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EFFECT OF THERMAL CYCLING

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The objective of this effort is to evaluate the stability of low expansion Zerodur, developmental Zerodur, ULE, and Cer-Vit as possible substrate materials for high energy laser mirrors. This effort will determine whether there is instability in developmental Zerodur, ULE and Cer-Vit over operating temperatures and coating temperatures (300-475K). Zerodur has already been shown to exhibit instability. Thermal cycling will be investigated as a possible approach to eliminate or reduce hysteresis. The effect of polishing on hysteresis will also be investigated.

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I. INTRODUCTION

The objective of this effort is to evaluate the stability of low expansion Zerodur, ULE, and Cer-Vit as possible substrate materials for high energy laser mirrors. This effort will determine whether there is instability in ULE and Cer-Vit over operating and coating temperatures (300 to 475 K). Zerodur has already been shown to exhibit instability (Refs. 1 and 2). Thermal cycling will be investigated as a possible approach to eliminate or reduce hysteresis. The effect of polishing on hysteresis will also be investigated.

Background

Figure 1 summarizes the low thermal expansivity properties of Zerodur, ULE, and Cer-Vit. Because of the importance of these materials it is essential that we understand their limitations in order to extend their usefulness and perhaps assist in improving their manufacture.

In 1984, Bennett et al. found that thermal cycling caused permanent deformation in a Zerodur mirror (Ref. 1) when it was heated above 500 K and rapidly cooled in air. Also at that time Jacobs et al. showed (Ref. 2), using laser interferometry, that Zerodur dilatometer samples exhibit hysteresis not only at high temperature (~450 K) but at low temperature (~250 K) upon thermal cycling. In 1985 there was published (Ref. 3) a discussion by the manufacturer of Zerodur in which the cause of Zerodur hysteresis at the higher temperature is attributed to the presence of MgO, deliberately included for its beneficial effect on the viscosity at the melting temperature as well as on obtaining low thermal expansivity. Thus, the presence of MgO aids in achieving uniformity, but at the same time it causes unwanted hysteresis. The article goes on to say that a modified glass ceramic with almost identical properties to those of Zerodur but without the relaxation effects (between 130 and 320°C) has been developed. This experimental Zerodur has been included in the present investigation.

Although inferred in the past, it is of interest to definitely establish the relation between the observed hysteresis and surface deformation. By hysteresis we mean failure to return to former length upon thermal cycling. A simple model suggests that with uniform heating and cooling very little permanent deformation should occur in surface figure, while for non-uniform heating there should be a deformation which does not fully disappear due to the material's failure to fully return to length.

In addition to correlating permanent figure distortion (due to nonuniform heating) with hysteresis (observed with uniform heating) we investigate

* whether ULE and Cer-Vit exhibit hysteresis effects,
* whether Schott's new experimental (developmental) Zerodur has reduced hysteresis,
* how thermal cycling rate and temperature uniformity affect hysteresis,
* whether (multiple) cycling reduces hysteresis, and
* whether a special polishing procedure developed by the Perkin-Elmer Corporation is effective in reducing Zerodur hysteresis.

