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MEASUREMENT OF DIMENSIONAL STABILITY

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CONTENTS

ABSTRACT

A technique has been developed for measuring, with a precision of one part in 10^9, changes in physical dimensions $\Delta L/L$. Measurements have commenced on five materials: Heraeus-Schott Homosil (vitreous silica), Corning 7940 (vitreous silica), Corning ULE 7971 (titanium silicate), Schott Zero-Dur, and Owens-Illinois Cer-Vit C-101. The original study was extended to include Universal Cyclops Invar LR-35 and Simonds-Saw Superinvar. As of this writing, funds are unavailable to continue.

SECTION I

INTRODUCTION

Recent studies of dimensional stability by Dr. Ben Justice of
Corning Glass Works, in collaboration with the National Bureau of
Standards, have stimulated great interest in further, more precise
measurements of this kind. Dr. Justice monitored interferometrically
the thickness of about a hundred 1-in. fused silica and glass-ceramic
blocks and found shrinking that amounted to about one part in 10^6 over
a three-year period. Further evidence of the shrinking of noncrystalline
materials was found by Professor U. E. Hochuli of the University of
Maryland, who slaved a laser to an empty Cer-Vit cavity and compared the
laser frequency with a frequency-stable laser over a three-month period.
His results can be interpreted as a steady shrinkage of two parts in
10^9 per day--a rate not unlike what Dr. Justice observed.

The present program has been initiated, in collaboration with
NBS, Corning, and the Optical Sciences Center, to measure with increased
accuracy the dimensional stability of five materials: Heraeus-Schott
Homosil (vitreous silica), Corning 7940 (vitreous silica), Corning
ULE 7971 (titanium silicate), Schott Zero-Dur, and Owens-Illinois
Cer-Vit C-101. The last three named were chosen because of their useful
ultralow expansion coefficient; Homosil was included because Sakuma,
at the Bureau International de Poids et Mesures, reported great dimen-
sional stability for this material, and this in turn raised the question
of whether Corning's fused silica was any less stable.

1

Late in 1974 Dr. Charles Freed of MIT Lincoln Laboratory provided samples of Universal Cyclops Invar LR-35 and Simonds-Saw Superinvar, which have been included in this study.

In the next section we describe the measurement technique, which is an outgrowth of one previously described (Ref. 1) for ultraprecise measurement of thermal expansion coefficients. Clearly, if one wants to determine temporal behavior, $L(t)$, he must have good knowledge of the thermal behavior $L(T)$ in order to separate this out or keep it negligible. A by-product of our results will be measurements of the stability of optical phase shifts on reflection from multilayer stacks and also dimensional stability of optical contacts.

SECTION II

THE BASIC TECHNIQUE

Very briefly, the basic technique is as follows: A frequency stable laser beam is shined through an optical resonator whose mirrors are spaced by the sample. Tunable optical sidebands are impressed on the stable laser beam (Fig. 1). These are used to track, in the electrical frequency domain, the drift of the cavity resonances caused by heating (Fig. 2). For example, when the cavity temperature is changed an amount ΔT, the subsequent cavity length change ΔL_c is

$$\Delta L_c = -(\Delta \nu / \nu) L_c, \tag{1}$$

where L_c is the cavity length, ν is the optical frequency, and $\Delta \nu$ is the change in cavity frequency.
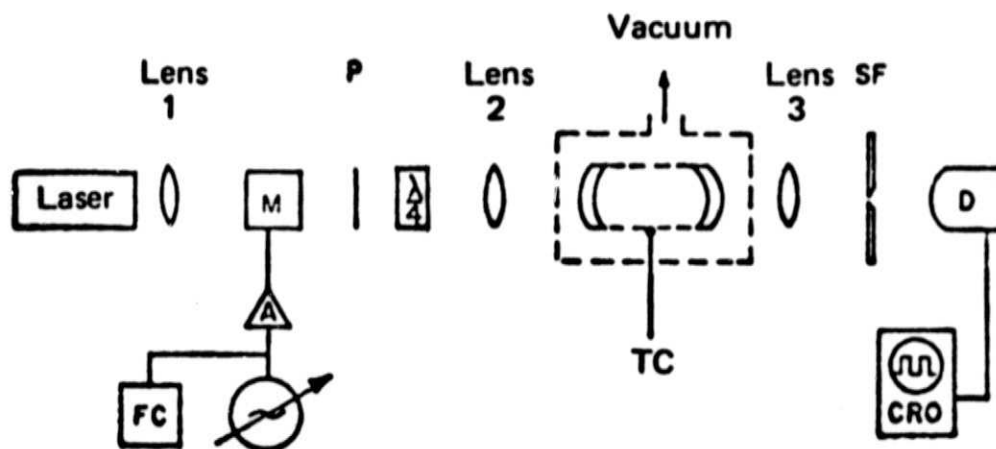

