

## CHANGES IN SURFACE FIGURE DUE TO THERMAL HYSTERESIS

*S. F. Jacobs, S. C. Johnston, J. M. Sasian,  
M. Watson, J. D. Targove, and D. Bass*

Optical Sciences Center  
University of Arizona, Tucson Arizona 85721

### ABSTRACT

Thermal cycling hysteresis affects surface figure in low-expansivity mirror substrates. Zerodur, ULE, and Cer-Vit 8-in.-diameter mirrors and dilatometer samples were thermally cycled at uniform rates of 6 K/hr and 60 K/hr, and somewhat faster for nonuniform heating. Figure distortions as large as  $\lambda/10$  were observed following nonuniform heating of standard Zerodur, which was the only material exhibiting thermal hysteresis. A new experimental Zerodur appears to be free of this problem.

### INTRODUCTION

Figure distortion as a result of thermal cycling of ULE and Zerodur was investigated interferometrically by Bennett et al., who reported a permanent surface figure change of more than  $\lambda/10$  in Zerodur after nonuniform heating and cooling.<sup>1</sup> In a parallel effort, our group observed thermal hysteresis effects in Zerodur dilatometer samples.<sup>2</sup>

We believe that hysteresis (failure of the material to return to former dimensions) causes figure distortion, provided the heating is spatially nonuniform. If heating is uniform, then isotropic thermal hysteresis in the material should not result in significant surface figure change (unless, of course, initial stress is present which is released by the cycling process), but isotropic scale change will occur. Nonuniform heating, on the other hand, can result in permanent figure change, since distortion caused by nonuniform heating does not completely relax upon cooling.

In the present work we investigate standard Zerodur, ULE, Cer-Vit, and experimental Zerodur, utilizing both interferometry and dilatometry. Using interferometry we find, when heating is uniform, no significant figure distortion occurs. When heating is nonuniform and hysteresis is present, figure distortion occurs; however, no significant figure distortion occurs when hysteresis is absent. Using dilatometry, we find that only standard Zerodur exhibits significant thermal hysteresis. Our nonuniform heating regime is similar to those used in batch coating procedures. These findings are especially relevant to directed-energy applications.

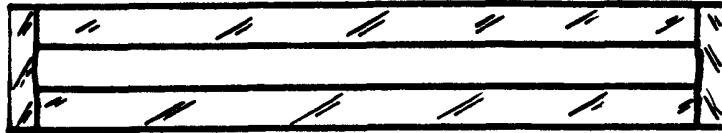
Hysteresis in Zerodur is attributed by its manufacturer to the presence of MgO, which is deliberately included for its beneficial effect on viscosity at the melting temperature and on obtaining low thermal expansivity.<sup>3</sup> Thus MgO helps achieve uniformity, but causes hysteresis. Schott Glaswerke has developed a modified glass-ceramic with properties almost identical to those of Zerodur, but without the hysteresis effects. This experimental material has been included in the present investigation.

Two additional questions addressed in this study are: 1) how the thermal cycling rate affects hysteresis, and 2) whether repeated thermal cycling reduces hysteresis. We find that hysteresis effects are more severe at fast cycling rates, and that repeated cycling does not alter the hysteresis behavior, but can remove residual stress.

## SAMPLE MATERIALS AND PREPARATION

Each of the following materials was obtained in two shapes: 8-in.-diameter discs 1-1/2-in. thick, and 4-in.-long cylinders of 1-1/4-in. diameter, which were made into etalons for dilatometer studies (see Figure 1).

1. Standard Zerodur, polished at the Air Force Weapons Laboratory (AFWL).
2. Standard Zerodur, polished by the Perkin-Elmer Corporation using a proprietary method designed to reduce distortion upon thermal cycling.
3. Experimental Zerodur, polished at the Optical Sciences Center (OSC).
4. Cer-Vit 101, polished at OSC.
5. ULE (Corning Type 7971) polished at OSC.



*Figure 1. Sample/etalon configuration for dilatometer studies.*

After all rough grinding and boring operations, each dilatometer sample prepared at OSC was stress-relieved by immersion in 30 HF acid for three minutes. Care was taken with these samples to avoid thermal shock during optical working (e.g., minimal use of heat in fastening samples during grinding and polishing). Scratch marks were not used for identification, as they can introduce stress.

