses a profile projector in connection with a solid-state camera. The sample image is projected onto an array of light-sensitive diodes which can be moved to intercept the image of the ends of the sample. The image length is determined by measuring the array shift and localizing the pixel where the light–dark transition occurs at both ends. Length and temperature are measured by interfacing the instrument to a CODAS (computer-operated data acquisition systems) and the same computer drives the camera and a positioning stepping motor. The dilatometer and its operation are described. Its resolution, precision, and measuring rate are analyzed. The instrument performance is checked by comparing results of its measurement of thermal expansion of tungsten with published data.

KEY WORDS: dilatometer; thermal expansion.

1. SPECIAL REQUIREMENTS OF HIGH-TEMPERATURE DILATOMETRY

Ceramics and some composite materials have low thermal expansion coefficients, namely, a few units in $10^{-6}\,K^{-1}$. Therefore their relative measurement with the push-rod dilatometer is impractical, since the sample and sample holder have very close thermal expansion coefficients. Absolute measurements, under steady-state conditions, also have serious difficulties at high temperatures with existing instruments. For example, optical comparators have the following drawbacks:

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(a) low measurement sensitivity requires long specimens, which makes sufficient temperature uniformity very difficult to achieve at high temperatures; and
(b) observation of fiducial marks becomes difficult at high temperatures.

In the case of interferometers, which have a high sensitivity and can use smaller specimens, deformation of the specimen holder may mask the specimen expansion.

A new experimental approach, the optical profile dilatometer, overcomes these difficulties since it has both a high sensitivity and stability. Descriptions of different implementations of this technique were recently presented [1, 2].

2. THE PROFILE PROJECTOR FOR THERMAL EXPANSION MEASUREMENTS

If the aperture stop, D (Fig. 1), of an objective lens, L, is placed in its back focal plane, the entrance pupil will be at infinity. Such a system is said to be telecentric in the object space.

In a pencil of rays which proceeds from each object point, there will be a ray which passes through the center of the entrance pupil and of the aperture stop. This ray is called the principal ray. The principal ray r of a telecentric system is parallel to the optical axis in the object space. Figure 1 shows that a small defocusing of the object caused by its displacement along the axis from 0 to 0′ does not change its height and therefore keeps the magnification unchanged. This property is exploited in measuring

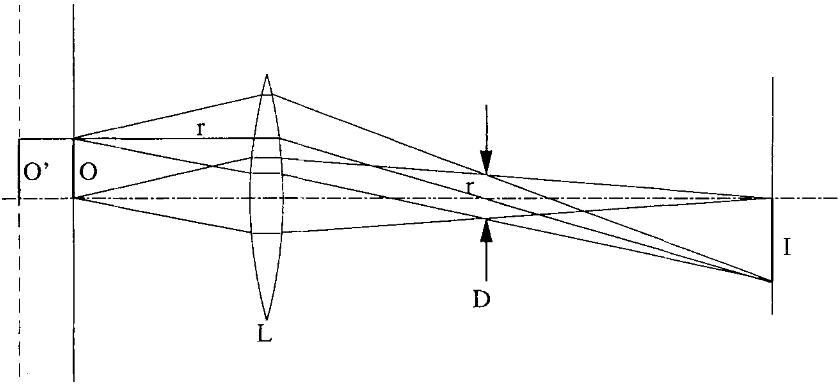

Fig. 1. Telecentric system. O, object; O′, displaced object; I, image; r, principal ray.

instruments such as scale readers and profile projectors. With the latter instruments a conveniently small aperture stop gives a large depth of field, so that the lens yields less confusing images of the object edges.

Profile projectors have been used for many years, but the incorporation of modern optoelectronic detectors has just entered into use for thermal expansion measurements. They present a number of advantages.

(1) Since the object is magnified, small specimens can be used. Temperature uniformity along the specimen can be achieved without any stringent design of the heater. Even high-temperature induction furnaces with large temperature gradients can be used with satisfactory results.