Fig. 1. Schematic diagram of apparatus used to measure thermal expansion coefficients.
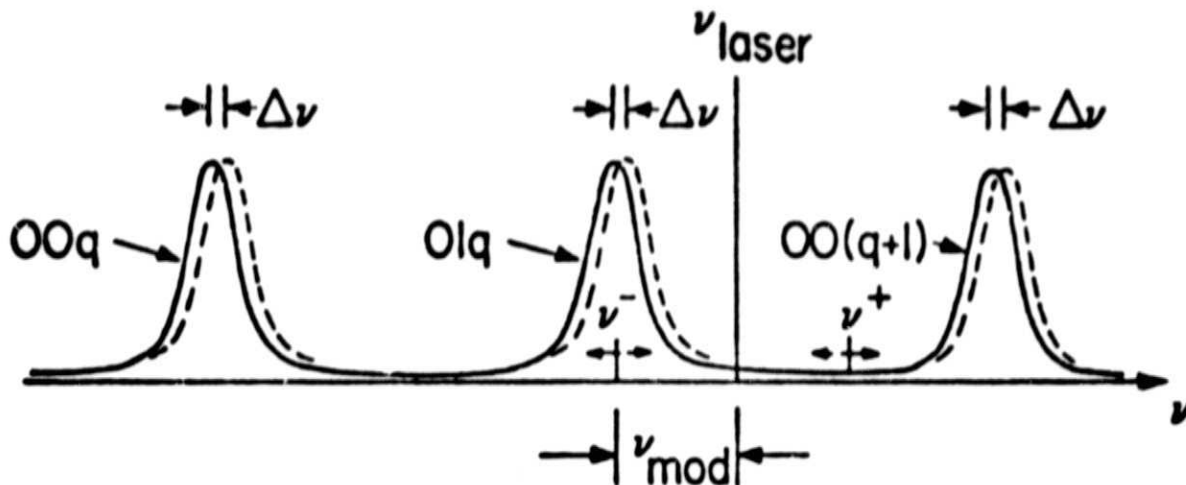
Fig. 2. Fabry-Perot transmission peaks vs frequency for two different temperatures (solid curve and dashed curve). Superimposed are the laser frequency and its sidebands.

Thermal expansion coefficient is measured by recording the change in electrical sideband modulation frequency $\Delta\nu$ necessary to maintain peak transmittance through the resonator after the temperature change ΔT has equilibrated. Then, using Eq. (1),

$$\alpha^- \equiv (1/L)(\Delta L/\Delta T) = -(1/\Delta T)(\Delta\nu/\nu).$$

The ultimate limit to the accuracy of this method is set by the frequency stability $\Delta\nu/\nu$ of the laser (over an equilibrium period of about $1\frac{1}{2}$ hours).