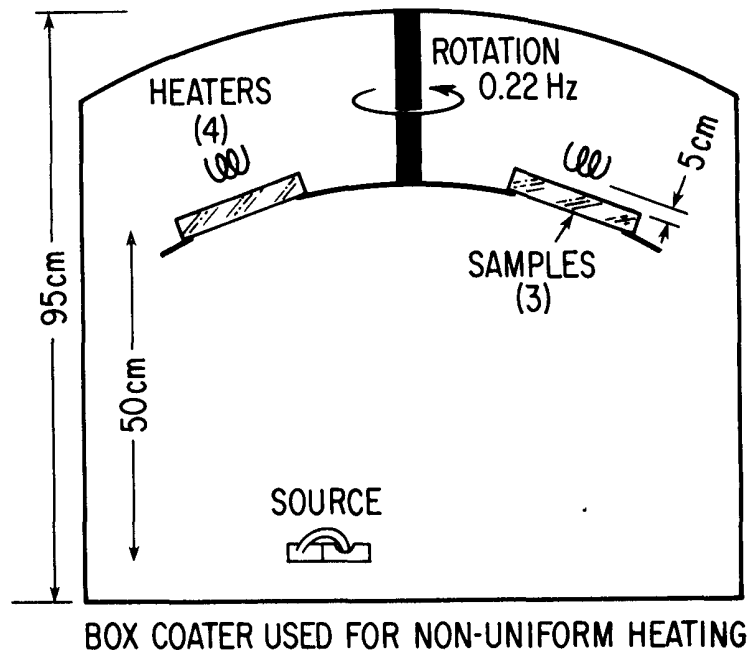
All 8-in.-diameter discs were polished flat to  $\lambda/4$  on both surfaces. Prior to surface figure measurements, each surface under test was chemically spray-silvered to improve measurement contrast and to avoid stray-light-interference effects from the unused sample surface. The thin silver coating was chemically removed before thermal cycling and then reapplied before surface remeasurement.

A major requirement that must be satisfied by a spray-coated film is that the nonuniformities of film thickness must be smaller than the thickness noise level of the measurement system. Reproducibility of data for different films on the same substrate indicated that this requirement was satisfied.

## THERMAL CYCLING AND SURFACE FIGURE MEASUREMENTS

During the uniform heat cycling process, one 8-in. disc of each type was temperature cycled, supported on edge, in an oven at the University of Arizona Mirror Laboratory. The discs were cycled from 300 K to 475 K and back to 300 K, at slow and fast rates of 6 K/hr and 60 K/hr respectively, with a one hour hold at the maximum temperature. A different set of five 8-in. discs was used for each cycling.

Nonuniform heating simulated the conditions of an accelerated coating cycle except that no coating was applied. The 8-in. discs were mounted, three at a time, in a Balzers BAK 760 Coater. Radiant heat was incident on the backside of each sample as it rotated past the heat source. Figure 2 shows the mounting geometry. From other experiments we were able to estimate the temperature of the samples. The temperature was cycled from 300 K to 475 K in one hour; it was held at 475 K for four hours, and then reduced to 300 K over a period of eight hours.



*Figure 2. Box coater used for nonuniform heating.*

To characterize the sample surfaces, a real-time Twyman-Green phase-shifting interferometer (RTI) was used (see Figure 3). During the early stages of this study, it became clear that in order to achieve data reproducibility, the measurement system had to be isolated: vibrations of the fringe pattern, fringe drifting, and fringe distortion could be readily observed in the real-time interference pattern. These vibrations were caused primarily by building vibration and sound, system temperature changes, and air turbulence.

To overcome these disturbances, the 4-ton granite table supporting the test setup was air cushioned. In addition, the masses of both the interferometer and the auxiliary optical-element mounts were augmented to decrease their natural resonance frequency. To reduce thermal effects, a thermal-insulation styrofoam enclosure was constructed to cover the entire test setup. This also improved the air stability in the optical testing path. The test instruments were left on throughout the test period, the testing room conditions were kept the same, and testing was always conducted at the same time of day. As a result of these precautions, a notable improvement in fringe stability was obtained, leading to  $\lambda/50$  rms data reproducibility.

A reference mirror was incorporated to monitor system performance and verify that for each measurement the test configuration remained the same (see Figure 3). The effective aperture diameter was approximately 7 in., as edge diffraction effects introduced spurious data, leading us to ignore the outermost rim of the mirror.

