Thermal Expansivity and Temporal Stability Measurements at The University of Arizona

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We describe 40 years of experience measuring thermal expansivity and temporal stability, eventually achieving accuracy of 1 ppb (ΔL/L) for studies of Corning ULE and Schott Zerodur near room temperature.

Subject Terms: Dimensional Stability, Thermal Expansivity, Schott Zerodur, Corning ULE.

1. Introduction

The term dimensional stability includes dimensional changes with temperature, time, radiation, etc. Thermal expansivity is generally measured by means of dilatometers, which are typically calibrated interferometrically to λ/10 accuracy. When greater accuracy is desired it becomes more of a problem of temperature control and uniformity. Fortunately, most people don't require expansion measurements more accurate than λ/100. (typically ~ 10 ppb in ΔL/L).

After 40 years of service we are closing the lab. Dr. Paul Gohman and I are both retiring. Over that period we have had over 40 different customers and have enjoyed much fruitful collaboration, with applications including space exploration, astronomy, frequency standards, and relativity experiments. Sometimes it's hard to get customers to level with you. I was chiefly looking for business; they often had other things on their minds, such as commercial competition, military secrecy, and lawsuits.

Recently there has been interest in temporal stability (length changes with time at constant temperature) for space applications and XUV photolithography optics. Such measurements have called for accuracy much greater than that needed for most thermal expansion measurements. Our crowning achievement was Gohman's extensive and unpublished* measurements of Corning ULE, Type 7962, and Schott Zerodur thermal expansivity and temporal stability near room temperature, with 1 ppb accuracy. To accomplish this it was necessary to quantify the effect of mismatched end mirrors on ultimate accuracy and to use new end mirrors made of matched material. Gohman also developed a thermal cycling schedule to assure Zerodur temporal stability better than 1 ppb.

We describe the measurement methods we have used to measure thermal expansivity, differential thermal expansivity, and temporal stability. Also several experiences we had adapting sheet samples and fiber samples to our Fabry-Perot method. Finally, we present a history of the lab, a list of collaborations, and a discussion of future prospects.

2. Measurement Method

In 1967 A. D. White at Bell Labs locked a tunable red HeNe laser to a Fabry-Perot cavity resonator and heterodyne beat the laser beam against a stabilized HeNe laser.[1] By varying the temperature of the Cer-Vit cavity resonator he measured thermal expansivity of Cer-Vit (See figure 1). Our original

system (figure 2) eliminated the tunable laser, replacing it with tunable sidebands generated by KDP modulating the beam from the stabilized laser. This worked satisfactorily, but there was always a struggle for S/N due to the limited power in the sidebands. Eventually we wanted to automate the system, which demanded better S/N. In 1980 Dean Shough built a system along the lines White demonstrated. (See figure 3) In order to avoid ambiguity in the sign of the heterodyne beat the tunable laser used a different isotope of Neon from the stabilized laser (Ne^{22} and Ne^{20} respectively).

3. Comparison with other labs

In the 1970's Bill Plummer at Corning used a λ/40 interferometer, R. B. Roberts at CSIRO used a polarization interferometer, and Guy White at CSIRO used an absolute capacitance dilatometer to measure expansion. Our measurements of ULE [2] and single crystal silicon [3] agreed.

4. Ultimate accuracy

Our accuracy is limited by laser stability for low cte materials, and by ΔT uncertainty for high cte materials.[4] Especially for temporal stability measurements it was necessary to quantify the distance an average optical contact grows together with time, since this was the way our end mirrors were usually attached to the sample. This was found to be approximately the rms surface roughness in a time of about four months.[5,6] Henry Hagy pointed out that for measurements of 1 ppb accuracy the curved mirror substrate must be made of material identical to that of the sample. Subsequent experiments verified the correctness of his analysis.

5. Sample Configuration

The gain bandwidth of a HeNe laser is about 1500 MHz. This limits the tracking range of the tunable HeNe laser. Mirror separation L=10 cm corresponds to 1500 MHz spacing between TEM_{00} Fabry-

Perot resonances (See figure 1). For ease of alignment we chose the confocal Fabry-Perot configuration and assembled a collection of curved mirrors with radius of curvature 10.000 cm. This established a requirement that sample length be 9.975 cm, (10.000cm – 2 sags). Samples were mounted with optic axis vertical to avoid mirror slipping.

6. Direct Measurements of Differential Expansivity between Pairs of Samples

A new method was devised to measure directly thermal expansivity uniformity of materials for large telescope mirrors.[4] Figure 4 shows the optical arrangement. Figure 5 shows the low temperature sample chamber. As explained in reference 4, measurements were made by plotting beat frequency, f_{beat}, at thermal equilibrium vs equilibrium temperature. The slope of this line divided by the optical frequency f determines the differential expansivity $\delta\alpha$.

$$\delta\alpha = 1/f\left(\Delta f_{beat}/\Delta T\right)$$

Limiting accuracy is no longer set by laser stability since there is no longer a stable laser. Instead, the limiting accuracy is set by accuracy of the beat frequency measurement and by uniformity of sample temperatures.