For example, for a specimen 20 mm long and a magnification of 10, it is necessary to keep the temperature uniform only over the 20-mm length, as compared to the 200-mm length required for a similar sensitivity using the comparator method. The reason for this fact is that the comparator microscopes, located at the ends of a long sample, magnify only the fields around the fiducial marks, while the projector magnifies the entire short sample to get the same resolution.

(2) No fiducial marks are required in the sample, since the measurement is done between its end faces. These can be conveniently detected at low temperatures with external illumination, or at high temperatures with sample self-emitted radiation, against a dark background.

(3) This system allows contactless measurements with no need for particularly fine machining. This is in contrast to the optical flats required by interferometers and is a great advantage in the measurement of thermal expansion of ceramics and composite materials. Contactless measurements allow constraint-free mounting of the samples so that the expansion is not affected by stresses that may develop with large changes in temperature.

(4) The sample end detection can be done photoelectrically, which allows the use of automatic data acquisition systems and, eventually, fast dynamic measurements.

(5) Variations of refractive index of air can slightly affect the dimension measurement of the image.

3. THE DMERU DILATOMETER

DMERU is the name of a dilatometer under development at the Department of Mechanical Engineering of the University of Rome Tor Vergata. It combines a profile projector with a solid-state camera. A schematic of the instrument is shown in Fig. 2.

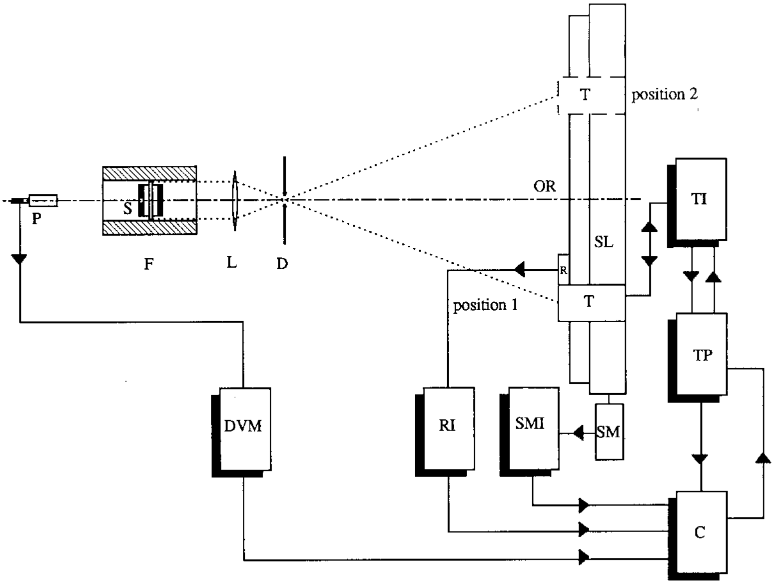

Fig. 2. DMERU dilatometer. S, specimen; F, furnace; P, pyrometer; L, lens; D, diaphragm in the focal plane of L; SL, slide; T, camera on carriage; SM, stepping motor; OR, optical rule; R, optical rule reader; C, computer; DVM, digital voltmeter; TI, camera interface; TP, camera processor; RI, reader interface; SMI, stepping motor interface.

3.1. Optical System

A telecentric system, consisting of lens L and field stop D in its back focal plane, projects the image of a specimen, S, onto a plane containing the sensitive area of solid-state camera T. Generally, the distance between the sample and the first surface of the optical system is constricted by the dimensions of the heating device. In order to increase this distance, an optical relay is inserted between the sample and the objective lens. The relay is a lens positioned for unit magnification, so that it carries the image at a convenient distance from the object. The aperture stop is conveniently inserted in the second focal plane of the lens.

The camera is mounted on a precision carriage running on slide SL, which is normal to the optical axis. To measure the sample image length the camera is positioned by a stepping motor SM on one sample end (position 1) and then shifted to cover the other end (position 2). Carriage displacements are measured with either the optical rule OR and optical reader R or a precision screw driven by the stepping motor.

The solid-state camera uses a linear photodiode array. Each element of the array detects an image element (pixel). Pixels are equally spaced at a distance of about 15 μm in the particular camera selected (Reticon array mounted on Cyclope camera, by Digital Design, France). For length measurement photodiodes are preferable to more conventional charge-couple devices (CCD), because they offer a more accurate pixel location. In our particular case, the sensor consists of 1024 elements making an optical rule about 15 mm long.