4

SECTION III

APPLICATION TO DIMENSIONAL STABILITY

This Fabry-Perot interferometer technique, with its great inherent accuracy, is applicable to dimensional stability studies. However, it suffers one great disadvantage in this application: If optical phase shifts change with time, they do not cancel, but add instead! It is necessary to build pairs of Fabry-Perot resonators with unequal lengths (Fig. 3), evaluate, and then correct for these phase shifts. This extra information may prove to be a valuable byproduct of the work, perhaps being separable into data on optical contact dimensional stability and optical phase shift stability.
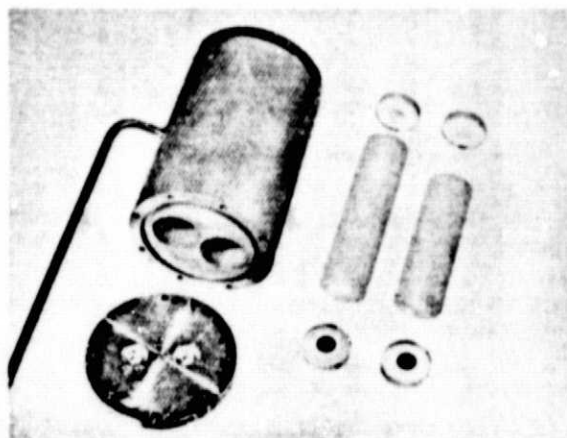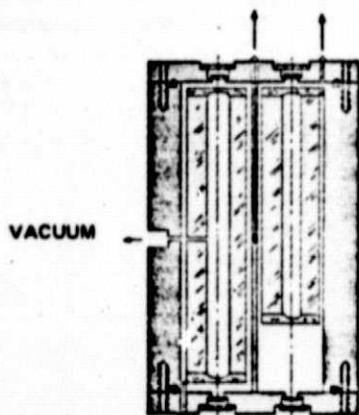

Fig. 3. Unequal-length Fabry-Perot resonators in evacuated copper housing (left), parts shown disassembled in photo.

It turns out that, for volume effects, where $\Delta L \propto L$ (as opposed to surface effects, where changes in optical path length are independent of L), the subsequent resonator frequency shifts $\Delta \nu$ are identical, irrespective of cavity length L. That is, $\Delta \nu_1 = \Delta \nu_2$, where

$$\Delta \nu_1 = -(\Delta L_1 / L_1)\nu$$

$$\Delta \nu_2 = -(\Delta L_2 / L_2)\nu$$

because for volume effects we have assumed

$$\Delta L_1 / L_1 = \Delta L_2 / L_2 = \text{constant.}$$

Therefore, as shown in the appendix, the difference in cavity resonance frequencies $\Delta \nu_2 - \Delta \nu_1$ measures only the nonvolume changes, which we identify as temporal changes in optical phase shift on reflection plus dimensional change in the optical contacts that attach the mirrors to the sample spacer. Figure 4 shows the arrangement for applying the stable laser tracking technique to a multiplicity of long and short sample pairs.
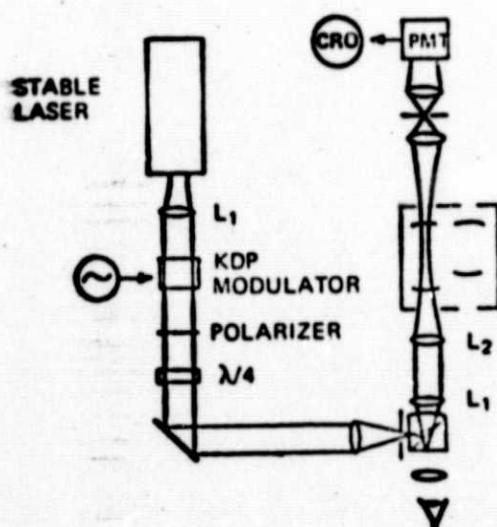

Fig. 4. Arrangement for monitoring cavity resonances of eight pairs of confocal resonators. Apparatus can be translated to each of the 16 stations. L_1 is collimating lens, L_2 is mode matching lens, $\lambda/4$ is quarterwave plate.