7. Unusual CTE Measurements

We were challenged to measure expansivity in an anisotropic material ($LiAlSiO_3$) in 3 orthogonal directions. Since a 10 cm cube would be hard to obtain, and would require a new, much larger sample chamber, we devised a technique for working with higher order modes in shorter resonators.[7] A simple, uncluttered mode structure was maintained by requiring resonator length exactly ¼ of the customary 10 cm.

It took some imagination to measure expansivity of Tonen carbon fibers 10 μm in diameter, room temperature to liquid nitrogen temperature. Figure 6 shows the dangling Fabry-Perot resonator that

solved the problem.[8] Kodak's sheet Invar material was folded into the shape of a book, standing with spine vertical to support the upper Fabry-Perot mirror.[9]

8. Temporal Stability Measurements

Since time costs money, we built a system that holds many samples at constant temperature. The other important consideration is being certain that the temperature is constant. If it is not, then length changes may be due to thermal expansion instead of temporal expansion. Our system employed several independent temperature monitors: a platinum resistance thermometer, a copper Fabry-Perot resonator, and a fused silica Fabry-Perot resonator. Figure 7 shows the copper mounting block that holds 37 samples for temporal stability measurements. Figure 8 shows the optical arrangement for temporal stability measurements.

One very important result was collaboration with Jet Propulsion Lab in developing a high purity invar whose room temperature temporal stability was better than 1 ppm/year, with thermal expansivity less than 1 ppm/K. This material had especially low carbon content and was made by powder metallurgy.

Recently there has been great interest in determining temporal stability of Corning ULE Type 7962 (TiO_2-doped fused silica) and Schott Zerodur (a glass-ceramic). Both these materials have extremely low room temperature CTE.

7. History of the Lab

1968 Lab organized.

1970 First thermal expansivity measurements by Bradford and Jacobs.[2]

1976 Round Robin thermal expansivity measurements of silicon.[10]

Additional thermal expansivity measurements of low expansivity materials published. First temporal stability measurements published.[5,6]

1980 Optical heterodyne technique replaced tunable sideband technique.

1981 Thermal expansion *uniformity* of Heraeus-Amersil fused quartz.[11]

Temporal stability of Invar and Superinvar.[12]

1982 New technique introduced for directly measuring *differential* expansivity, eliminating stabilized laser.[4]

1983 Expansion hysteresis upon thermal cycling of Zerodur.[13]

Dimensional instability of Invars.[14]

1986 Temporal stability of various metals, alloys, welded joints, etc.[15]

Effects of γ-ray irradiation on dimensional stability of Cer-Vit.[16]

1989 Improved dimensional stability of Corning 9600 and Schott Zerodur.[17]

1990 "Unstable Optics" summary published.[9]

1992 Temperature and age effects on temporal stability of Invar.[10]

"Variable Invariables" Critical Review published.[18]

1993 Dimensional stability of high purity Invar 36.[19]

1999 Thermal expansivity of borosilicate glass, Zerodur, Zerodur M, and unceramized Zerodur at low temperatures.[20]

2000-2007 Extensive unpublished measurements of ULE and Zerodur thermal and temporal stability.

9. Collaborations

- Ben Averbach (MIT) supplied a sample of Superinvar (1982).
- Hal and Jean Bennett (NWC) time stability of many materials.
- Ramsey Melugin (NASA Ames) funded liquid helium cryostat.
- Karlheinz Rau (Heraeus-Amersil) uniformity of fused quartz.
- Karl Prewo (UTRC) graphite fiber reinforced glass matrix composite (1983).
- Pete Avizonis (AFWL) low temperature expansivity materials for directing high power laser beams; thermal cycling of Zerodur.
- John Alexander (TRW) time stability of Mo and Kovar welds and joints, TZM, Si_3N_4, SiC, Ti-6Al-4V, Cu, Al_2O_3, Elkonite.
- Jan Hall (NBS) stable cavity to check laser frequency stability.
- Henry Hagy (Corning) pointed out need to match mirrors for 1ppb accuracy.
- Guy White (CSIRO) very low temperature measurements and round robin comparison.
- Otto Lindig (Schott) understanding hysteresis effects in Zerodur.
- Dick Sumner provided invaluable constructive criticism and incredible skill at optical contacting.
- Clarence Babcock (inventor of Cer-Vit) many measurements for Owens-Illinois.
- David Schwab (AiResearch) long term measurements of Invar 36 (1983).
- Aden Meinel (OSC) suggested vertical sample orientation to avoid slipping mirrors.
- Jim Stonehouse (Brush-Wellman) HIP beryllium expansivity at low temperatures (1986).
- Tim O'Donnell and Witold Sokolowski (JPL) development of ultrastable Invar 36 for *Cassini spacecraft.*
- Roger Angel (Steward Observatory) thermal expansion homogeneity of Schott, Ohara borosilicate glasses and Heraeus-Amersil fused quartz for spin casting.