Thirty data sets were taken for each sample before and after heat treatment. Each RTI data set provided a surface contour. Averages were then computed and the difference (before vs. after) of those averages was obtained to determine the change in optical figure. This change is presented as a surface contour for each sample in the right-hand column of Figures 5 through 7, and the number given represents the rms surface figure change in waves of 633 nm. The data processing was accomplished with WISP software from the WYKO Corporation. The results are listed below.

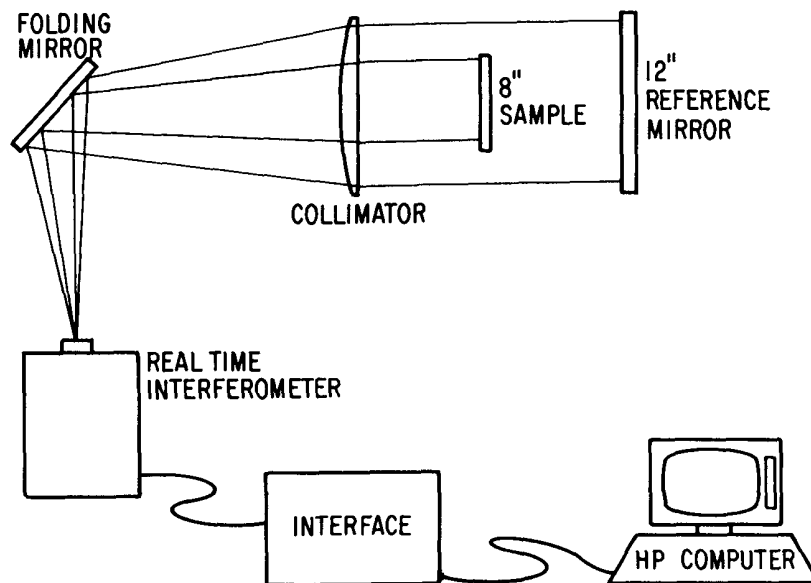


Figure 3. Real-time interferometer arrangement.

1. Uniform heating - 60 K/hr (Figure 5)

The 8-in. discs were thermally cycled 300-475-300 K in steps of 60 K/hr to determine figure changes. Our conclusion is that 60 K/hr uniform heating caused no significant change in surface figure.

2. Uniform heating - 6 K/hr (Figure 6)

In all cases but one, we observed less distortion at 6 K/hr than at 60 K/hr, as might be expected. The exception was ULE, which showed astigmatic distortion. However, because of the excellent ULE fast-cycling behavior, as well as previous experience with ULE (ours and Bennett's) we believe that this sample probably contained unusual stress.

3. Nonuniform heating (Figure 7)

The results were striking. Both standard Zerodur samples (AFWL and Perkin-Elmer) showed significant distortion (mainly a change in power), while the other materials did not. (The experimental Zerodur showed no significant distortion.)

### DILATOMETER MEASUREMENTS

Sample configuration is shown in Figure 1. Polished, highly reflective end mirrors were optically contacted to each end of the sample, forming a confocal Fabry-Perot resonator. Each sample/etalon was then mounted sequentially in an evacuated copper chamber and a tunable HeNe laser beam was frequency locked to a cavity resonance. As shown schematically in Figure 4, part of the tunable laser beam was split off to compare (beat) it with a stabilized reference laser beam, in order to monitor changes in cavity resonant frequency. As the sample length  $L$  changed by an amount  $\Delta L$ , the cavity resonant frequency  $\nu$  changed by an amount  $\Delta\nu = (\nu/L)\Delta L$ , which was sensed electrically as a change in the beat frequency. Thus a tiny length change caused a large change in beat frequency, enabling convenient measurement.

