INSTITUTE OF PHYSICS PUBLISHING MEASUREMENT SCIENCE AND TECHNOLOGY

Meas. Sci. Technol. **17** (2006) 689–694 doi:10.1088/0957-0233/17/4/013

# Interferometric dilatometer for thermal expansion coefficient determination in the 4–300 K range

**G Bianchini[1], M Barucci[2], T Del Rosso[1], E Pasca[2] and G Ventura[2]**

[1] Istituto di Fisica Applicata 'Nello Carrara', CNR-IFAC, Florence, Italy
[2] Dipartimento di Fisica, Università degli studi di Firenze, Italy

E-mail: gb@ifac.cnr.it

Received 2 June 2005, in final form 23 December 2005
Published 10 February 2006
Online at stacks.iop.org/MST/17/689

**Abstract**
The measurement of thermal and mechanical properties of materials at cryogenic temperatures gains more and more importance in the field of the application of novel high-tech materials to aerospace industry and in developing scientific instrumentation. We present a simple and inexpensive interferometric dilatometer for the measurement of the thermal expansion of materials in the 4–300 K range. The dilatometer consists of a Michelson tilt-compensated interferometer in which the path difference is given by the variation in length of a sample enclosed in a 4 K cryostat. The compensation for misalignment permits a fast and simple operation routine that configures the instrument as a valuable tool for materials engineering.

**Keywords:** thermal expansion, characterization of materials, interferometry, cryogenic temperatures

## 1. Introduction

High sensitivity determination of thermal expansion of solid materials makes possible the investigation of material properties at low temperatures, where expansion coefficients are small. Also, a high sensitivity method permits us to determine thermal expansion properties of low-expansion materials of high technological interest.

While thermal expansion measurement in the high-temperature range has been thoroughly explored [1], and various experimental methods are available even as commercial instrumentation, measurements at cryogenic temperatures have been confined to the field of high-precision laboratory experiments, needing large experimental efforts and expenses, and often also suffering from intrinsic limitations.

Capacitive methods, first developed by White [2], have been widely used for high sensitivity thermal expansion measurements in the 0–300 K range [3–5]. While this method provides probably the highest sensitivity, it relies on calibration using well-known reference materials [6]; another limitation of this kind of measurement is the limited linear expansion range in which it is applicable. This requires that the geometry of the sample is varied depending on the expansion coefficient to obtain the desired plate spacing.

Several mechanical–optical thermal expansion measurement methods have also been developed, such as the *optical comparator* or the *twin telemicroscope* techniques [1]. In these kinds of measurements optical techniques are used only as a method to amplify the sample displacement, while characteristic calibration problematics typical of mechanical methods are still present.

A purely optical method like the interferometric technique permits us to overcome such problems. A typical application of this method consists in a Fabry–Perot interferometer in which the spacer between the reflectors is made of the sample material [7]. This method has been used to measure the expansion coefficient over a wide temperature range, both at high temperatures [8, 9] and at cryogenic temperatures [10, 11]. It should be noted that even in this case, the sample material mechanical properties, mainly machinability and dimensional stability, were determinant in the realization and alignment of the interferometer, limiting the measurement to metals and optical materials.

To overcome this limitation, the technique here described is based on a Michelson interferometer in which the sample

  

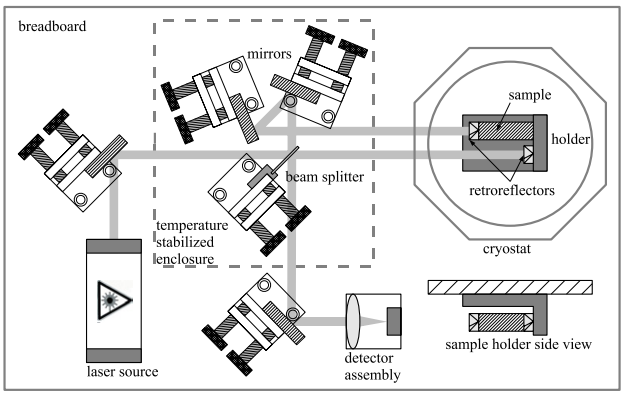


**Figure 1.** Experimental set-up for interferometric determination of linear expansion coefficient. Variations of sample length with temperature are detected as optical path difference variations on the interferometer.

acts as a spacer between two identical retroreflectors. This technique thus exploits the advantages of purely optical measurements to obtain a relatively simple method for the determination of linear expansion coefficients of materials with very different thermal, mechanical and electrical properties. In fact, even though this technique does not provide ultimate sensitivity as previously cited works do, it is less critical regarding sample mechanical properties, due to the tilt-compensation of the interferometer with respect to sample deformations given by the use of cube-corner retroreflectors, and has been successfully applied to measure metallic, amorphous plastic and fibre-reinforced plastic samples [12, 13] in the 4–300 K temperature range. It should be noted that a similar technique has previously been applied to measurement near room temperature [14], but has not been extended to cryogenic temperatures.