- Jeff Steele (Kodak) long term stability of sheet Invar.
- Nick Koumvakalis (Litton) stability of laser gyro resonators.
- Dan Vukobratovitch (KPNO) time stability gradients in Al(6061-T6) and SXA metal matrix composites for lightweight mirrors.
- Jason Gwo (Stanford) low temperature fused quartz expansivity for G*ravity Probe B* relativity experiment.
- Jim Burge (OSC) low temperature expansivity of thin face sheet materials for *Next Generation Space Telescope.*
- Lester Cohen (SAO) graphite-cyanate ester composites for NASA Advanced Xray Astrophysics Facility.

10. Future Prospects

Two areas that need further exploration are temporal stability and thermal expansivity at temperatures below that of liquid nitrogen (77K). Typical of the surprises that await us at very low temperatures are the CTE zero-crossings of borosilicate crown glass (~31K) and unceramized Zerodur (~82K). (See Figure 9). There is also continuing interest in homogeneity and gradients of thermal expansivity.

Regarding our Fabry-Perot method, its main claim to fame is that measurements are absolute, provided mirror material matches the sample material. It is therefore excellent for calibrating dilatometers that are far less costly to operate.

11. Acknowledgements

The early years of the lab were supported in part by Project Themis (AFOSR) and by The Air Force Space & Missiles System Organization. We are grateful to Dr. G. K. White of CSIRO who suggested we undertake these measurements and who supplied the single crystal silicon for

interlaboratory comparison measurements. Later work was supported in part by NASA (Marshall Space Flight Center), RADC, and DARPA. We are grateful to Lester Cohen of Smithsonian Astrophysical Observatory for donation of a frequency stabilized HeNe laser.

FIGURE CAPTIONS

Figure 1 The expansion measurement technique.

Figure 2 Our original system. KDP modulator M impressed tunable sidebands on laser beam.

Figure 3 Automated system used to measure expansivity.

Figure 4 Differential expansivity system. Optical arrangement.

Figure 5 Differential expansivity system. Low temperature sample chamber.

Figure 6 Dangling Fabry-Perot used to measure carbon fibers.

Figure 7 Copper mounting block that holds 37 samples for temporal stability measurements.

Figure 8 Optical arrangement for temporal stability measurements.

Figure 9 Low temperature CTE of several materials useful for optical engineering

* For XUV photolithography application, for Corning, and for Schott.

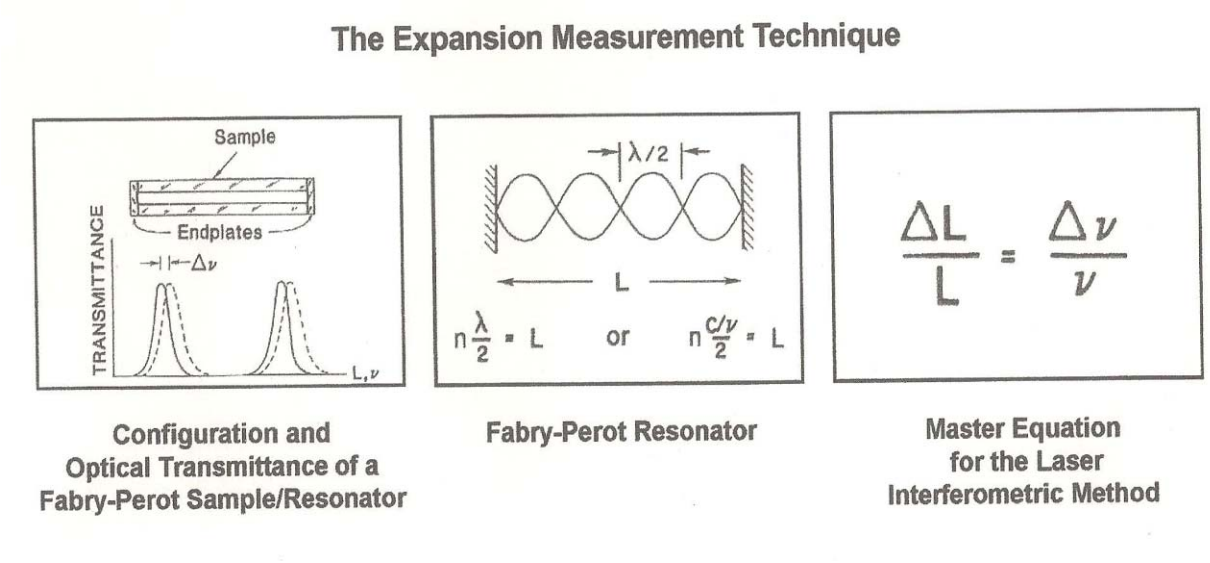

Figure 1 The expansion measurement technique.