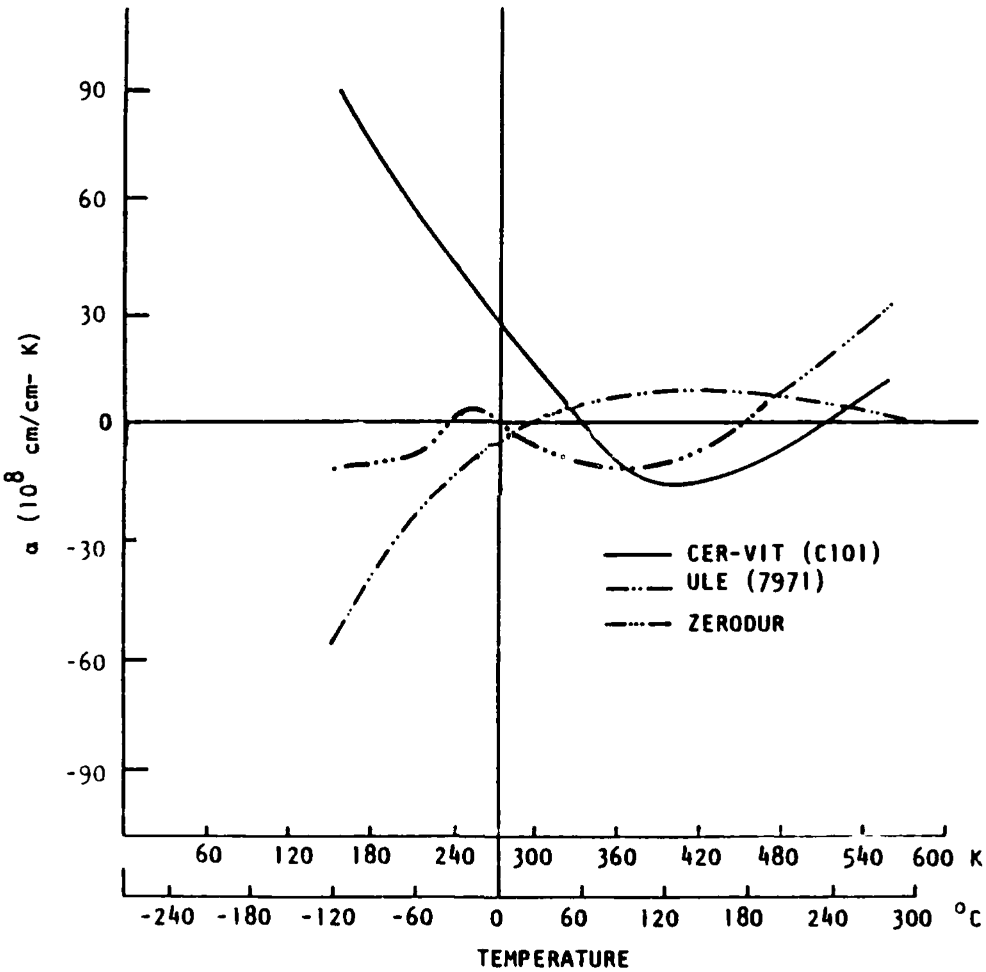


Fig. 1. Thermal expansivity versus temperature for Zerodur, ULE, and Cer-Vit.

II. SAMPLE MATERIALS

Samples investigated are listed below.

1. Standard Zerodur, manufactured by Schott and polished by AFWL. We call this "Zerodur (AFWL)" or Z_{AFWL}.
2. Standard Zerodur, manufactured by Schott and polished at Perkin Corporation by a proprietary method designed to reduce figure distortion upon thermal cycling. We call this "Zerodur (PE)" or Z_{PE}.
3. Experimental (developmental) Zerodur, prepared by Schott for reduced hysteresis and reduced distortion upon thermal cycling and polished at Optical Sciences Center. We call this "Developmental Zerodur (OSC)" or Z'_{OSC}.
4. Cer-Vit, manufactured by Owens-Illinois Co., obtained through US Naval Weapons Center. We call this "Cer-Vit (OSC)" or C_{OSC}.
5. ULE (Corning Type 7971) polished at Optical Sciences Center. We call this "ULE (OSC)" or ULE_{OSC}.

Each material was supplied in two shapes: 8-in.-diam × 1 1/2-in. discs, to be made into polished mirrors for surface figure studies, and 4-in. long × 1 1/4-in.-diam cylinders, to be made into etalons for dilatometer studies. Figure 2 shows the etalon configuration.

The first two materials were government furnished equipment, while the remaining material was obtained with the understanding that each supplier would be informed of the experimental results. The original plan was to obtain sufficient material so that figure distortion measurements could be performed with threefold redundancy; e. g., three discs cycled rapidly (60 K/hr) and three discs cycled slowly (6 K/hr). As it turned out, only two Zerodur discs (and four dilatometer samples) were received from Perkin-Elmer, polished by their proprietary method. Schott supplied three discs of experimental Zerodur, as did Corning and Owens-Illinois. The program thus had to proceed with the material available, which meant foregoing the redundancy and going with single samples for each heat treatment.