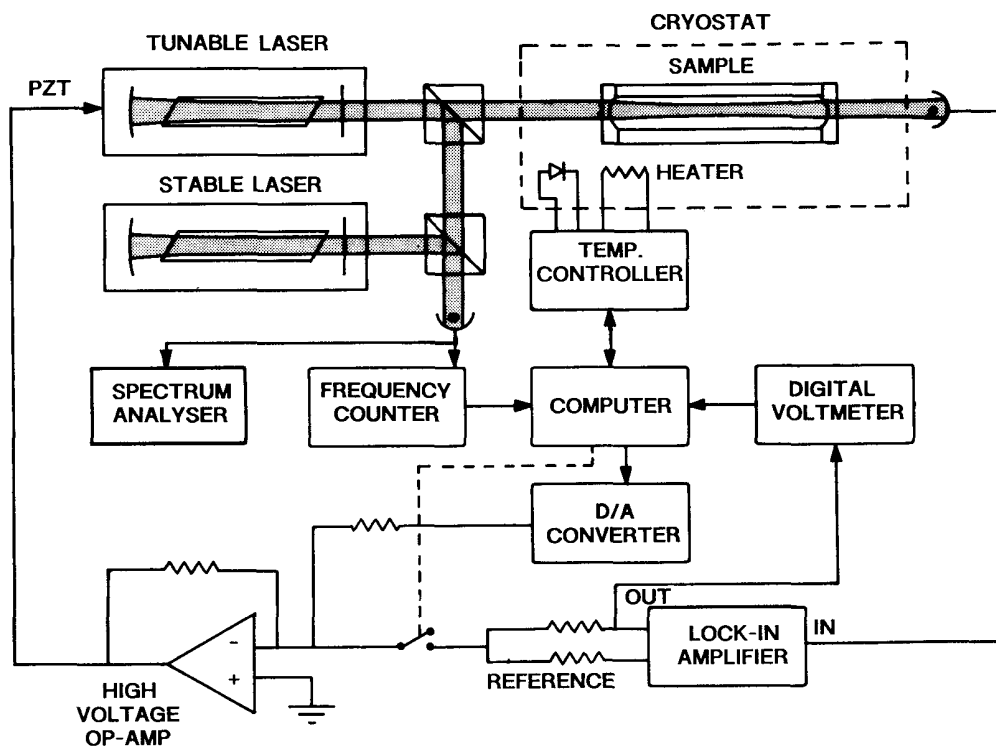


Figure 4. Laser-interferometric dilatometer arrangement.

The limiting measurement error in  $\Delta L/L$  is set for samples of low expansivity by the stability of the reference laser, which is better than  $10^{-9}$ . For samples whose thermal expansivity is not low, the accuracy is limited by temperature measurement and control:  $\Delta L/L = \alpha \Delta T$ . In our apparatus the error in  $\Delta T$  is approximately 0.01 K.

The general procedure was to thermally cycle, in 25 K steps, from 300 K to 475 K and back to 300 K. The rate of temperature change was controlled by an HP-85 computer and a Lakeshore DRC-81-C temperature controller. To reach thermal equilibrium, the system time constant was about three hours.

The central columns of Figures 5 through 7 show that only standard Zerodur exhibited marked hysteresis, leading to the conclusion that Schott Glaswerke has indeed eliminated hysteresis in its new experimental material. Concerning hysteresis in Zerodur cycled quickly vs. slowly, we found the magnitude of the effect was less in the slowly cycled case.