This capability of doing measurements with small preparation effort, and without much dependence on sample characteristics, together with the use of a simple, low-cost, experimental set-up, configures the described method as a valuable tool for routine material characterization in the field of materials engineering, aerospace engineering and cryogenic instrumentation development. For this purpose a detailed description of the apparatus is given to be used as an operative guide for the experimenter who is mainly interested in the characterization results with the minimum experimental and economic effort.

## 2. Experimental set-up

The interferometric dilatometer consists of a rather simple and small Michelson interferometer, in which the two arms of the interferometer are parallel, and of a $^4$He cryostat, in which the samples are held. The optical path difference between the two arms depends on the sample length; hence variation in the sample's length determines an interference signal.

### *2.1. Optical instrumentation*

The Michelson interferometer (figure 1) consists of a He–Ne laser source[3], four mirrors[4], two cube corner prisms[5], a 50% beam splitter (BS)[6] and a silicon photodiode detector[7] placed in the focal plane of a 50 mm focal length biconvex lens.

The laser beam is directed by a folding mirror towards the BS, and thus split into two arms: one is transmitted by the BS and directed towards the sample, by means of two more folding mirrors, the other is reflected by the BS and directed towards the sample holder. Laser beams are then back-reflected by the cube corner prisms that are fixed on the sample and on the sample holder, respectively. Since cube corner prisms are able to make the reflected beam exactly parallel to the incident beam, the interferometer is tilt independent.

The reflected laser beams get back to the BS by the same path, but 2 mm translated in the vertical direction; the BS lets a part of the two beams go towards the photodiode sensor, and the other part of the beams reaches the laser source (off axis, therefore giving no feedback effect).

The interferometer as a whole is 30 × 30 cm$^2$, the total optical path difference between the two arms of the interferometer, for a sample length of about 50 mm, is of the order of 10 mm or less. As we will see later (section 4), reduction of the maximum optical path difference contributes to the minimization of systematic error coming from laser frequency fluctuations.

To minimize systematic effects due to thermal effects on the interferometer assembly, the interferometer plate is actively stabilized to a temperature slightly higher than room temperature and insulated from air currents through a polystyrene foam shield. In figure 2 is shown a schematic of the stabilization circuit. The temperature fluctuation of interferometer support is kept below some tens of mK. As we will see later (section 4), this reduces to a negligible effect the variations of optical path due to thermal expansion of the interferometer assembly.

### *2.2. Cryogenic instrumentation*

Since measurements are made from 4 K to 300 K, samples are put in a two-stage $^4$He cryostat in which the sample is thermally connected to the liquid helium reservoir and enclosed in a thermal shield itself connected to the liquid helium reservoir. A second thermal shield connected to a liquid nitrogen reservoir encloses all the liquid helium system, permitting us to perform the warming up cycle at different speeds. Without cooling the second shield to liquid nitrogen we obtain a fast warm-up, particularly for the lower temperature range, while with the liquid nitrogen shield the warm-up from 4 K to 77 K is slowed down. This possibility is useful to check if some lag between thermometer read-out and actual sample temperature is present.

A small window[8] enables laser light to enter the cryostat vacuum chamber and to reach the sample through small holes in both thermal shields.

Samples are fixed to a copper support which is in good thermal contact with the liquid helium reservoir. The method used to attach the sample to the holder depends on the properties of the sample itself: if it can be machined, the sample is threaded and then screwed on the holder; or else

[3] AEROTECH: model OEM06XR, 150 mm cavity length.
[4] Edmunds Optics: Mirror PR AL 16 × 23 mm CO (cod. 30875).
[5] Edmunds Optics: PRISM CORNER CUBE 7.16 mm (cod. 43305).
[6] Edmunds Optics: BS PLATE 12.5 × 17.5 50R/(cod. 45316).
[7] Hamamatsu: model S1336-5BK.
[8] Edmund Optics: Window B270 30 mm DIA CTD (cod. 45254).