6

The following considerations are vital to obtaining meaningful dimensional stability results:

(1) Knowledge of thermal expansion. To be sure that we are observing temporal and not thermal variation of dimensions, we must have accurate measurements of α vs temperature (Fig. 5). For Cer-Vit, Zero-Dur, and ULE, each sample is maintained close to the temperature where its thermal expansion coefficient $\alpha = 0 \pm 1 \times 10^{-9}$ cm/cm°C. Homosil and Corning 7940 have zero expansion at inconvenient temperatures (below -90°C). These materials are maintained at a fixed temperature near 25°C rather than at cryogenic temperatures, which would be costly to maintain and unwise to cycle to.
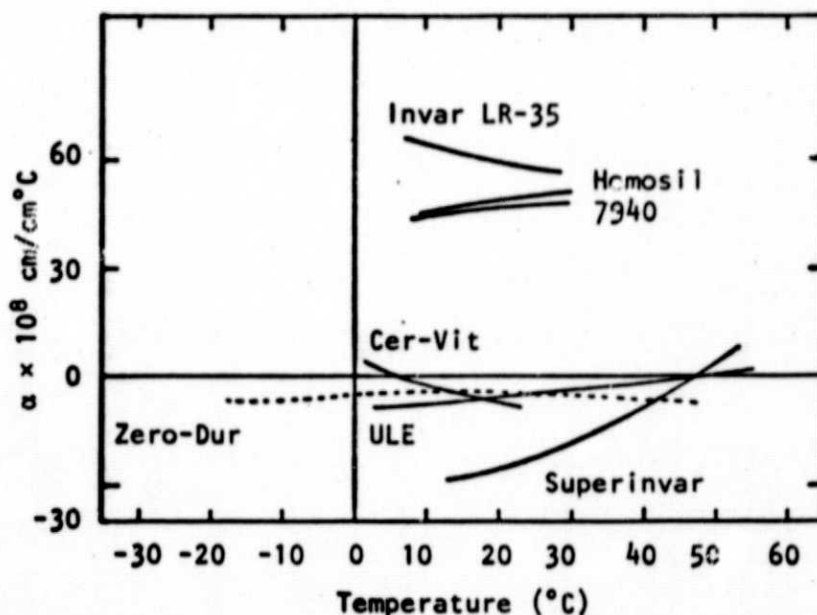
Fig. 5. Thermal expansion coefficients measured at the University of Arizona for dimensional stability materials.

7

2. Temperature control and measurement. It is seen from Fig. 5 that for all the low expansion materials, if the temperature is maintained within $0.02°C$ where α is about 5×10^{-8} cm/cm°C, $\Delta L/L$ due to thermal fluctuations cannot exceed one part in 10^9. This is easily accomplished. However, for the two fused silica samples, where α is about 5×10^{-7} cm/cm°C, it is desirable to always measure at the same temperature within $0.002°C$ in order to ensure that thermal $\Delta L/L$ is no more than one part in 10^9. By taking many measurements we could work with $0.005°C$ temperature control. The selected and aged thermistors being used have demonstrated stability better than 5 mdeg per year. Using these in conjunction with a Rosemount platinum resistance thermometer, we have obtained stability of about 2 mdeg per year.

3. Optical Alignment. The resonant frequencies of a spherical Fabry-Perot (confocal) interferometer depend on the round-trip path length of light through the interferometer. Hercher (Ref. 2) has analyzed this interferometer and shown quantitatively how the resonant frequency depends on what part of the mirrors are used and also on how close to confocal the interferometer is. Hercher's expression (see Fig. 6) for the round-trip phase increment for perfect alignment of a not-quite-confocal cavity is

$$\delta_1 = \frac{2\pi}{\lambda}\left[\frac{\rho^4}{r^3} + \frac{4\varepsilon\rho^2}{r^2} + 4(r + \varepsilon) \right].$$

The same cavity imperfectly aligned has a phase increment

$$\delta_2' = \frac{2\pi}{\lambda}\left[\frac{\rho_1^2\rho_2^2 \cos(2\theta)}{r^3} + 2\varepsilon \frac{\rho_1^2 + \rho_2^2}{r^2} + 4(r + \varepsilon) \right].$$
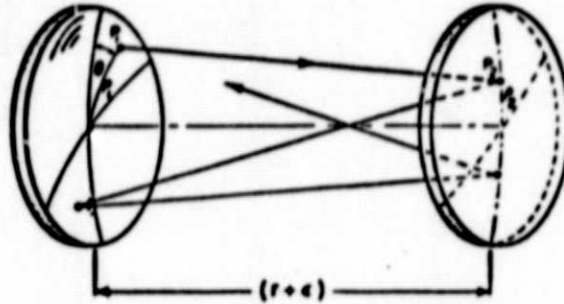
8

Fig. 6. General ray path in a near-confocal Fabry-Perot interferometer. r = mirror radius of curvature, θ = skew angle of entering ray.