When a profile is projected onto the array, it causes an illumination transition in a particular region of the array whose width depends on the image sharpness. The video signal is digitized and processed by a computer and an interpolation is carried out to determine the position on the sensor where the second derivative of the transition curve is zero. The length measuring procedure can be easily understood if we consider the array as a reticule of a micrometric eyepiece. A pixel corresponds to a scale trait. Pixel interpolation is equivalent to linear interpolation between two adjacent traits. A resolution of 1/10 of a pixel (1.5 μm) is achievable. Figure 3 represents the illumination distribution on the array and the coordinate of the maximum slope (on the vertical line).

To perform a sample length measurement, the camera is placed on one end profile and the position reading n_1 is determined on the array. Then the camera is shifted to the other end by an amount l and the new position reading is n_2. The sample (magnified) length is

$$L = l + (n_2 - n_1) \tag{1}$$

The process can be repeated a number of times, allowing the average and the standard deviation to be computed. With a lens magnification of

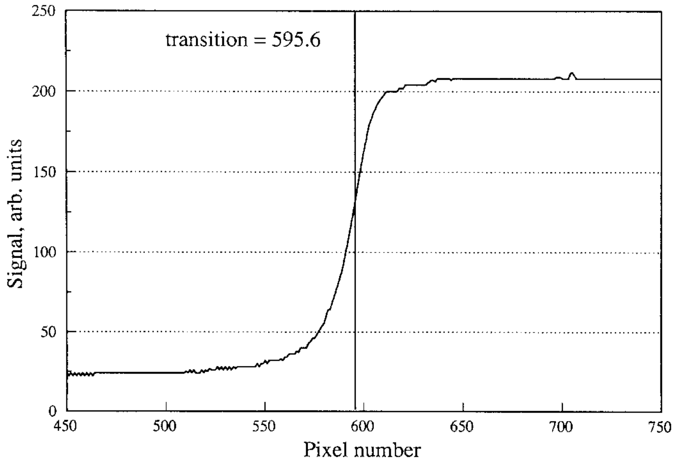

Fig. 3. Illumination distribution vs pixel number of the array, with coordinate of maximum slope.

10×, a length resolution of 0.15 μm is achieved, which, for a sample 20 mm long yields a relative expansion resolution of 7.5×10^{-6}.

An alternative procedure consists of placing twin cameras on the optical bench so that the sample-end images are projected onto fixed arrays. The twin cameras are shifted only in a preliminary run to determine the reference length at room temperature. They are then positioned so that the sample-end images fall on the array during the entire expansion process. In this way, the dilatometer can be used in dynamic measurements, since dead times for array positioning are avoided.

3.2. Thermal System

As seen in Fig. 2, sample S is located in the center and normal to the axis of tubular furnace F. The sample temperature is measured by a photoelectric pyrometer P, which is focused on a small hole drilled in a ceramic screen enclosing the sample to create blackbody conditions. The sample ends protrude from the screen and are seen along the axis. At low temperatures a platinum resistance thermometer is used for the temperature measurements.

If the specimen reacts with air, then the ends of the furnace tube are closed with optically flat transparent windows and inside there is either a vacuum or an inert atmosphere.

The length measurement is made with the sample self-emitted radiation at high temperatures. At low temperatures the sample is illuminated by an external light source with a condenser lens and a diagonal mirror on the optical axis. The mirror is pulled out of the field when measuring the temperature.

3.3. Automatic Control and Data Acquisition System

Measurements are performed automatically by means of a central processor C (Fig. 2).

An interface TI actuates the camera and acquires a signal at each pixel. Driving circuits control the exposure time (in the present case, from 256 μs to 100 ms), scan each pixel in sequence, and perform a 6-bit A/D conversion of the signal of each diode. This conversion has a frequency of 4 MHz and yields 64 gray levels.