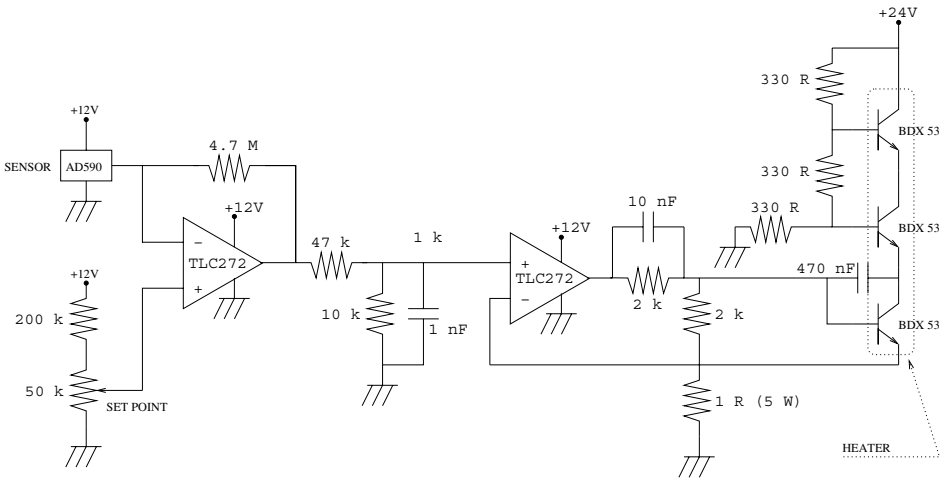


**Figure 2.** Thermal stabilization circuit used to keep the interferometer assembly temperature constant.

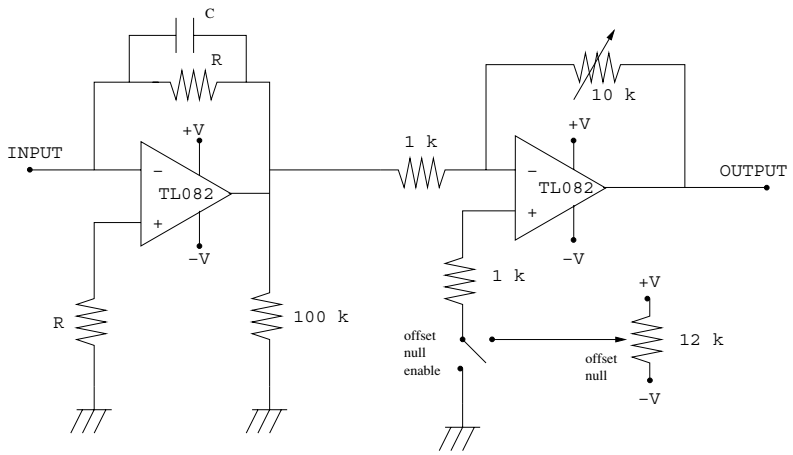


**Figure 3.** Current to voltage amplifier used to read the photodiode signal. *R* is chosen to obtain the desired current to voltage gain, and *C* is adjusted to match the time constant of the system to the data points acquisition interval.

it is glued to the holder with a proper cryogenic glue such as GE-Varnish. On the other end of the sample, the cube corner prism is fixed just as the sample is fixed to the holder. The second cube corner prism is fixed to the holder next to the sample.

The distance between the optical axes of the two arms of the interferometer is made as small as possible, considering a maximum distance limit due to window diameter (30 mm) and a minimum distance limit due to the diameter of the two retroreflector prisms and holders. The actual distance in the system is 11 mm. Minimizing this distance is also useful to reduce systematic effects due to misalignment.

Good thermal contact between sample and holder is granted by the copper tape wound around the sample; this also provides good thermal uniformity of the sample itself, together with the copper shield that covers the sample and the holder.

A calibrated carbon thermometer, put on the sample holder, measures sample temperature. Since a complete thermal warm-up, from 4 K to room temperature, takes more than 6 h, the temperature variation is slow enough to consider the sample and the sample holder isothermal.

The cryostat is mounted on the same breadboard on which the room temperature optics are by means of a kinematic mount so that it can easily be repositioned after sample changing with only minor alignment adjustments required to obtain an interferometric signal.

### *2.3. Data acquisition and processing*

The fringe interference signal is detected by a photodiode. Reading of the photodiode signal is by means of a simple current to the voltage amplifier, shown in figure 3. A Keithley 2000 Voltmeter is used to acquire the circuit output.