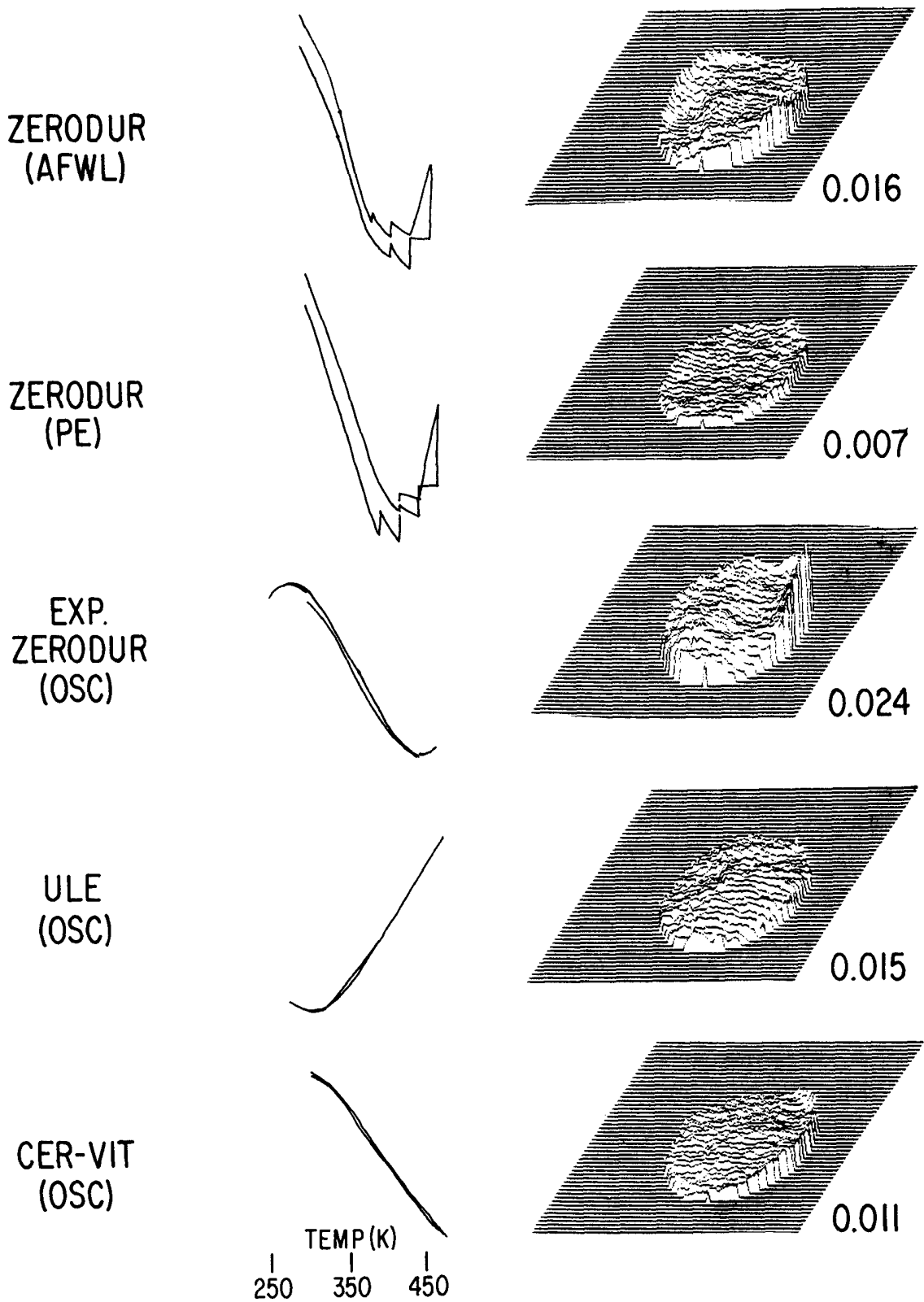


Figure 5. Uniform heat 60 K/hr: dilatometer samples (length vs. temperature) and surface figure change (units of  $\lambda_{rms}$ ).