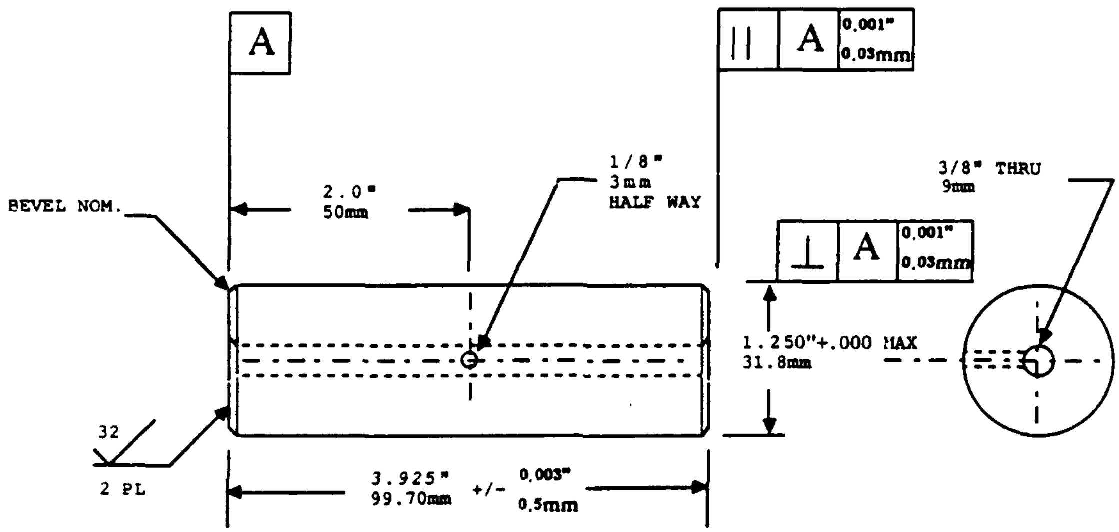


Fig. 2. Dilatometer sample configuration.

III. SAMPLE PREPARATION

After all rough grinding and boring operations, each dilatometer sample prepared at Optical Sciences Center was stress relieved by immersion in 30 percent hydrogen fluoride acid for 3 min. Care was taken with these samples to avoid thermal shock in the process of optical working (e.g. minimal use of heat in fastening samples during grinding and polishing). Care was also taken not to use scratch marks for identification purposes, as this can introduce strain.

All 8-in -diam discs were polished $\lambda/4$ on both flat surfaces. Prior to surface figure measurements each surface under test was chemically spray silvered to improve measurement contrast. To preserve the excellent surface finish from chemical degradation, the thin (~5 nm) silver coating was removed before thermal cycling and then reapplied before surface remeasurement.

Spray silvering is a simple process to increase the reflectivity of a glass substrate. The main advantages over an evaporation process are that it can be done in a comparatively short period of time, no heat or ion bombardment treatments are required, and the cost is low. The important requirement to meet by a spray coated film is that the nonuniformities of the film thickness must be smaller than the thickness noise level of the measurement system. Reproducibility of data for different films on the same substrate justify the choice of spray silvering as the coating process preferred for this study.

IV. THERMAL CYCLING

Uniform Heating

The 8-in discs were temperature cycled, supported on edge, in an oven at the Mirror Laboratory. The two cycling runs were controlled to increase temperature from room temperature and to decrease temperature at 6 and 60 K/hr, respectively with 1 hour holds at the maximum temperature (475 K).

Nonuniform Heating

Nonuniform heating was done simulating the conditions of a standard coating cycle except that no coating was applied. The 8-in. discs were mounted, three at a time, in a Balzers BAK 760 Box Coater. Radiant heat was incident from the backside of each sample as it rotated past the heat source. Figure 3 shows the mounting geometry.

Temperature was cycled from room temperature to 475 K in 2 hours. Then it was held for several hours before cooling back to room temperature in 2 hours.

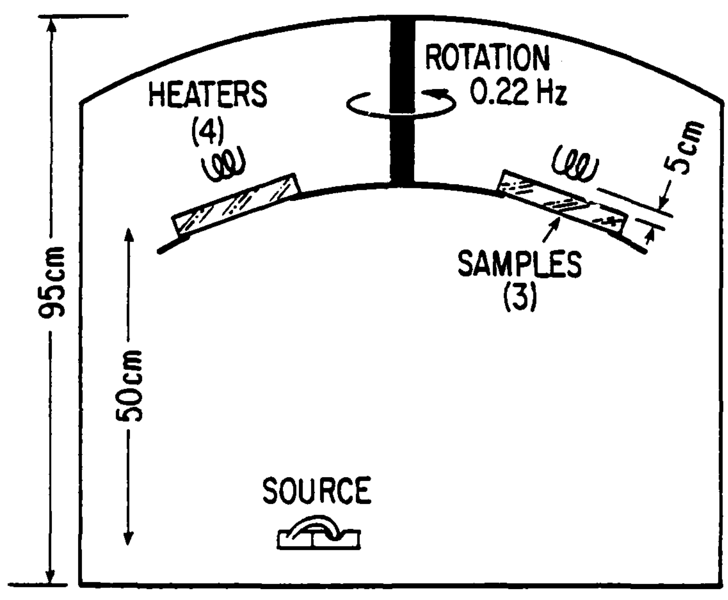


Fig. 3. Box coater used for nonuniform heating.

V. DILATOMETER MEASUREMENT METHOD

Samples were fashioned into hollow cylinders with parallel ends and a hole down the symmetry axis. Polished, highly reflecting end-mirrors were attached, optically contacted to each end, forming a Fabry-Perot resonator. The sample/etalon was then mounted 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 to monitor changes in cavity resonant frequency. As the sample length L changed by an amount ΔL, the cavity resonant frequency ν 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 causes a large change in beat frequency, which can be measured conveniently.