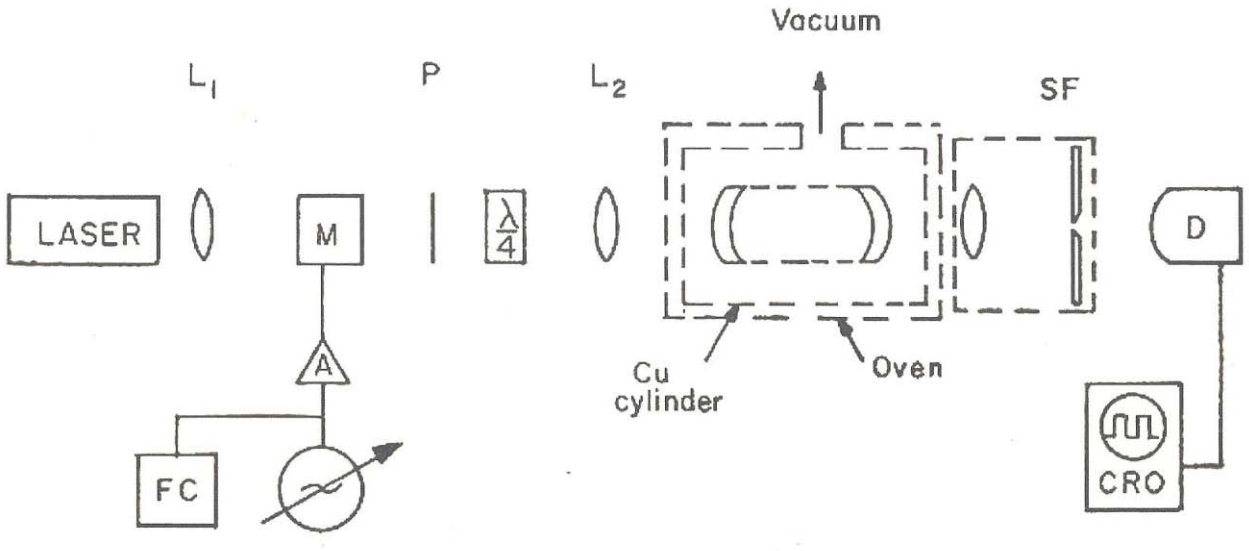

Figure 2 Our original system. KDP modulator M impressed tunable sidebands on laser beam.