In the perfectly aligned case, $\rho = (r\lambda/2\pi)^{\frac{1}{2}} = 0.1$ mm; in the imperfectly aligned case ρ_1 and ρ_2 may be larger. This misalignment can give rise to variation in the resonant frequency. To analyze the worst $(\delta_2 - \delta_1)$ allowable, we set $\rho_1 = \rho_2$, set $\theta = 0$, and solve for ρ corresponding to $\Delta L/L = \Delta\nu/\nu < 10^{-9}$. Thus $\delta_2 - \delta_1 = 8\pi L\Delta\nu/c < 4 \times 10^{-3}$ rad, leading to $\rho_1 = \rho_2 < 0.3$ mm. This means that great care must be exercised to ensure reproducible alignment.

4. **Overall** precision of measurement. All the above considerations indicate that $\Delta L/L \sim 10^{-9}$ is achievable. We must also be able to judge the center of Fabry-Perot peaks to about 0.5 MHz ($\Delta\nu/\nu = 10^{-9}$). This presents no problem, as we previously reported Fabry-Perot fullwidths at half maximum of 2 to 5 MHz for 10-cm cavities. Finally, we must be sure that the long-term (years) frequency stability of our laser is better than one part in 10^9. We are using an iodine-stabilized helium-neon laser whose long-term stability is reported (Ref. 3) to be about two parts in 10^{11}.

5. Sample treatment. To relieve stresses induced in the material in the grinding and polishing processes, we etch each sample in hydro-fluoric acid to a depth of 0.003 in.

IV. EXPECTED RESULTS

In addition to measurements of change in physical length $\Delta L/L$ vs time we expect to obtain considerable data on the combined drift of phase change on reflection lumped with optical contact interface dimensional changes. Because we assume that all the mirrors are identical and therefore will age identically in similar environments (vacuum, near 25°C), the scatter in these measurements will give an estimate of the variance of the optical contact effect.

An auxilliary experiment (Fig. 7), which will take place simultaneously, should yield the magnitude of the average optical contact interface dimensional change. Using Cer-Vit material similar to that in the main experiment we have constructed two cavities of very nearly
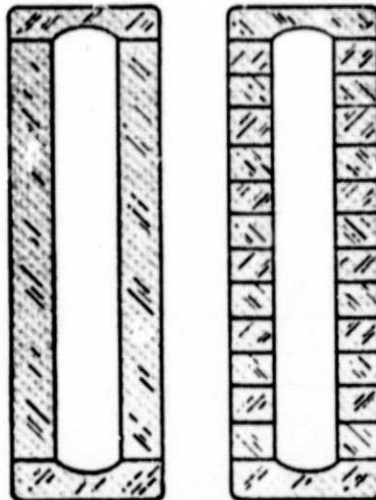
Fig. 7. Experiment to measure average optical contact interface dimensional changes

equal length, one involving only two optical contacts in series, the other involving 12. The difference between their resonant frequencies should be a measure of the cumulative dimensional shrinkage of 10 extra optical contacts. Thus we expect to have, for the first time to our knowledge, an estimate of the magnitude and variation of optical contact dimensional drift.

V. RESULTS

To determine the ambient temperature for each sample and/or to compensate for temperature variations, it is essential to know the thermal expansion coefficient for each sample. These were determined for all samples in this study as shown in Fig. 5.

Figure 8a shows the construction of the temperature-controlled enclosures. Individual temperatures can be maintained within 0.003°C for over three days. All the sample chambers are enclosed in a large freezer (Fig. 8b), which maintains a lower-than-room-temperature environment against which servo-controlled heaters work to maintain a constant temperature.