A computer system connected to instrumentation by a GPIB bus records interferometric fringe signal, sample temperature and interferometer assembly temperature at a rate of 1 Hz, even if it is simple to modify the data rate in order to keep under consideration different physical properties of the sample (for example, a higher data rate has been shown to be useful in the case of high thermal coefficient, where the interferometric fringe frequency is higher).

An example of a fringe signal is shown in figure 4, upper panel. The analysis of the fringe signal is done by the following steps: first the signal is smoothed through a Savitzky–Golay

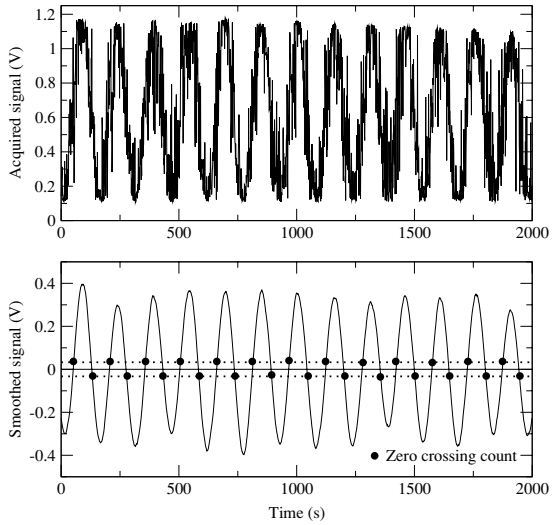


**Figure 4.** Successive steps in data processing. Upper graph: detected fringe signal. Lower graph: smoothed fringe signal; the two lines show the threshold levels used to count fringes without being affected by fluctuations due to noise.

algorithm, which parameters are optimized considering the average fringe frequency, in order to reduce noise without affecting the fringe signal.

After this step, another smoothing process is performed through a running average on a window large enough to enclose about 10 fringes, thereby obtaining the average zero level of the fringe signal. This level is then subtracted from the smoothed signal obtained in the previous step.

Last, the number of zero crossings in the obtained datafile is counted with a simple algorithm with hysteresis, in which the threshold level is optimized in order to avoid counting zero crossings due to residual noise in the datafile, as shown in figure 4, lower panel.

Since the distance between zeroes, in terms of sample length variation, is $\lambda/4$, where $\lambda$ is the laser wavelength, the total expansion of the sample is $\Delta l = N\lambda/4$. We can assess that the maximal theoretical accuracy of this system is $\lambda/4$, the limit coming from the fact that the system is not able to estimate fractional fringe variations. It should be noted that for the kind of measurements herewith performed, this limit is not a problem (see section 4).

## 3. Measurements

During a typical measurement run, the sample is first cooled down to 4 K. In order to ensure that the warming of the sample is sufficiently slow, in the standard measurement procedure a small amount of liquid helium is left in the vessel after thermalization, while the nitrogen vessel is filled. In this way, the warming up cycle is performed in two steps: the first, from 4 K to 77 K, starts when liquid helium finishes, and is slowed down through the presence of the 77 K shield, the second step, from 77 K to room temperature, is then performed when even liquid nitrogen ends.

In these conditions the system reaches room temperature in more than 6 h. A slow warming cycle is fundamental to ensure thermal homogeneity of sample and holder. In figure 5