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 ΔT is about 0.01 K.

The general procedure was to thermally cycle, in 25 K steps, from room temperature up to 475 K and back to room temperature. The temperature rate of change was computer-controlled by an Hewlett Packard 85 computer and Lakeshore DRC 81 C temperature controller. To reach thermal equilibrium the system's normal time constant was about 3 hours. However, for Zerodur, long waiting times are associated with the hysteresis phenomenon. To show this, we made a detailed study of one sample in which we recorded these waiting times. We also repeated the cycling to determine whether repeated cycling can reduce the hysteresis.

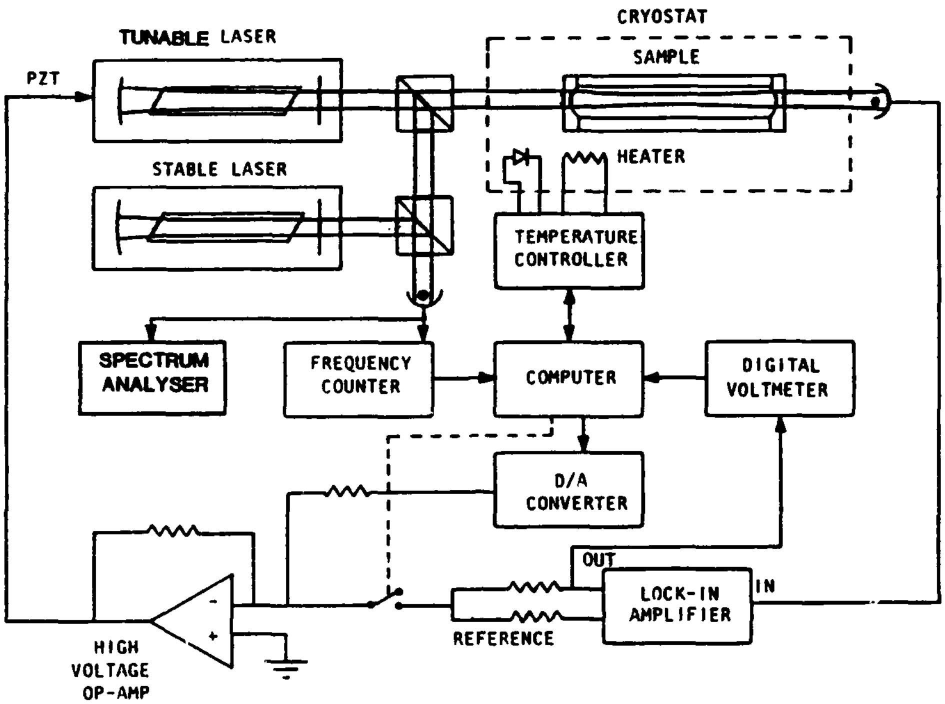


Fig. 4. Laser-interferometric dilatometer arrangement.

VI. DILATOMETER RESULTS

Repeated cycling of standard Zerodur

As the data for "Zerodur 6 K/hr" showed unusually poor reproducibility after one thermal cycling, this sample was selected for detailed study to investigate what is and is not improved by repeated thermal cycling. Referring to Figure 5, the numbers indicate what Schott calls the waiting time, in hours, for each 25 K equilibrium increment. A number of features are noteworthy.

The first temperature rise traces a path which is never again repeated. Subsequent cycling results in a fairly repeatable path *which includes hysteresis.* Interpretation of the altered preliminary behavior is that this particular sample had some strain, probably from thermal shock during optical working, whose effects were removed by thermal cycling. We believe this because measurements of another sample, "Zerodur 60 K/hr", showed much better reproducibility, despite its faster cycling which would be expected to cause worse hysteresis. Note that waiting times were unusually long for a nonhysteresis temperature region. This behavior may be associated with annealing out of strain.

Slow versus fast cycling

A. Zerodur (AFWL), 6 and 60 K/hr (Figs. 6a and 7a). The slower cycling was discussed previously. Note that the faster cycled sample has better reproducibility (less strain) but worse hysteresis. ("Sawtooth" peak-to-valley depth is larger).

B. Zerodur (PE), 6 and 60 K/hr (Figs. 6b and 7b). Both show presence of hysteresis. Hysteresis is more pronounced in the faster cycled case. In both cases there is about 1 ppm of displacement (released strain) on the return to room temperature.

