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Recent Advances in Optical Methods for Thermal Expansion Measurements[1]

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Progress in optical instrumentation for thermal expansion in the 1980s is reported. These instruments present some common features: (1) they are designed to operate at high temperatures; (2) they use optoelectronic detection so they can be linked to automatic data acquisition systems; and (3) they provide high-rate data acquisition. Three instruments follow a geometrical approach (profile projector or location with a laser beam), while two other ones are interferometric.

KEY WORDS: dilatometry; thermal expansion; high-speed methods; optical methods; high temperatures.

1. INTRODUCTION

Thermal expansion determination requires the measurement of temperature and length of the specimen. Optics are involved in both measurements at high temperatures, where radiation thermometry provides a means for temperature determination. In this field, optics supply the principle of operation and the guidelines for instrument design. The treatment of optics, as applied to thermometry, does not enter the scope of the present paper.

All methods of dimensional metrology can be applied to expansion measurement. A survey of dilatometric techniques is presented by Ruffino [1] and Kirby [2]. Two optical methods are followed in length measurement: geometrical and interferometric. The former makes use of

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optical imaging, light rays, or photometry, while the second utilizes interference of light waves.

1.1. Geometrical Systems

Before the use of interferometers the most precise instrument for length measurement was the *optical comparator*. It consists of two pillars standing on a base and supporting a horizontal beam on which two micrometric microscopes are mounted. To measure expansions at low temperatures (0 to 100°C), the specimen in a thermostated bath and a standard rules at room temperature are placed on a traveling carriage on the base. The twin microscopes compare the two lengths by means of micrometers which consist of cross-hairs that can be displaced across the image field by precision screws.

At high temperatures (up to 1600°C or more), thermal expansion measurements present several problems.

(a) The specimen must be heated in a furnace, which requires a reasonable working distance between the specimen and the microscope front lens. The solution involves providing the microscope with a relay lens between the specimen and the objective lens. This modified instrument is a telemicroscope.

(b) The specimen temperature must be well defined; therefore thermal gradients must be either measured to a accounted for, or suppressed. In the latter case, multisection tubular furnaces may be used, with up to five heater sections which can be individually controlled.

(c) Fiducial marks on the specimen must be well defined (sharp and thin) even when the specimen is light emitting. Marks can be made with wires clipped on the specimen in front of holes drilled through it, or by placing nicks on the specimen.

The most advanced dilatometer of this kind, working up to 1600°C, was described by Rothrock and Kirby in 1967 [3]. It appears that the extension of the temperature range and the improvement in the solution of the aforementioned problems require a new optical approach.

1.2. Interferometric Dilatometers

Interferometry was first applied to thermal expansion measurements by Fizeau in 1866. In the 1960s, a great step forward was triggered by the application of laser light sources which substantially increased the fringe contrast, by virtue of their long coherence paths (up to several meters), and reduced the photodetection requirements due to their high output power. Use of polarized light, photoelectric fringe detection, and electronic signal processing improved the resolution to a fraction of a fringe (to 1/100th), resulting in a tremendously superior metrological performance over the comparator in terms of resolution and accuracy. However, at high temperature, the same problem of temperature nonuniformity arises as in the case of the comparator, with the additional drawback of support and sample deformation that may cause end face rotations and consequently spurious fringe counts. Moreover, the majority of dilatometric interferometers requires the specimen end faces to be flat and polished, a condition which is difficult to maintain at high temperatures.

In 1976, Bennet [4] used a double-pass polarizing interferometer to measure thermal expansion with great accuracy up to 700°C. This is the upper limit at which this method retains its superiority. A Michelson interferometer was constructed by Wolff and Eselun [5] for measurements on long specimens of low-expansion composite materials, in the range below and moderately above room temperature. A Fizeau interferometer was constructed by Harrison [6] and used in 1980 by Preston [7] to measure the expansion of small annular specimens. A differential polarizing interferometer, based on the method of optical path modulation, was described by Roblin and Souche [8] in (1974 for applications in dilatometry requiring ultrahigh resolution (±0.005 Å). This interferometer was subsequently used at cryogenic temperatures to study dimensional changes during phase transitions in magnetic compounds [9].

This was the state of the art of interferometric dilatometry in the late 1970s. Since then, advances in interferometric methods have been brought about by their application to pulse heating techniques.