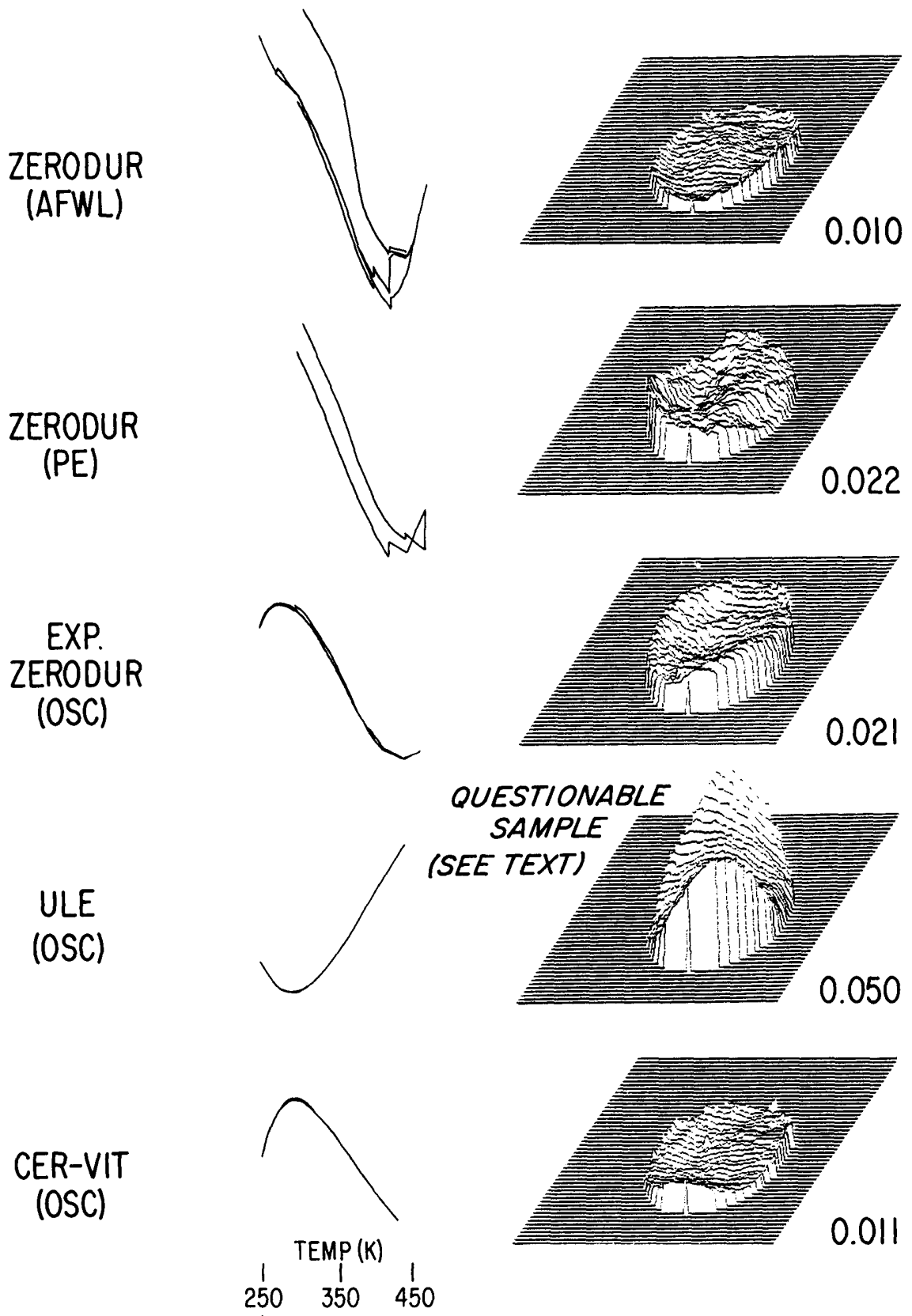


Figure 6. Uniform heat 6 K/hr: dilatometer samples (length vs. temperature) and surface figure change (units of  $\lambda_{rms}$ ).