C. Developmental Zerodur, 6 and 60 K/hr (Figs. 6c and 7c). Only the slightest trace of hysteresis.

D. ULE, 6 and 60 K/hr (Figs. 6d and 7d). No trace of hysteresis.

E. Cer-Vit, 6 and 60 K/hr (Figs. 6e and 7e). No trace of hysteresis.

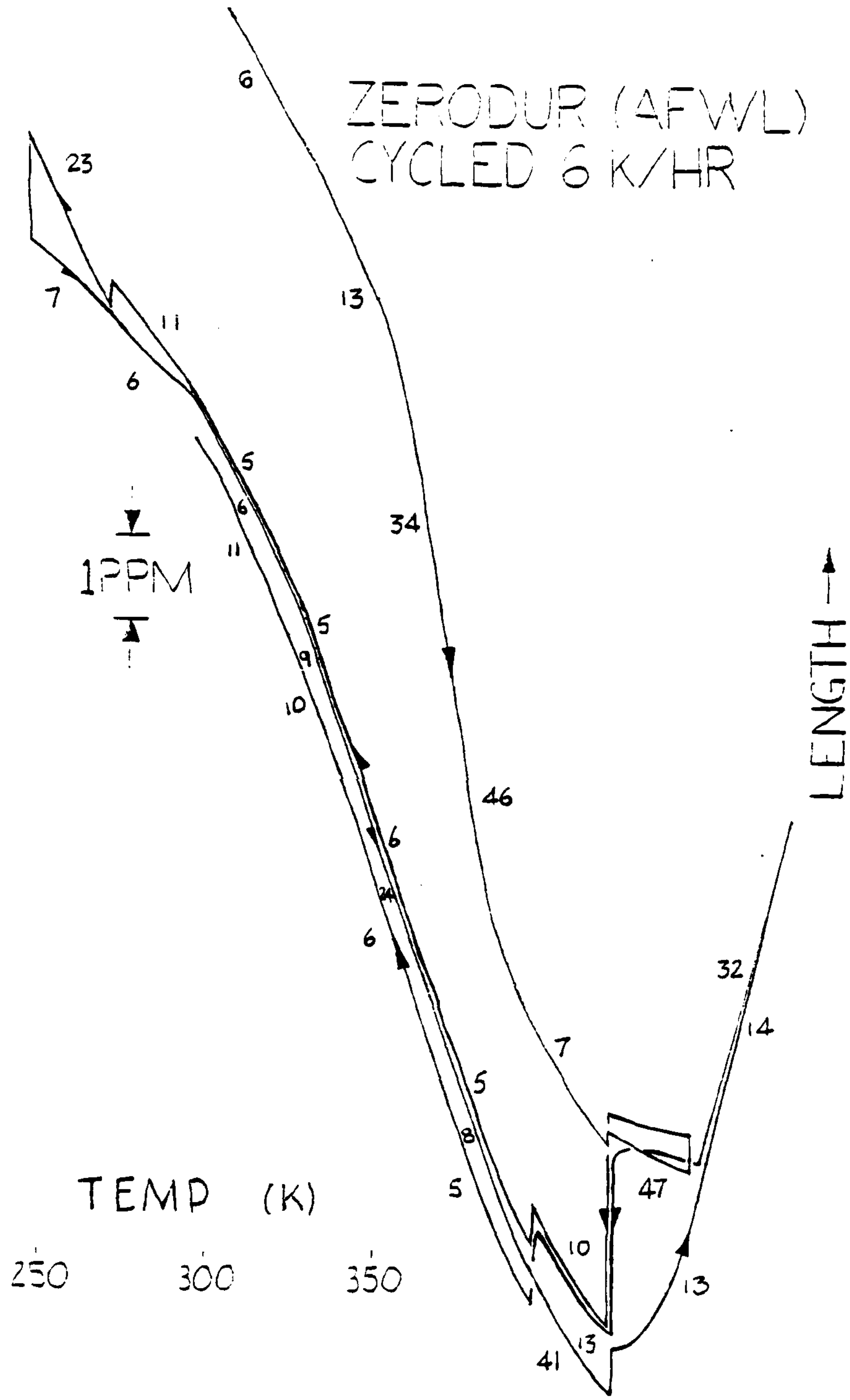


Fig. 5. Detailed study of Zerodur twice cycled 6 K/hr, showing waiting times.