2. GEOMETRICAL DILATOMETERS

Recent developments in dilatometry have involved the application of modern optoelectronic detection to measuring instruments known as profile projectors. These instruments use an aperture stop in the back focal place of the objective lens, which places the entrance pupil at infinity. Such systems are said to be *telecentric in object space* and have the important property that a small defocusing displacement of the object along the optical axis will not change the image size, that is, the magnification.

Dilatometers based on profile projectors have a number of advantages.

(a) Since the object is magnified by an accurately known amount, small specimens can be used. Therefore good *temperature uniformity* along the specimen can be achieved even with heaters which exhibit large temperature gradients, as in the case of high-temperature induction furnaces.

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

(c) This system allows noncontacting measurements, with the further advantage that the end faces do not need particular machining, like the optical flats required by interferometers. This advantage is particularly important in the measurement of thermal expansion of ceramics and composite materials. Of course noncontacting measurements allow constraint free mounting of the specimens.

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

Profile projector dilatometers that have been recently implemented differ mainly in the image measurement approach and in the photoelectric detection system.

2.1. The IVTAN 2 Dilatometer

This name is given to the second of two instruments described in 1987 by Chekhovskoi et al. of the Institute for High Temperatures of the Academy of Sciences of the USSR (IVTAN) [10].

A schematic diagram of the dilatometer is shown in Fig. 1a. The enlarged image of the light-emitting specimen, 1, is reflected by means of the rotating mirror, 3, through a slit diaphragm, 4, which acts as the entrance window of photomultiplier, 6. The rim of the rotating mirror acts as the aperture stop, and if its rotation axis is placed in the focal plane of the lens, the system is telecentric. The authors do not describe the instrument in detail, but from their schematic diagram it is apparent that fiducial marks are used: one is the beveled right-hand edge of the specimen and the other is the left-hand bevel of a hole drilledthrough the specimen. This arrangement is used because the designers want equal pulses to be generated by the photomultiplier when the mark images cross the slit. The duration δ between the pulses generated at the specimen ends is measured by an electronic clock, 7. The mirror is driven by a synchronous motor with revolution period θ.

The principle of the length measurement can be easily understood

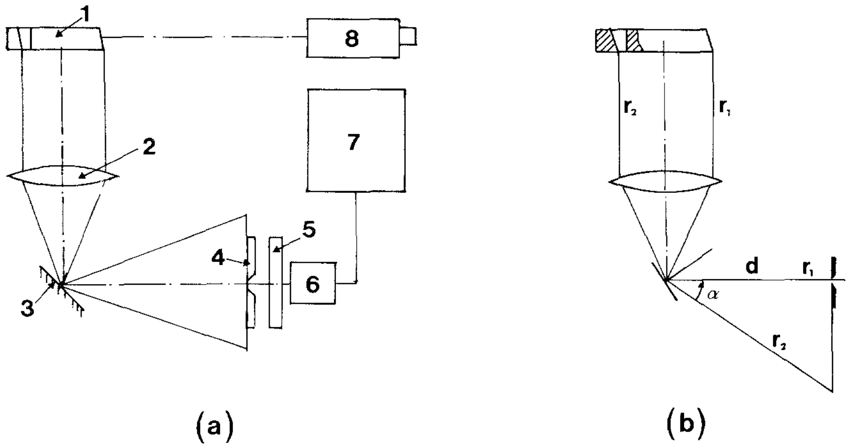

Fig. 1. (a) Schematic diagram of the IVTAN 2 dilatometer. (b) Optical paths in the IVTAN 2 dilatometer.

from Fig. 1b. It shows the mirror position which reflects the principal ray, r_1, exiting the first end mark to the slit. The principal ray, r_2, exiting the other end mark is simultaneously directed at an angle α to the first principal ray. A rotation of the mirror through angle $\alpha/2$ will direct the second principal ray to the slit. If the distance between the rotation axis and the image plane is d, then the image length is $l = d \tan \alpha$, where $\alpha = 4\pi\delta/\theta$. Therefore the image length is proportional to $\tan 4\pi\delta/\theta$ and its measurement is derived from two durations. This instrument is a length-to-frequency converter.

The length resolution depends on the slope of the leading edges of the pulses, the time resolution, and the acquisition time of the length. The pulse shape depends on the image definition and the rise time of the electronic circuit. This can be made quite fast to match the clock resolution and the definition can be improved by processing the signals with successive derivatives.