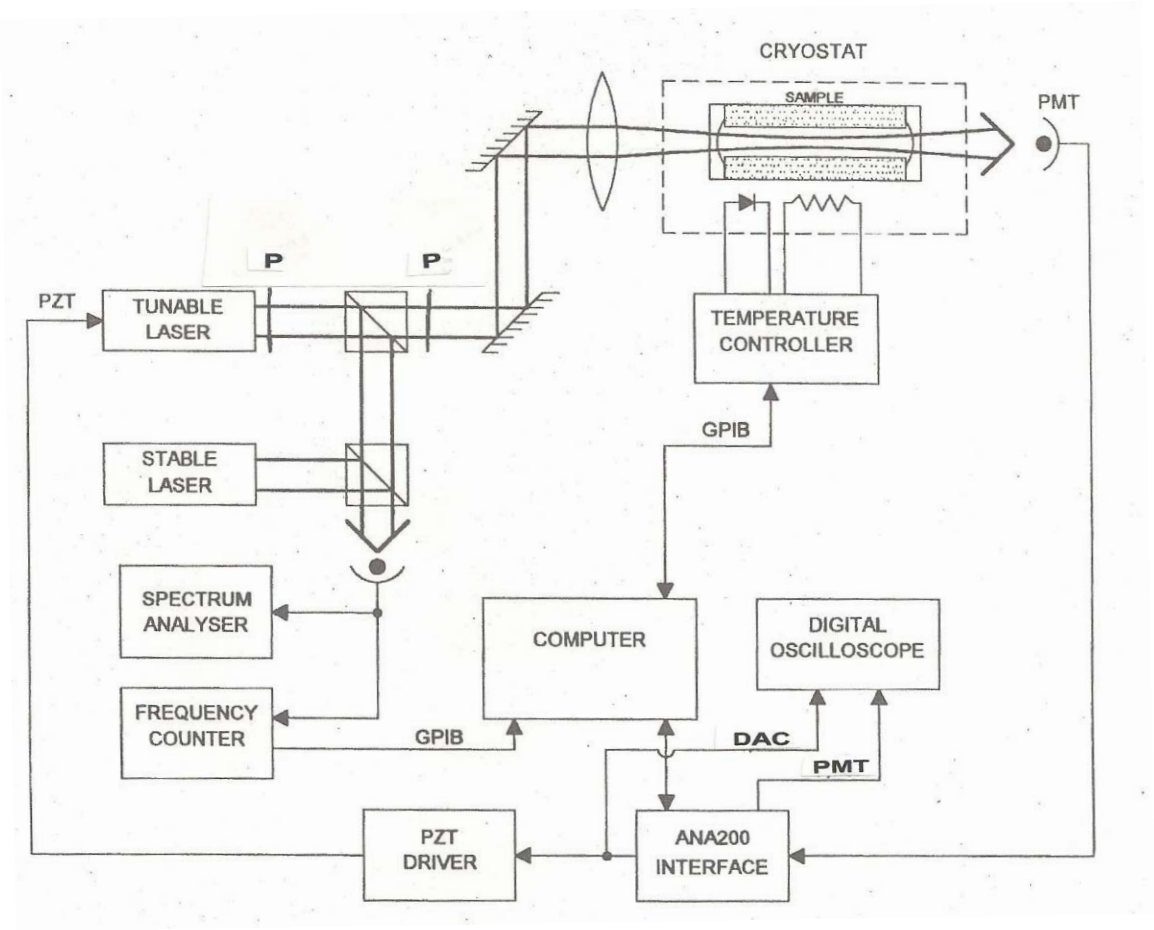

Figure 3 Automated system used to measure expansivity.

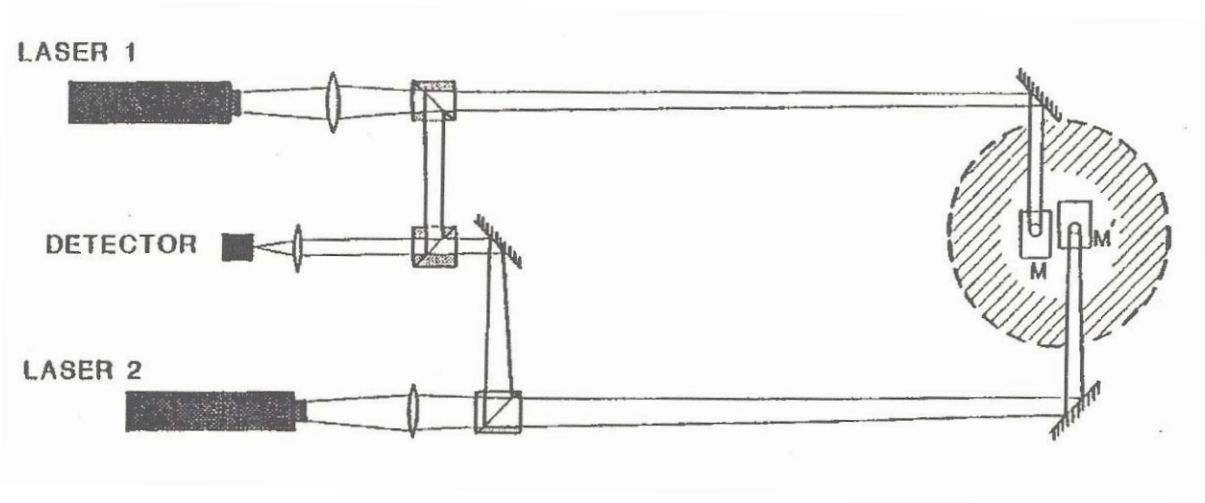

Figure 4 Differential expansivity system: optical arrangement.