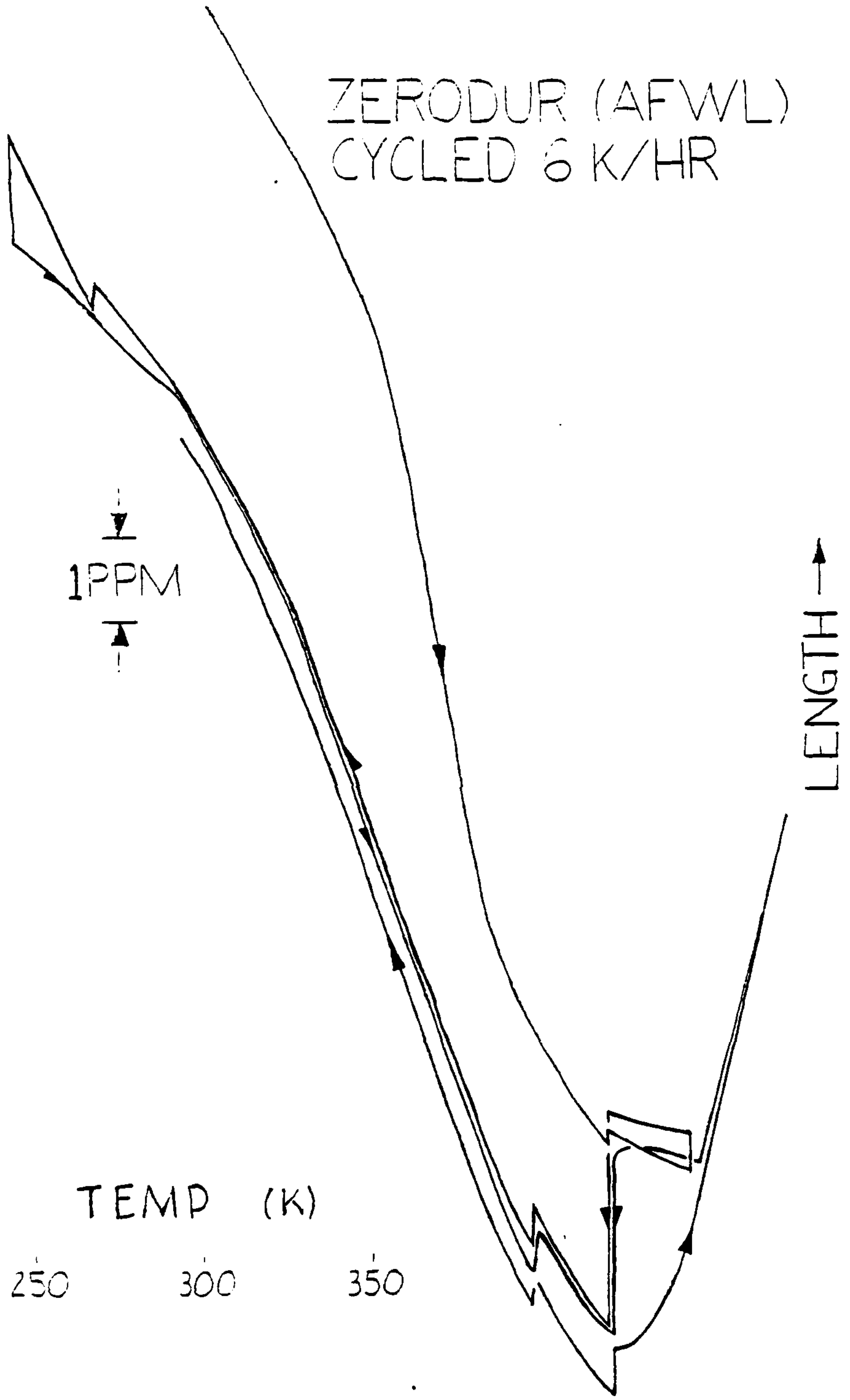


Fig. 6a. Dilatometer sample of Zerodur cycled 6 K/hr.