With $\tan \alpha = 1$, only 1/16 of a cycle is used to take a reading; the rest is dead time. Higher reading frequencies can be achieved with a polygonal rotating mirror. For the same value of $\tan \alpha$, a 16-sided polygon can be used, thus increasing the acquisition frequency by a factor of 16 with the same motor speed. With 16 readings per second, the length acquisition time should be 62.5 ms. Then, if the time resolution is 0.1 μs, the length resolution becomes 1.6×10^{-6}.

The specimen is heated in an inert gas atmosphere or in vacuum by an induction heater. Its dimensions are nominally 30-mm length and 10-mm diameter. The temperature is measured by an optical pyrometer, 7. The authors claim an overall dilatometer accuracy of 1%.

2.2. The DMERU Dilatometer

This is the name of a dilatometer under development at the Department of Mechanical Engineering of the University of Rome Tor Vergata.

The schematic diagram of a particular configuration of the instrument is shown in Fig. 2. A telecentric system consisting of lens, L, and field stop, D, in its back focal plane projects the image of the specimen, S, on a plane where the end faces are detected by two solid-state cameras, T_1 and T_2. The twin cameras are mounted on two chariots running on a slide normal to the optical axis. The camera positions are measured with an optical ruler, OR. Additional items are the camera driver cricuits, DR; the digital signal processor, SP; and the data acquisition system, DAS, which feeds the pyrometer and the camera signals to a computer, C.

Solid-state cameras use either charge coupled devices (CCD) or photodiode arrays. Each element of the array detects an image element (*pixel*). For the purpose of length measurement, photodiodes are preferable because they offer more accurate pixel location. In our particular camera selected (Reticon array mounted in a Cyclope camera by Dygital Design, France), the sensor consists of 1024 elements, spaced equally at a distance of about 15 μm; this is equivalent to an optical ruler about 15 mm long.

Driving circuits control the exposure time (from 256 μs to 100 ms) and scan each pixel in a sequence. Thus, the sensor yields a video signal

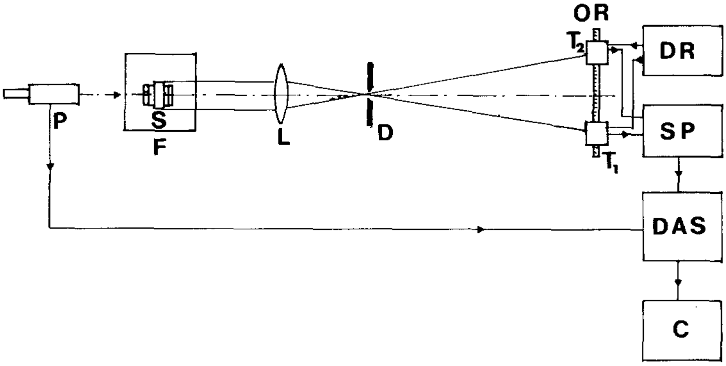

Fig. 2. Schematic diagram of the DMERU dilatometer.

which can be digitized. Our circuit performs a 6-bit conversion, which gives 64 gray levels, at a frequency of 4 MHz.

When a profile is projected on the array, it causes an illumination transition in a particular region whose width depends on the image sharpness.

It we set a threshold on the analog video signal or, in other words, if we make a one bit digitization, we can locate the pixtel where the black–white transition occurs. A sharp profile can be located with one pixel resolution (15 μm). But a fuzzy line could be located with several pixel uncertainty. In this case a pixel interpolation should be carried out by determining the position on the sensor where the second derivative is zero. For this purpose, the video signal is digitized and processed by a computer. A resolution of 1/10 of a pixel (1.5 μm) is achievable.

The reference length, namely, the specimen length at 293 K, is taken by measuring, with the optical ruler mounted on the cross-slide, the displacement of one camera between two positions in which the camera detects the end profiles. Readings of the pixel locator at each position are algebrically added to the ruler reading to get the length. The overall accuracy of this measurement is about 5 μm on the image (0.5 μm on the specimen).

With a lens magnification on 10 ×, a length resolution of 0.15 μm is achieved, which, for a specimen 20 mm long, yields a relative expansion resolution of 7.5×10^{-6}. The expansion measurement requires two simultaneous camera readings with an acquisition time of 256 μs. With direct memory access, a measurement frequency of several thousand readings per second should be possible, which makes the instrument suitable for dynamic measurements.