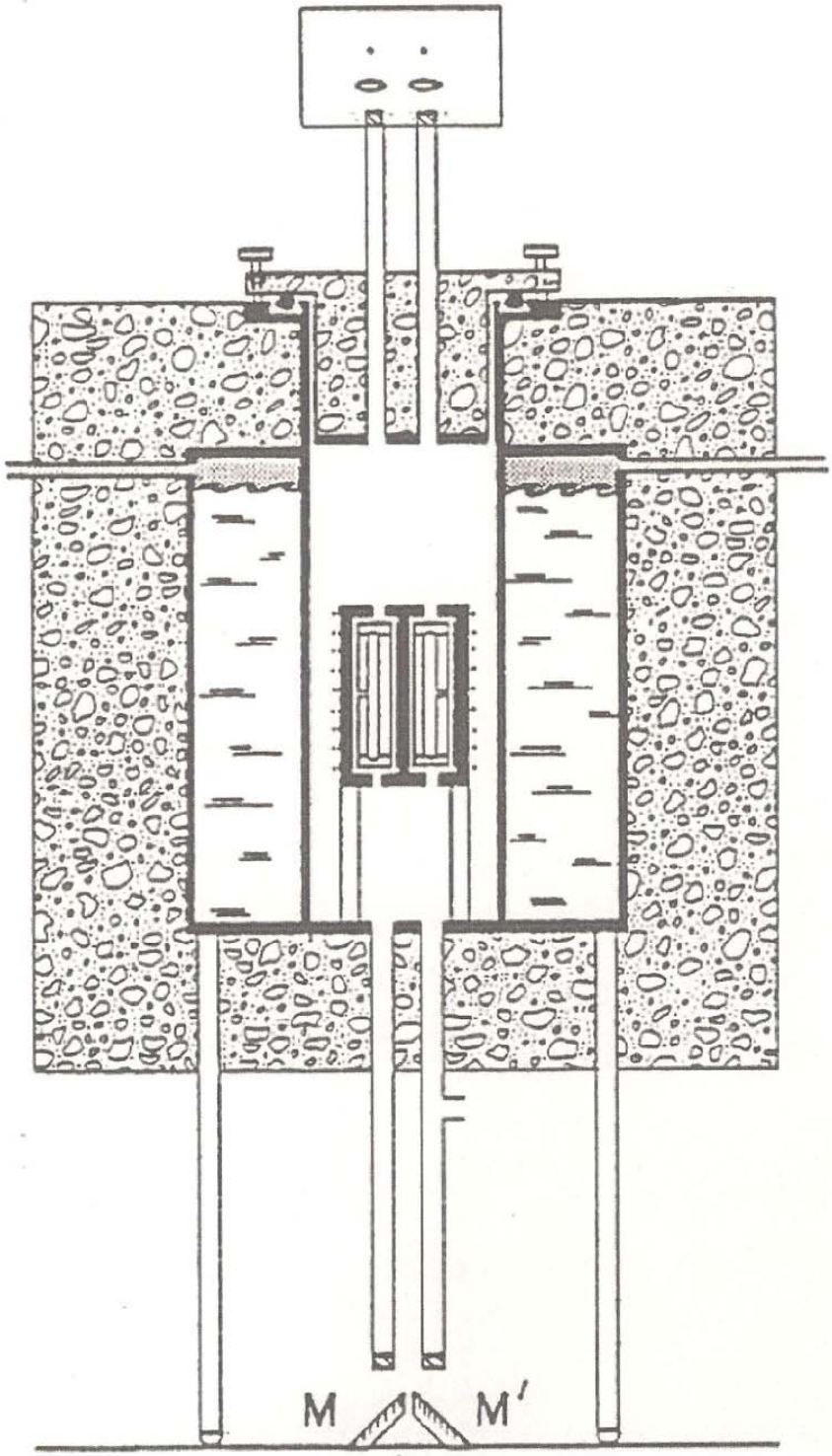

Figure 5 Differential expansivity system. Low temperature sample chamber.

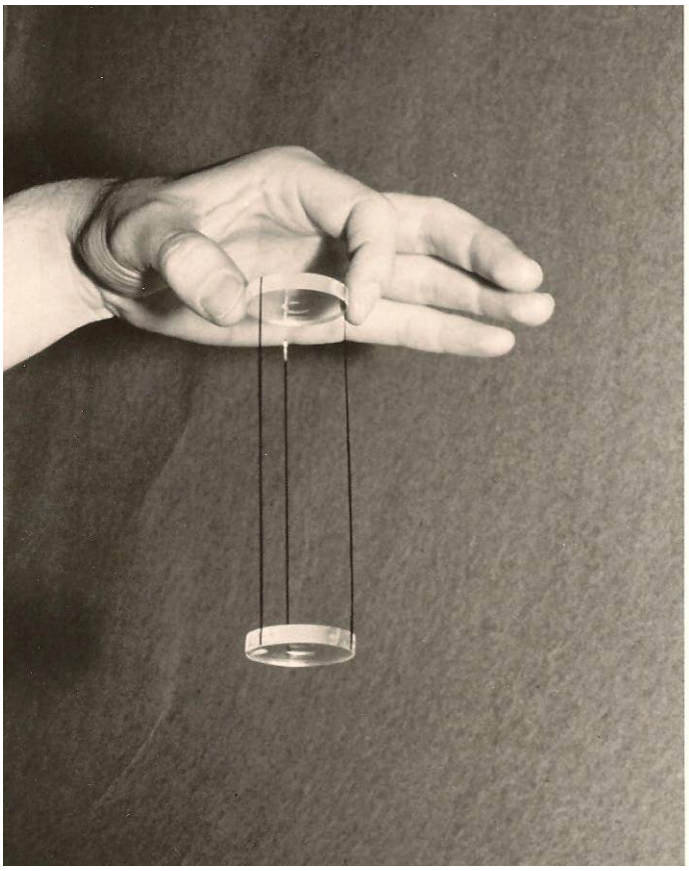

Figure 6. Dangling Fabry-Perot used to measure carbon fibers.