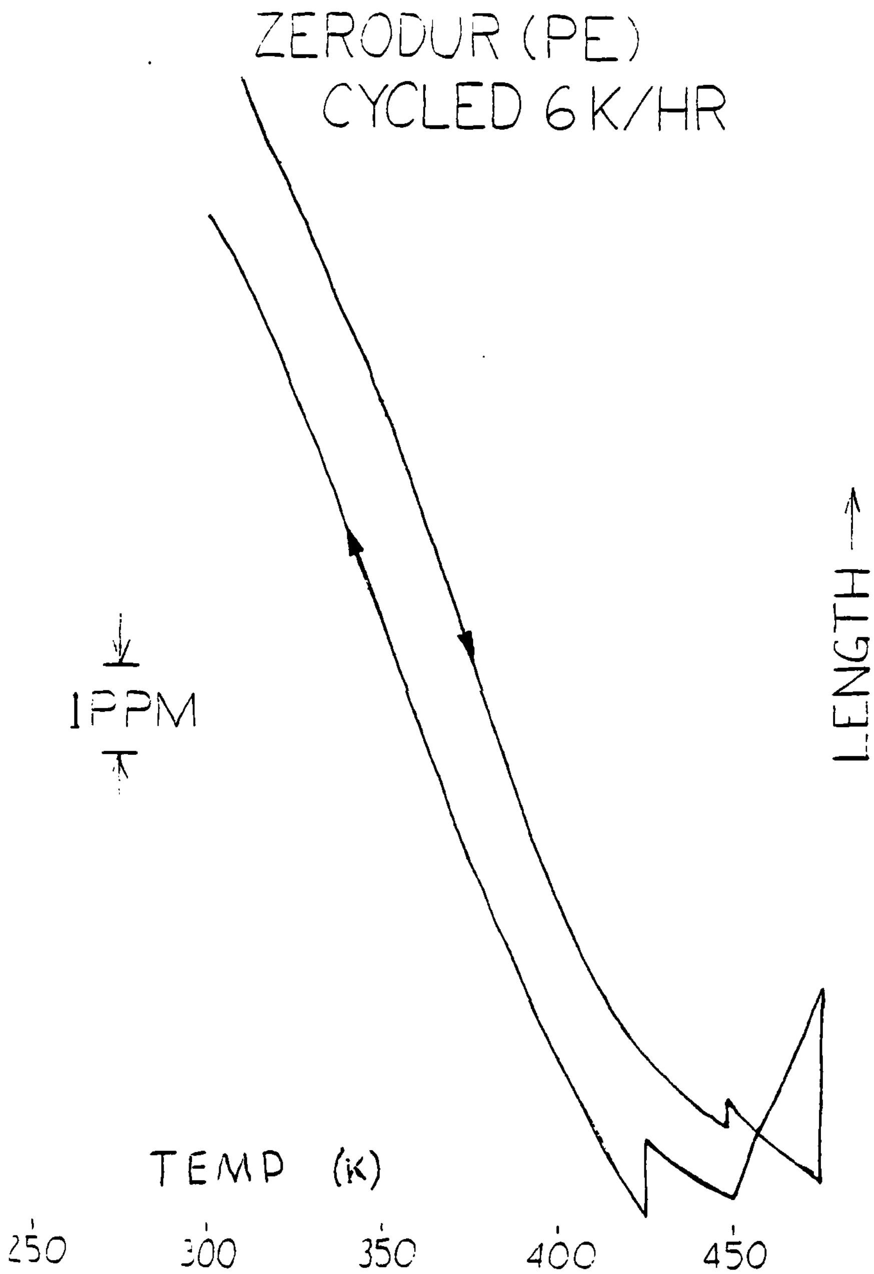


Fig. 6b. Continued.