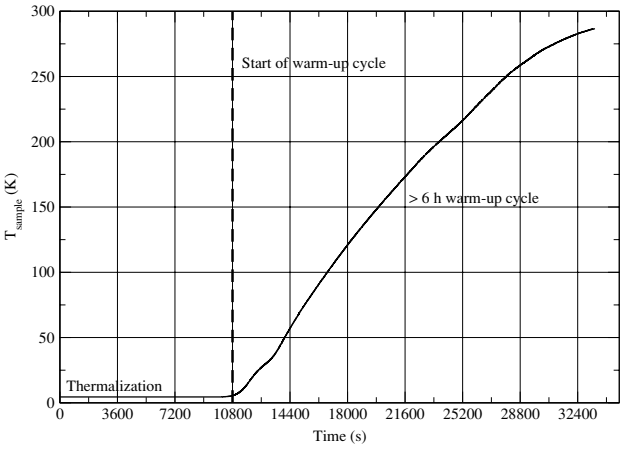


**Figure 5.** Measured thermal behaviour of the sample during a typical 6 h warming cycle from 4 K to room temperature.

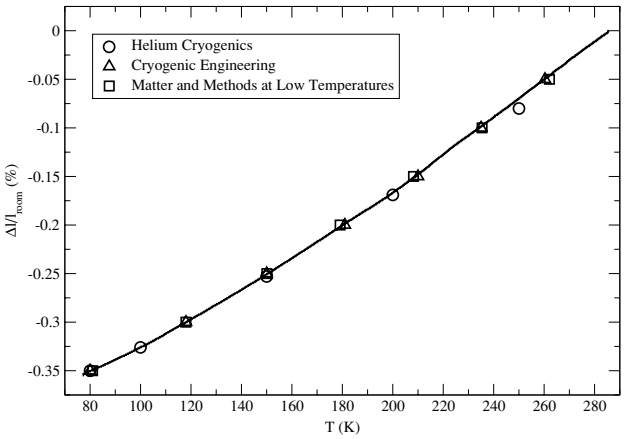


**Figure 6.** Measured thermal expansion of brass in the 77–290 K temperature range in comparison with some data points reported in the literature [15–17].

we show a typical warming cycle. It should be noted how the two stage warming cycle gives a homogeneous curve with a more or less constant slope: the average $\mathrm{d}T/\mathrm{d}t$ is about 1 K min$^{-1}$ and decreases only near room temperature.

In figure 6 the thermal expansion of brass measured with the described set-up is shown. In comparison, some data points present in the literature [15–17] are also shown. The interferometric method gives the advantage of a continuous measurement in which the whole expansion curve of the material (with respect to some discrete data points) is acquired.

In figure 7 thermal expansion curves relative to polypropylene and nylon, which show a total expansion $\Delta l/l$ between 4 and 300 K of about 1%, are shown. With such a great amount of expansion, and the consequent deformations of the sample, a method in which the sample geometry is critical could not be applied, while in the tilt-compensated set-up described, these effects are hardly detectable.

The same measurement method has also been applied to a carbon fibre composite characterized by $\Delta l/l$ of about 0.1%, demonstrating that it is applicable to materials which range over orders of magnitude in thermal coefficients.

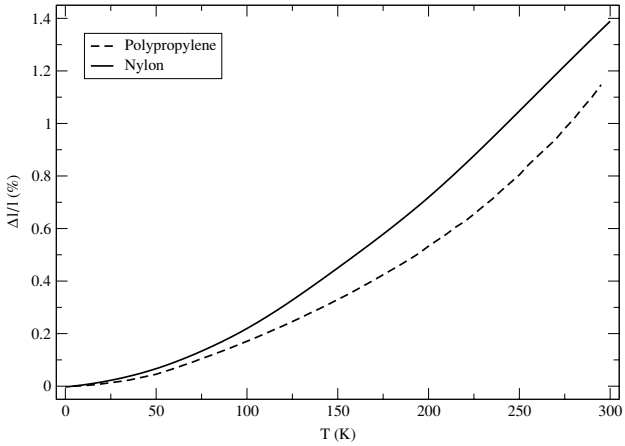


**Figure 7.** Measured thermal expansion of high thermal coefficient materials (polypropylene and nylon) measured in the 4–290 K temperature range.

## 4. Uncertainty budget

As we have seen before (section 2), the maximum theoretical resolution of our measurement system is of the order of 1 zero-crossing count, due to the simple fringe counting method used. The real accuracy of the system is limited by three different kinds of uncertainties identified in the experimental system described.

### *4.1. Thermometric issues*

The calibration of the sample thermometer gives a negligible effect, a maximum error of about 30 mK over all the operating region. The thermalization between sample and thermometer (and the thermal uniformity of the sample) are ensured by the shielding of the sample itself and by the very slow heating cycle used (>6 h from 4 K to 300 K). Comparison with faster cycles shows no variation in the results.

### *4.2. Thermal issues*

Various sources of error come from the thermal dilatation of parts of the system.

First, when the sample is machinable, the sample is fixed to the sample holder through a screw that threads for about 2 mm inside the sample itself; the sample retroreflector is also fixed to the sample edge through a similar screw. The effect due to different thermal expansion coefficients of the screws and the sample has been estimated varying the length of the part of the screws which are threaded inside the sample. A maximum effect of about 10 zero-crossing counts has been estimated, in the worst case (i.e. the maximum difference in thermal coefficients), for the normal measurement configuration.