Temperature measurements are made with platinum resistance thermometers and thermistors. We calibrated these with a water triple point cell obtained from Jarrett Instrument Co. Absolute temperatures can be determined with an accuracy of ±0.001°C for the thermometers and ±0.005°C for thermistors. In general, thermistors are elements that loose their calibration with time. Our thermistors are glass-bead thermistors that were selected by their manufacturer for good stability over a 4-month period. Their drift should not exceed 0.005°C per year.
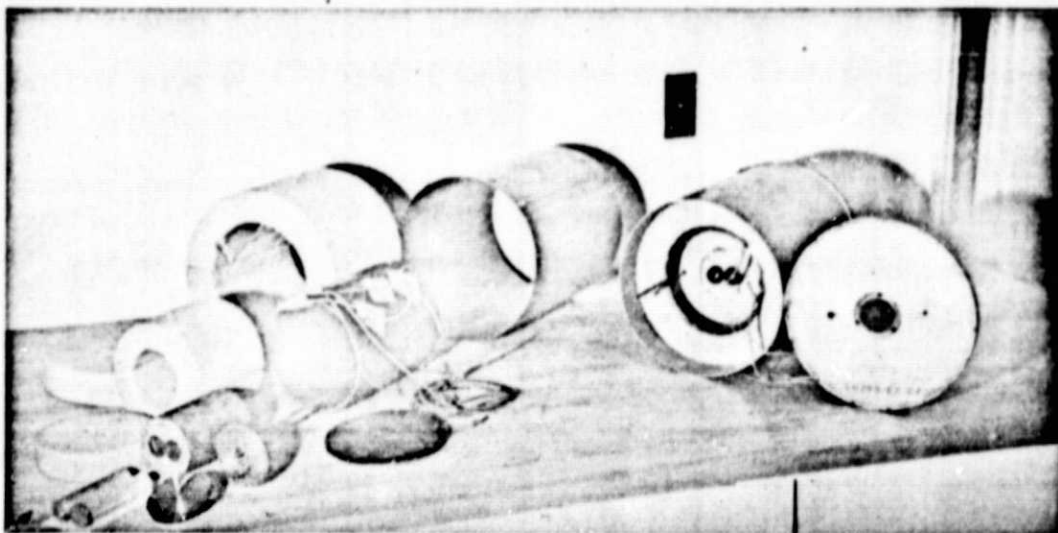
Fig. 8a. Construction of temperature-controlled enclosures. Components are shown at the left. Assembled enclosure is at right.
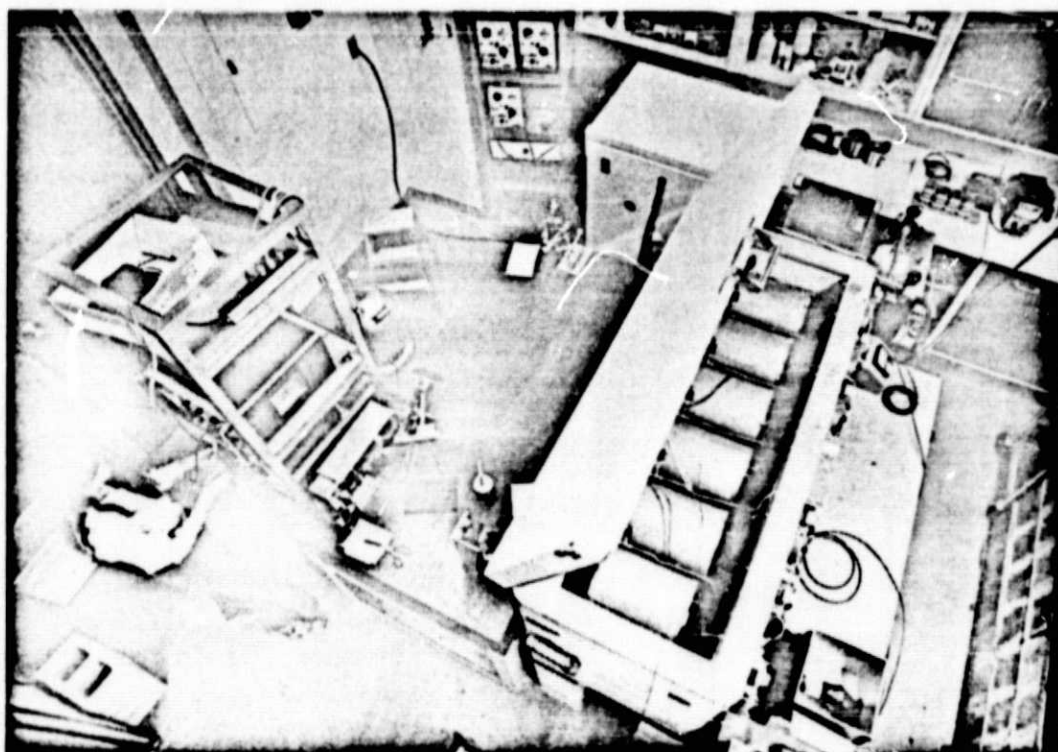