The camera processor TP drives interface TI, stores image data in a buffer memory, and transfers the data to the central computer. The computer processes the image data to locate the profile. After a profile is recorded the computer actuates the stepping motor by means of an interface SMI, which also sends the position readings of the carriage driving screw to the computer. Position data are also sent to the computer by the optical rule reader R via reader interface RI.

The temperature is measured by pyrometer P, whose signal is generated by a silicon photodiode integrated into an operational amplifier. It is read by a digital voltmeter, DVM, and sent to the computer which contains the pyrometer calibration data.

The furnace temperature is controlled by means of a temperature controller and programmer.

4. EXPERIMENTAL

The determination of the coefficient of thermal expansion is based on the measurements of sample length and corresponding temperature. Each point is determined by alternating a number of readings (about 20) of temperature and length. If the temperature remains reasonably constant during the run, the point is defined by the average of both quantities. Standard deviations are calculated and displayed to estimate the metrological quality of the measurements. A computer program is run to determine and store the point coordinates.

A preliminary run is used to determine the room temperature (with a platinum resistance thermometer) and the corresponding sample length.

A number of temperature points is selected in the desired range and the furnace temperature controller is set at each of them in increasing order. Temperature is monitored on the computer screen during the transient, until the new value is stabilized. Then the program to take the point is run. Temperatures and lengths are stored in a table until the upper temperature limit is reached. Data are fitted by a polynomial of adequate degree, depending on the material. At this point the reference length, namely, the sample length at 293 K, is calculated and a new polynomial is generated to give the relative expansion as function of temperature. Finally, the derivative of this polynomial yields the coefficient of thermal expansion.

The sample used to test the dilatometer was the pure tungsten strip of a pyrometric lamp. Its nominal width was 4 mm. From calibration data, the radiance temperature of the sample at 650-nm wavelength is known. The true temperature was calculated on the basis of tungsten emissivity and window transmittance [3]. The width of this specimen was small with respect to what can normally be accepted by the instrument (20 mm); this poses severe conditions on the test.

The expansion resolution is 4×10^{-5}. A possible source of systematic error could be the band twisting: a band rotation of 0.5° could cause an error equal to the instrument resolution. A detailed analysis of this effect has been omitted in the preliminary test.

Three length measurements have been determined, each consisting the average of 15 readings. The first one was made at room temperature, giving the reference length $\bar{L}_0$.

Table I. Temperature, Length and Its Standard Error, Expansion and Its Standard Error, and Recommended Values of Thermal Expansion of Tungsten

T (K)	$\bar{L}$ (mm)	$s_{\bar{L}}$ (mm)	$10^3\,\Delta\bar{L}/\bar{L}_0$	$10^3\,s_{\mathrm{E}}$	$10^3\,\Delta\bar{L}/\bar{L}_0$ [4]
293	42.754	0.010	—	—	—
1570	43.034	0.013	6.55	0.41	6.45
1920	43.145	0.018	9.14	0.48	8.44

The expansion standard error s_{E} has been estimated on the basis of the computed standard deviations s_L and s_{L_0} of the length measurements according to the propagation of error formula:

$$s_{\mathrm{E}}=\left[\frac{s_{\bar{L}}^2}{n}+\left(\frac{\bar{L}}{\bar{L}_0}\right)^2\frac{s_{\bar{L}_0}^2}{n}\right]^{1/2}\bar{L}_0^{-1} \tag{2}$$

The errors caused by the temperature uncertainty were estimated to be substantially smaller than the length measurement errors.

The test results are presented in Table I, which displays temperature T, image length $\bar{L}$, standard error $s_{\bar{L}}$ of $\bar{L}$, the computed expansion $(\bar{L}-\bar{L}_0)/\bar{L}_0=\Delta\bar{L}/\bar{L}_0$, its estimated standard error, and recommended values of the expansion [4].

The disagreement of the two sets of data is lower than 1.5 times the standard error.

6. CONCLUSION

A thermal dilatometer using a profile projector and a solid-state array camera has been developed and tested. Results prove the target specifications to be quite realistic. Improvements can be made to the focusing system and to the mechanical rig to increase resolution and stability so that the precision of the length measurements will better match the camera resolution.

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