The principle of the dilatometer operation has been experimentally checked in the laboratory of Microtecnica, Turin, a firm which has a cooperation contract with the University of Rome Tor Vergata for non-contacting precision length measurements with profile projectors. The experimental setup is shown in Fig. 3. A lamp, an optical condenser, the specimen, the lens with unit magnification (an optical relay), and a linear CCD camera are mounted on an optical bench. The specimen is a cylindrical rod set on a carriage mobile on a cross-slide normal to the bench. The carriage is driven by a stepping motor and its shift is measured with a resolution of 1 μm by means of an optical rule mounted on the cross-slide. The camera is driven by a control circuit and the position of the rod profile on the array is detected by the pixel locator. As the pixel pitch is 13 μm with the actual camera, and the magnification us unity, 1-bit digitization is equivalent to a resolution is 13 μm. A computer receives, through serial ports, the pixel and the rule readings and actuates the stepping motor. It

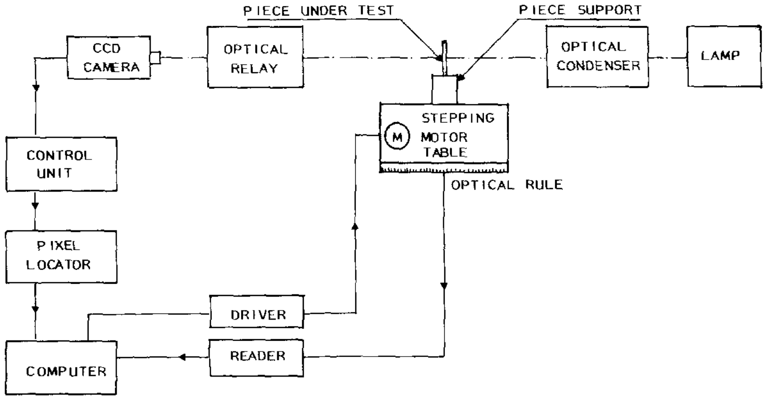

Fig. 3. Block diagram of the noncontacting length measuring instrument with-solid state camera.

calculates the specimen diameter by positioning the camera successively on the opposite sides of the cylinder.

A preliminary test has been carried out at room temperature. The true specimen diameter has been determined with a measuring machine whose accuracy is better than 1 μm: 10 readings yielded a mean of 9.480 mm with a standard deviation $\sigma = 0.5\ \mu$m. A total of 23 measurements was made with the camera, giving a mean of 9.483 mm and a standard deviation of 16 μm, which is almost one pixel dimension.

2.3. The IVTAN 1 Dilatometer

Another dilatometer was developed at the IVTAN by Checkhovskoi et al. [10]. Its operating principle consists of the comparison of the transit times of a laser beam across the specimen and across a standard length at different temperatures.

The setup, shown diagrammatically in Fig. 4, consists of a cylindrical tungsten heater, 500 mm long and 20 mm in diameter, inside a vacuum chamber. The specimen, of about 70-mm length and 10-mm diameter, 4, is suspended in the central portion of the heater. A blackbody cavity is fabricated in the specimen, and its temperature is measured with an optical pyrometer. The laser beam is reflected by means of a mirror, 3, mounted on the shaft of a low-speed synchronous motor. The standard is a rectangular diaphragm, 5, placed between the vacuum chamber and the rotating mirror. The laser beam passes through the gap between the edges of the specimen and the diaphragm and is directed with the aid of a system of lenses and mirrors, 6 and 7, to the photodetector, 9. The background light

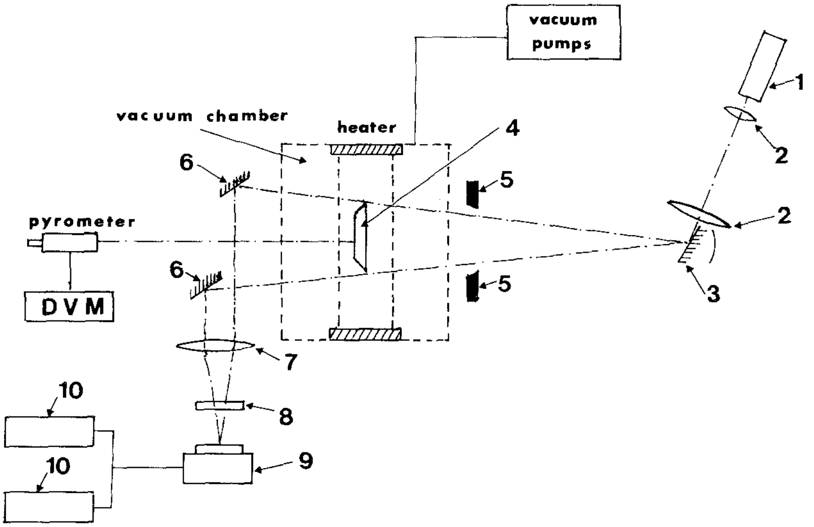

Fig. 4. Schematic diagram of the IVTAN 1 dilatometer.