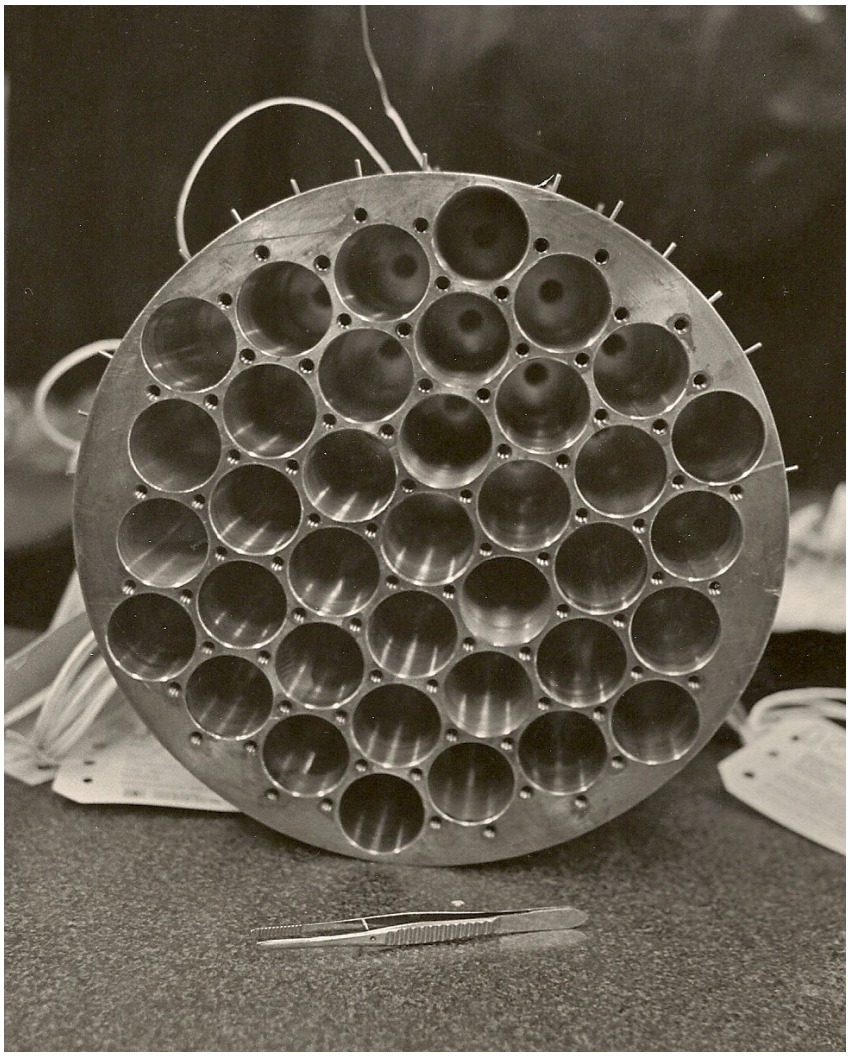

Figure 7. Copper mounting block that holds 37 samples for temporal stability measurements.