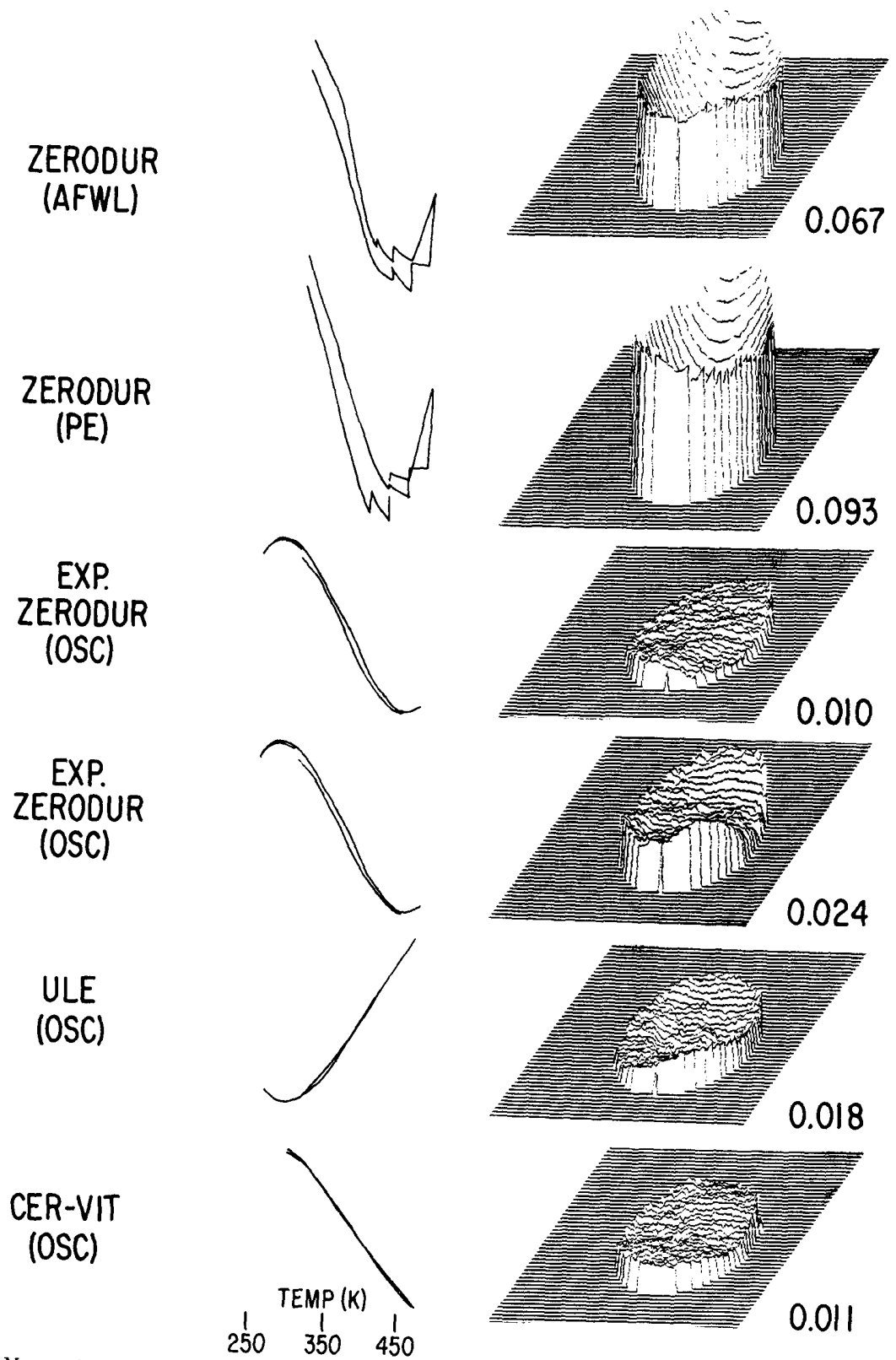


Figure 7. Nonuniform heat: dilatometer samples (length vs. temperature) and surface figure change (units of  $\lambda_{rms}$ ).



As the data for Zerodur (AFWL) cycled at 6 K/hr showed a large amount of change after one thermal cycling, we selected this sample for detailed study to investigate the effects of repeated thermal cycling. Referring to Figure 8, a number of features are noteworthy:

In temperature ranges where hysteresis occurred, waiting times became much longer than those in nonhysteresis ranges. The first temperature rise traced a path which was never again repeated. Subsequent cycling resulted in a fairly repeatable path which included hysteresis. Waiting times on the first temperature rise were unusually long for a nonhysteresis temperature region. Our interpretation of this preliminary behavior is that this particular sample contained some internal stress, probably as a result of thermal shock during optical working, the effects of which were removed by thermal cycling.

### SUMMARY AND CONCLUSIONS

This investigation has successfully answered several questions regarding the best of the low-expansion mirror materials presently available. The investigation has demonstrated quantitatively the relation between hysteresis and figure distortion, and has extended the earlier findings of Bennett et al. In addition, the present results should prove useful as guidelines in the manufacture of improved dimensionally stable materials.

To summarize our findings:

1. Cer-Vit and ULE exhibited no significant hysteresis figure distortion under the conditions of this study, even though the Cer-Vit showed obvious stress birefringence upon crossed-polaroid examination.
2. In standard Zerodur, hysteresis appeared to be responsible for surface figure distortion (about  $\lambda/10$  rms focus error across 7 in.). Additional strain (several ppm) was sometimes present, and was eliminated by thermal cycling. This strain may have been due to thermal shock or machining procedures.
3. The new developmental Zerodur was virtually free of hysteresis and surface figure distortion under the conditions of these tests.
4. Concerning effect of rate of cycling on hysteresis, cycling at 60 K/hr caused more severe hysteresis than cycling at 6 K/hr in standard Zerodur.
5. Uniform heating (300-475-300 K) caused no significant surface deformation in any of the materials studied here; however, nonuniform heating did cause surface deformation in materials which exhibit hysteresis. This agrees with a simple model of thermal relaxation. Bennett et al. found that standard Zerodur became distorted when uniformly heated to 250°C (523 K) and 300°C (573 K) and then quenched (rapidly air-cooled). The present results show that distortion can occur at even lower temperatures ( $\sim 450$  K, where hysteresis sets in), but it is due to nonuniformity of heating. This suggests it was quenching that introduced nonuniformity into the cooling process in the Bennett study.