is minimized by a narrow-band transmission filter, 8, centered at the laser wavelength. The transit times of the laser beam across the specimen t and across the diaphram τ are measured by means of two clocks, 10. The arc subtended by the specimen edges is equal to the product of the specimen distance to the mirror, the half-angular speed of the mirror, and the transit time t. In terms of the arc subtended by the diaphragm edges, the former is a quantity proportional to t/τ. Therefore the arc expansion, which is the same as the linear expansion, is proportional to $(t/\tau)_T - (t/\tau)_{293}$, when it is referred to room temperature (293 K). The values of τ in the two terms might not be equal, due to slight speed variations for each run. This is the reason why a reference length (the rectangular diaphragm) is placed in this system.

The same remarks made about the resolution and acquisition time of the IVTAN 2 dilatometer apply to this instrument as well.

3. INTERFEROMETRIC DILATOMETERS

The latest development in interferometric dilatometry has been its application to dynamic measurements. Two different approaches have been followed, resulting in completely different instruments.

3.1. The NBS High-Speed Dilatometer

Miiller and Cezairliyan, at the National Bureau of Standards (NBS) in the United States, first reported on the developed of an interferometric high speed-dilatometer in 1978 [11] and gave its full description in 1982 [12].

This instrument is a polarized Michelson interferometer and involves a number of optical techniques. As shown in Fig. 5, the specimen is inserted in the measuring leg of the Michelson interferometer, so that two opposite faces of it, in connection with lenses L1 and L2, make two retroreflectors: in this way any (small) rotation of the specimen does not cause an appreciable variation in the optical path difference (for an error analysis of this rotation refer to the Appendix of Ref. 12).

A polarizing beam splitter, PB1, separates the linearly polarized beam from the laser into two component beams. One component (s-polarized) is reflected around the specimen and into the detector by PB1, the pentaprism/lens combination PP1/L3/L4, plane mirror M1, and a second polarizing beam splitter, PB2, and serves as the reference beam. The other beam (p-polarized) is transmitted by PB1 and is directed to the quarter-wave plate, QP1, which has its optical axis oriented at 45° to the polarization plane. The emergent circularly polarized beam is retroreflected with a reverse sense of circular polarization, by the combination of lens L1 and the specimen face. After a second pass through QP1 the beam is again linearly polarized, but now with s-polarization so that it can be reflected around the specimen by PB1, pentaprism PP2, and mirror M2. By similar consideration of the optical elements PB2, QP2, and L2, one can show that after reflection from the back surface of the specimen, the component beam

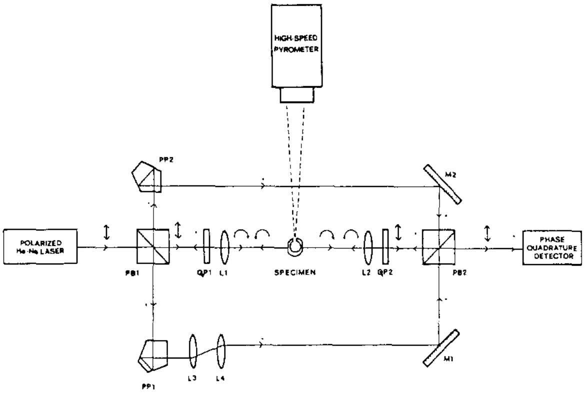

Fig. 5. Schematic diagram of the NBS high-speed interferometric dilatometer.

ultimately emerges from the interferometer with its original, i.e., p-polarization. The light output from the interferometer consists of two superimposed beams which are orthogonally polarized and cannot interfere to produce fringes unless they are brought to the same polarization plane. A novel polarizing device constructed by Miiller and Cezairliyan, which performs this operation is based on an earlier design (unpublished) by Hocken of the NBS. The device, when combined with suitable electronics, produces two signals proportional to sin δ and cos δ, respectively, where δ is the phase difference between the two output beams from the interferometer. The signals, therefore, are in quadrature and are ideally suited for bidirectional counting of the fringe movements. The signals must be conditioned before being fed to the counter. They are either amplified and clipped or used to actuate a Schmidt circuit to give two square waves in quadrature. These signals are sent to a commercial A-quad-B counter.