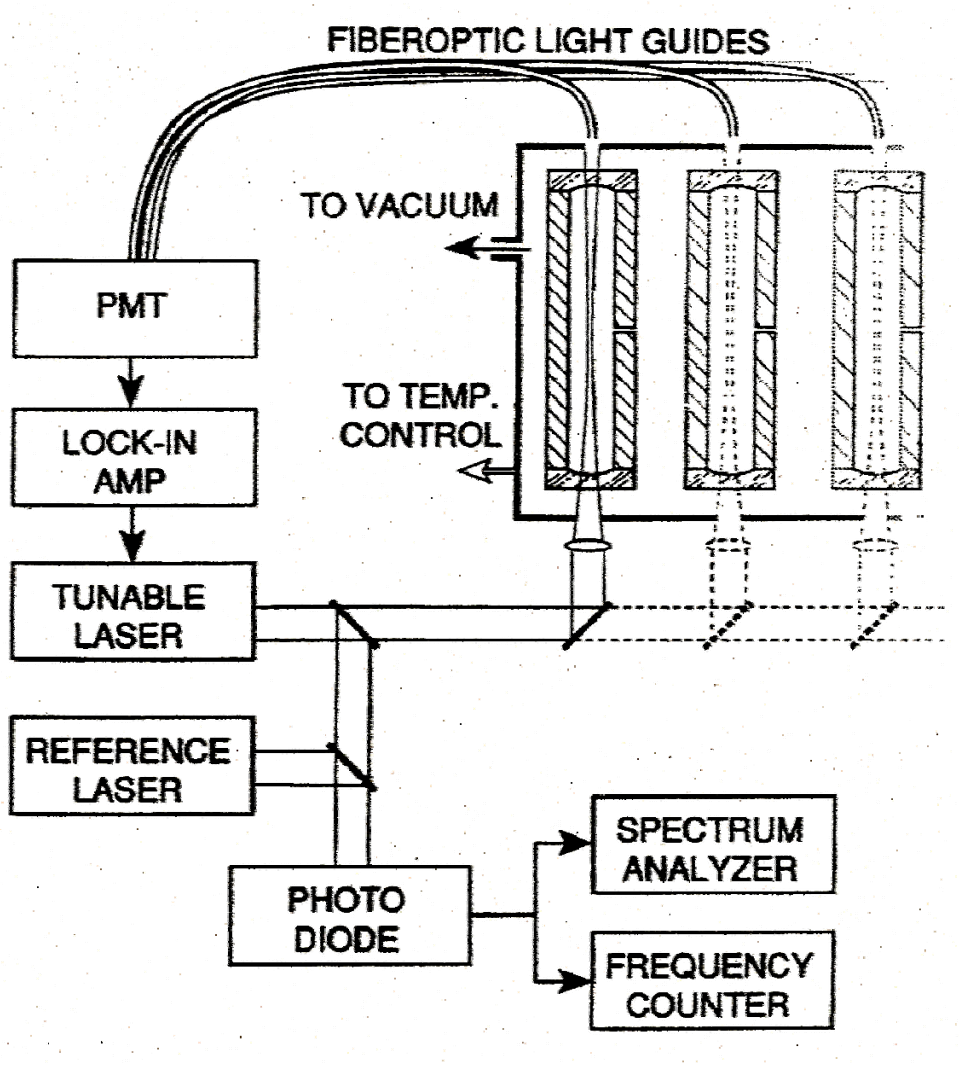

Figure 8. Optical arrangement for temporal stability measurements.

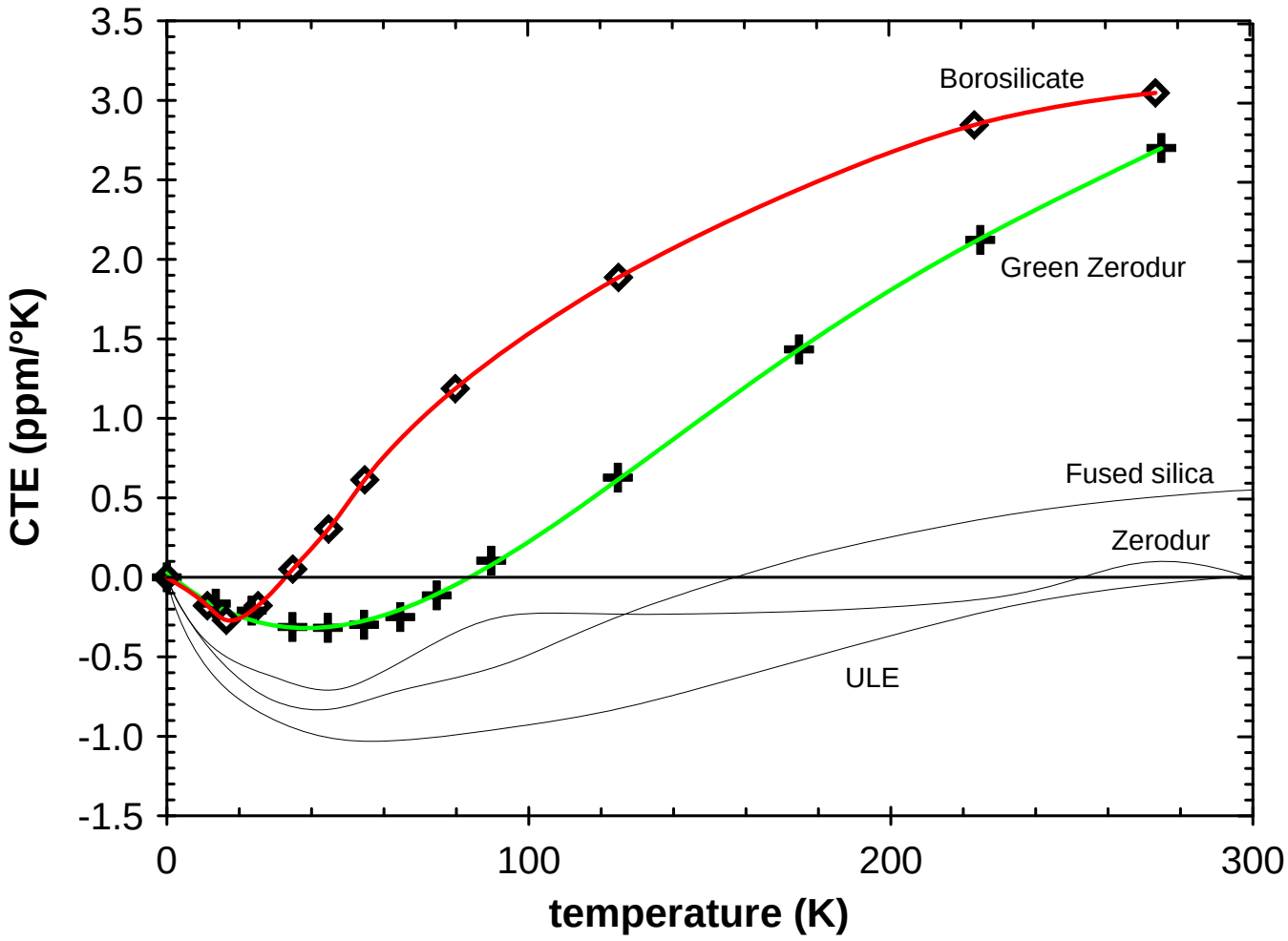

Figure 9. Low temperature cte of several materials useful for optical engineering

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