Another important error source is the interferometer thermal dilatation. We measured this effect in about 4 counts/K by heating the interferometer assembly while keeping the sample at constant temperature.

To overcome this effect the interferometer is actively stabilized with an accuracy of some tens of mK, so the result is negligible (well below 1 count).

Last, the uneven thermal dilatation of the two cube corners and holders due to the possible difference in the thickness of the two cube corners assembly gives an extra contribution to the measured thermal dilatation of the sample. This effect can be estimated through a measurement made without sample (the two retroreflectors are fixed directly on the sample holder).

### *4.3. Optical issues*

If the laser beam directed towards the sample is not exactly aligned on the sample axis, measurement may be underestimated of about a factor $\alpha^2$ where $\alpha$ is the angle between the beam directed on the sample and the sample axis. The maximum misalignment permitted by the mechanical design is about 0.05 rad, which gives a maximum uncertainty of about 0.3%.

Cube corner retroreflectors compensate for any kind of translation of the sample holder due to thermal dilatation of internal components of the cryostat. While the main effect of the thermal dilatation of the cryostat is a vertical translation, a small rotation of the sample holder is still possible, giving an error of the order of $D * \delta$ in the total dilatation of the sample, where $D$ is the distance between the two beams (11 mm) and $\delta$ the rotation of the sample holder in the horizontal plane. Even this effect can be estimated with a measurement without sample.

The laser frequency stability depends mainly on thermal effects on the laser tube, the laser is well insulated from air flow, but some degree of temperature variation (about 10 °C) still remains. Relative laser frequency variation depends on relative thermal dilatation of the laser tube, giving a maximum effect of about $2 \times 10^{-5}$ for a temperature variation of 5 K. Considering the experiment optical set-up, in which the maximum path difference between the two arms of the interferometer is about 1 cm, this gives a maximum error of about 2 zero-crossing counts. This effect cannot be estimated correctly with calibration measurements with the sample at fixed temperature, because laser thermal fluctuations are not reproducible, so we assume the worst case estimate of 2 counts as an additional uncertainty.

### *4.4. Calibration measurement*

Various measurements without a sample have been performed showing a total count of about 3 zero-crossings on the 4–300 K temperature interval; as we have evidenced before, this is due to various effects such as sample holder rotation during cryostat heating, and mechanical differences in the two retroreflectors or holders.

We can estimate a total uncertainty budget, in terms of zero-crossing counts (corresponding to 1/4 of the laser wavelength), due to the aforementioned error sources:

- 0.3% of total expansion due to laser beam alignment,
- 2 counts (0.3 $\mu$m) due to laser frequency stability,
- 3 counts (0.5 $\mu$m) due to sample holders and retroreflectors,
- 10 counts (1.6 $\mu$m) due to sample fixings.

Total error can be estimated as 0.3% of the total measured dilatation plus 2.4 $\mu$m, that, for example, in the case of brass (total dilatation between 4 K and 300 K of a 50 mm sample equal to 0.2 mm), results in a total error of about 3 $\mu$m or 1.5% of the total measured dilatation.

## 5. Conclusions

We have presented a low-cost interferometric method for the measurement of thermal expansion coefficients of a wide range of materials in the 4–300 K temperature range. The method is relatively independent of the mechanical properties of the sample, without requiring the sample itself to be machined up to optical tolerances. This is achieved through the use of optical retroreflectors. The latter also give misalignment compensation of the interferometer, making the instrument less sensitive to sample deformation and thus permitting the measurement of materials characterized by very high thermal coefficients and/or anisotropic properties (e.g. fibre reinforced plastics).

Moreover, the experimental set-up described has been applied also to the characterization of materials with very low thermal coefficients, demonstrating effective operation over a range of various orders of magnitude in terms of thermal expansion.

A limitation of this method is that it is not able to detect an inversion of the fringe counting, as could happen in the case of a material characterized by non-monotonic thermal expansion.

A further development of the instrument will include an improved interferometer set-up that could provide two quadrature outputs, either by using polarizing components [18] or a semi-absorptive beam-splitter [19]. In this way a bidirectional fringe counting algorithm can be implemented permitting nanometric accuracy [9], and thus the measurement of low expansion materials even in the presence of a change in the sign of thermal coefficients with temperature.

Another valuable improvement that will be implemented is the use of a stable diode laser source [21] which will permit a sensible reduction of the size of the room temperature optics system and the integration of the laser source and the interferometer on a single small breadboard temperature stabilized within a few mK [21].

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