Some unique features of this apparatus are as follows.

(a) The optical path difference of the "specimen" leg is independent of rigid body translations by the specimen because of the successive front surface/back surface reflections. This fact is of vital importance in dynamic measurements where translational motion may be generated by large current pulses through the specimen. But it is also very important in static or quasistatic measurements because such translations are unavoidable with temperature variation, due either to support deformation or to friction effects on the supports.

(b) This interferometer uses parallel light, and therefore, it ideally produces "infinite-breadth" fringes. In practice this is not the case, due to imperfect collimation and reflecting surface irregularities. The wavefronts of the two emerging beams can be brought to near-equality by adjusting the distance between lens L3 and lens L4. Then the fringes are broad enough to produce almost equal illumination on the sensor surface.

Finally, it should be noted that the two legs of the interferometer have different optical paths (see Fig. 5). In addition, these optical paths are through air which may have differences in refractive index that could potentially influence the length measurements. This fact is acceptable in the present arrangement since the expansion measurements are completed in milliseconds, over which time the index changes will be negligible. For experiments of longer duration the design could be changed so that these paths would be *in vacuo* except where the beams are in coincidence.

A unique feature of the high-speed dilatometer is that the expansion measurements are taken between two lateral faces, on a transverse section of the specimen where the temperature should be uniform.

3.2. The IMGC High-Speed Dilatometer

Another pulse dilatometer was constructed in 1986 by Righini et al. [13] at the Istituto di Metrologia G. Colonnetti (IMGC) in Italy. In this instrument, the longitudinal expansion of a long specimen (cylinder, tube, or strip) is measured with a laser interferometer while a short-duration current pulse is passed through the electrically conducting specimen causing it to undergo rapid resistive self-heating. At the same time, the temperature profile of the specimen is measured with a high-speed scanning pyrometer that was developed for these measurements [14]. The expansion and the temperature profile are correlated to obtain the thermal expansion in the temperature range of the experiment.

A schematic diagram of the equipment is shown in Fig. 6. The electric current is fed to the specimen throught two clamps, one of which is fixed and the other is free to move in the axial direction. A total reflection prism is attached to the fixed clamp and corner cube retroreflector is attached to the mobile clamp. The prism and retroreflector are part of the measuring arm of a commercial Michelson interferometer. The distribution of the radiance temperature along the specimen is scanned by a rotating mirror attached to a fast pyrometer. The temperature of the specimen ends is outside the pyrometer range and is measured by two thermocouples, TC1 and TC2.

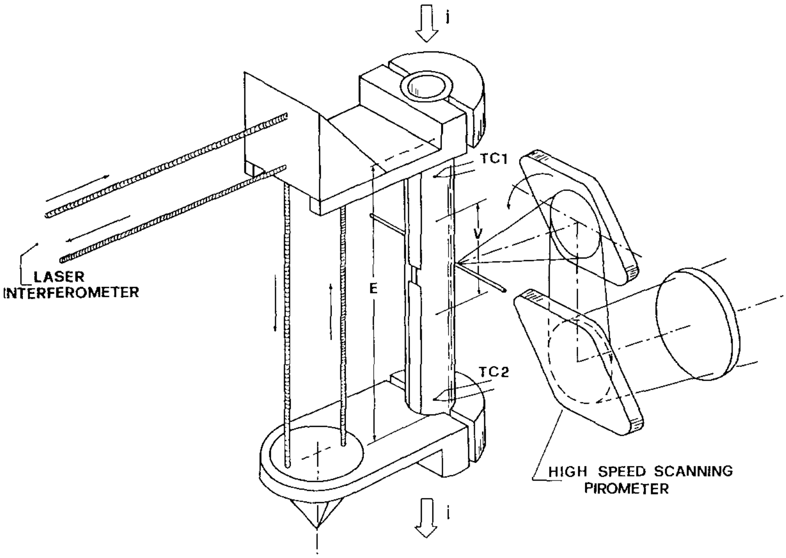

Fig. 6. Schematic diagram of the IMGC high-speed interferometric dilatometer.

The experimental data, temperature profile, and expansion are fitted using an analytical technique resulting in a polynomial expression relating the thermal expansion to the temperature. The fitting technique may be found in Ref. 12.

4. CONCLUSION

Over the last 6 years a number of innovative ideas in the design of optical dilatometers have been produced. They are intended mainly for measurements at high temperatures carried out with automatic data acquisition systems. They positively respond to the demand for new measurement techniques to investigate novel technological materials.

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