Fig. 8b. Arrangement of experimental apparatus. Freezer with sample chambers can be seen on the right.

VI. PROBLEM AREAS

(1) Alignment. As explained above, optical resonant frequencies have some dependence on optical alignment. This means that variations in our measurements due to optical alignment must be kept small. Considerable time went into developing a technique for aligning the optics to each sample in a sufficiently reproducible way to make possible one part in 10^9 for $\Delta L/L$.

(2) Stable laser. The life of the laser plasma tubes is only about 6 months. We tried to improve this by making an iodine absorption cell with reduced loss, thus prolonging the useful life of a failing laser plasma tube. No appreciable improvement was attained, probably because of impurities (air) in the (radioactive) iodine fill.

(3) Oil contamination. Just as we were ready to put the samples in their evacuated chambers and to commence measurements (Jan. 1975), we realized that oil from our mechanical pump would contaminate our reflective films. We therefore had to rebuild our vacuum system to eliminate all oil pumps. We turned instead to cryogenic pumps.

VII. CONCLUSION

We commenced taking data on June 18, 1975. We now believe that
we can achieve the desired precision of one part in 10^9. Unfortunately,
we have been notified that no continuation funding is now available
from NASA.

APPENDIX

EVALUATION OF $\Delta\phi$ USING THE TWO-CAVITY METHOD

Figure 9 depicts the situation where a frequency-stable laser shines through a long and a short cavity in turn, and the mode structure is monitored with the aid of movable sidebands produced by an electro-optic modulator.
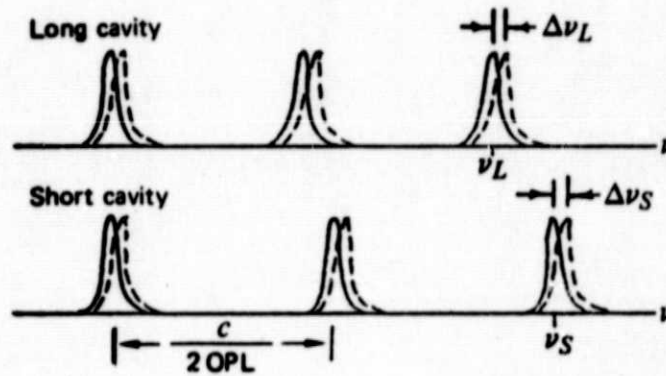

Fig. 9. Frequency-stable laser shining through a long and a short cavity in turn.