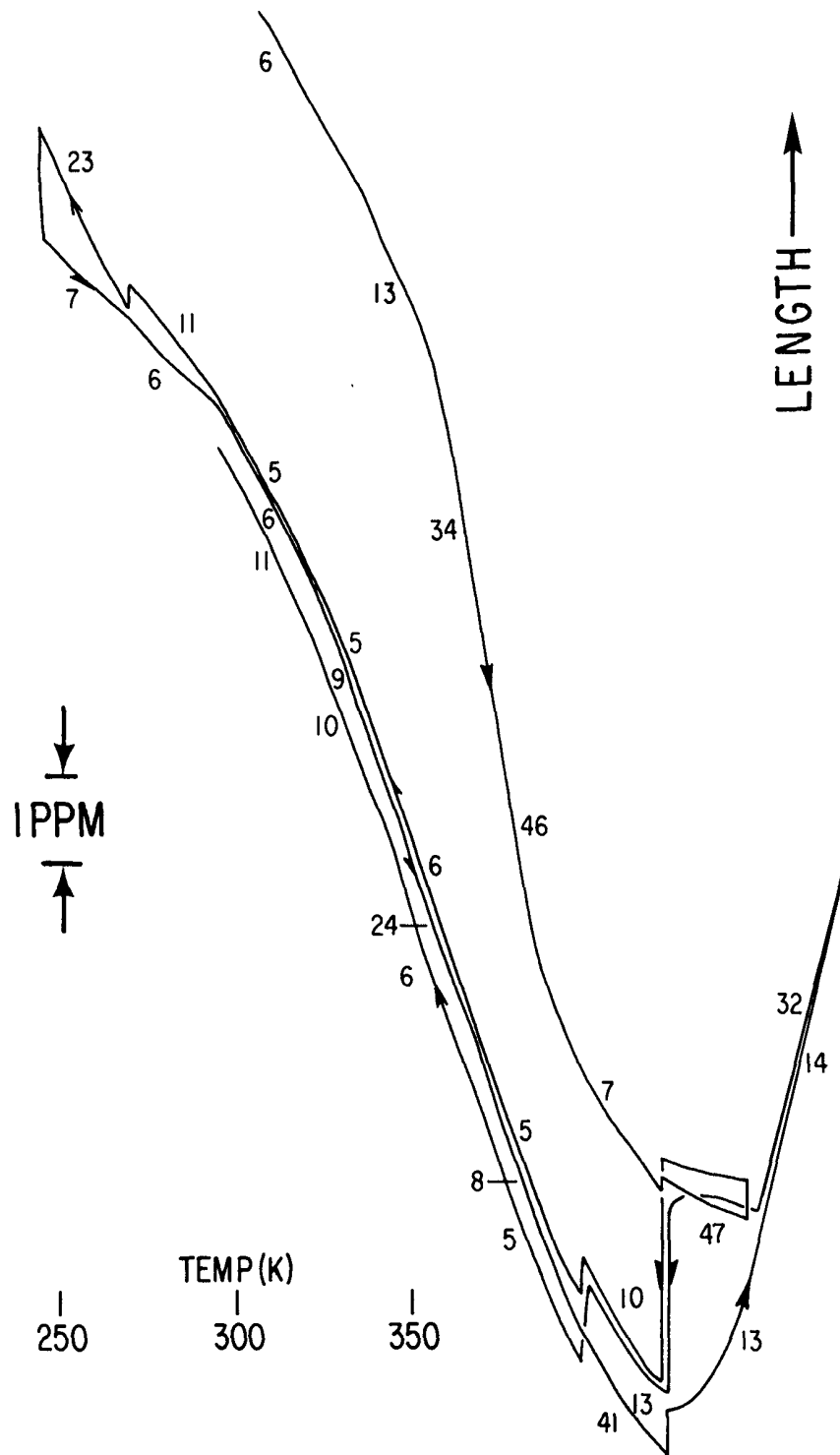


Figure 8. Details of Zerodur dilatometer measurements (length vs. temperature). Numbers indicate waiting times in hours.

## RECOMMENDATIONS FOR FUTURE WORK

Uniformity of thermal expansion is as important as low thermal expansion in the performance of a telescope mirror substrate. For this reason, the results presented here regarding the Schott experimental material should be followed up with evaluation of its thermal expansion uniformity. Since the new experimental Zerodur was presumably produced by altering the MgO content, and since the role of MgO in Zerodur is to reduce viscosity to facilitate thermal expansion uniformity, there is reason for concern regarding this aspect of the new material.<sup>4</sup> Other characteristics which should be evaluated for the new material are its expansivity at low temperatures and whether there is still hysteresis at low temperatures ( $\sim 250$  K) as Lindig et al. have postulated.<sup>2,3</sup>

## ACKNOWLEDGMENTS

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