After some time has elapsed, the cavity modes will have shifted an amount

$$d\nu/\nu = -d\text{OPL}/\text{OPL},$$

where OPL (optical path length) = $L(t,T) + (\lambda/2\pi)\phi(t,T)$, L is the physical cavity length, and ϕ refers to the optical phase shift on reflection. Thus we obtain

$$d\text{OPL} = (\partial L/\partial T)_t dT + (\partial L/\partial t)_T dt$$

$$+ (\lambda/2\pi)[(\partial\phi/\partial T)_t dT + (\partial\phi/\partial t)_T dt].$$

We cannot separate changes in optical phase shifts due to temperature from those due to time, so we lump both terms together:

$$(\lambda/2\pi)\Delta\phi(t,T) \equiv (\lambda/2\pi)[(\partial\phi/\partial T)_t dT + (\partial\phi/\partial t)_T dt].$$

Furthermore, we include in this term all changes in optical path length that are not proportional to the initial length; i.e., $\Delta\phi(t,T)$ includes not only changes in phase shift on reflection but also changes in optical contact interface separation. We define

$$\alpha_T \equiv (1/L)(\partial L/\partial T)_t$$

and

$$\alpha_t \equiv (1/L)(\partial L/\partial t)_T.$$

Thus

$$\frac{d\nu}{\nu} = -\frac{\alpha_T L_0 dT + \alpha_t L_0 dt + (\lambda/2\pi)\Delta\phi(t,T)}{L_0 + (\lambda/2\pi)\phi(t_0,T_0)},$$

where L_0 is the initial cavity length and $\phi(t_0,T_0)$ is the initial phase shift on reflection. Small changes in time and temperature may be substituted for the differentials in the above expression under the reasonable assumption that α_t and α_T are slowly varying functions of time and temperature over small intervals. Thus we obtain

$$\frac{\Delta\nu}{\nu} = -\frac{\alpha_T \Delta T + \alpha_t \Delta t + (\lambda/2\pi L_0)\Delta\phi(t,T)}{1 + (\lambda/2\pi L_0)\phi(t_0,T_0)}.$$

Measurement of the difference $\Delta\nu_L - \Delta\nu_S$ (the resonance shifts in long and short cavities) makes possible evaluation of the changes in this lumped optical phase shift $\Delta\phi$:

$$\frac{\Delta\nu_L - \Delta\nu_S}{\nu} = -(\alpha_T\Delta T + \alpha_t\Delta t)\left[\frac{1}{1 + \dfrac{\lambda}{2\pi}\dfrac{\phi(t_0,T_0)}{L_L}} - \frac{1}{1 + \dfrac{\lambda}{2\pi}\dfrac{\phi(t_0,T_0)}{L_S}}\right]$$

$$-\frac{\lambda}{2\pi}\Delta\phi(t,T)\left[\frac{1}{L_L\left(1 + \dfrac{\lambda}{2\pi}\dfrac{\phi(t_0,T_0)}{L_L}\right)}\right.$$

$$\left.-\frac{1}{L_S\left(1 + \dfrac{\lambda}{2\pi}\dfrac{\phi(t_0,T_0)}{L_S}\right)}\right].$$

The first term on the right-hand side is negligible within our experimental error, which we take to be smaller than one part in 10^9, as we know that for all the materials

$$\alpha_T\Delta T = \Delta L/L < 10^{-6}$$

in the temperature range of interest. Our experience with $\alpha_t\Delta t$ is more limited. In the worst case of vitreous silica, Dr. Justice measured $\alpha_t\Delta t < 10^{-6}$. We therefore assume that

$$\alpha_T\Delta T + \alpha_t\Delta t < 10^{-5}.$$

We can also be sure that

$$\phi(t_0, T_0) < \pi,$$

so, expanding the product,

$$(\alpha_T \Delta T + \alpha_t \Delta t)(1 - \frac{\lambda}{2L_L} + \ldots - 1 + \frac{\lambda}{2L_S} - \ldots) < 10^{-10}.$$

We conclude that $\Delta \nu_L - \Delta \nu_S$ measures the change of phase shift

$$\Delta \phi(t, T) \simeq \frac{2\pi}{\sigma} \left[\frac{L_L L_S}{L_L - L_S} \right] (\Delta \nu_L - \Delta \nu_S).$$

L_L and L_S can be determined to one part in 10^5 by measuring $\sigma/2L$ using sidebands for probes. This means that $\Delta \phi$ can be measured to that accuracy.

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