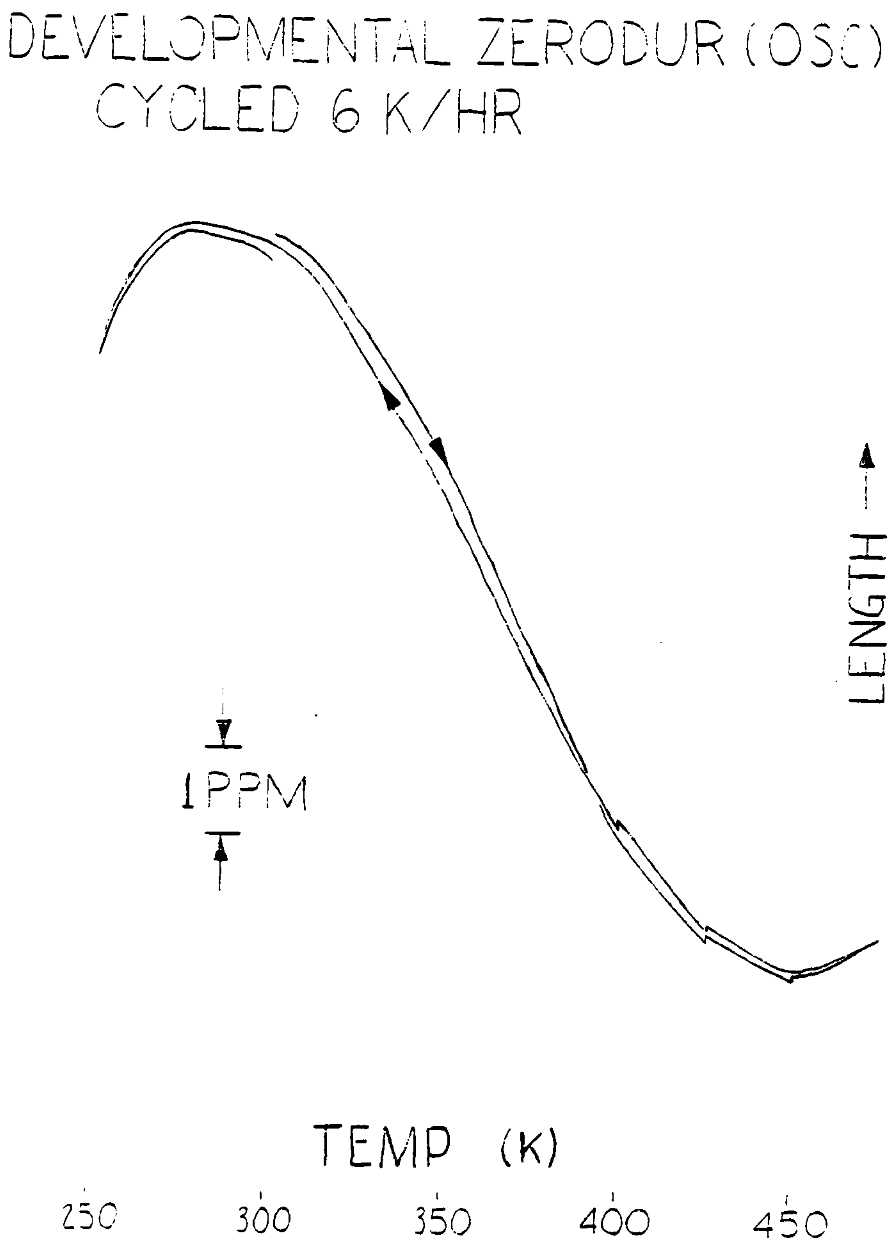


Fig. 6c. Continued.

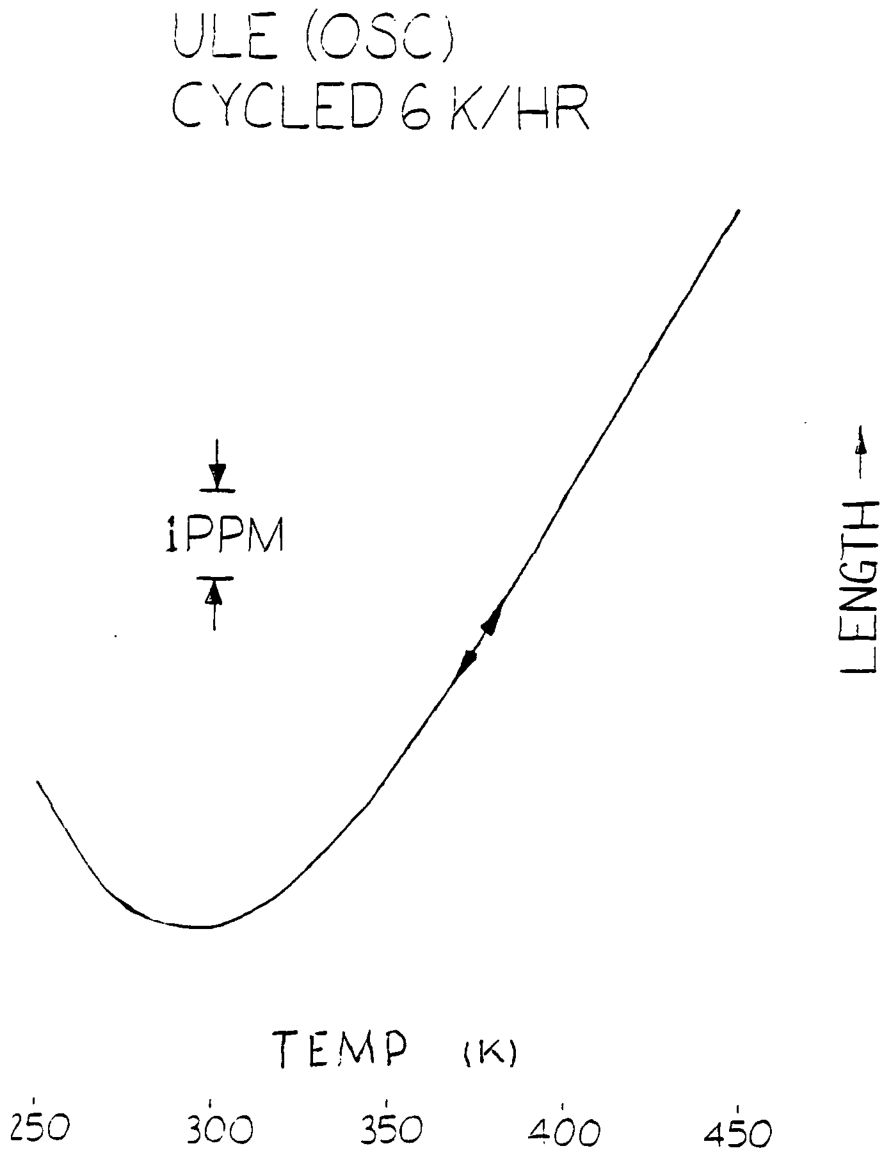


Fig. 6d. Continued.

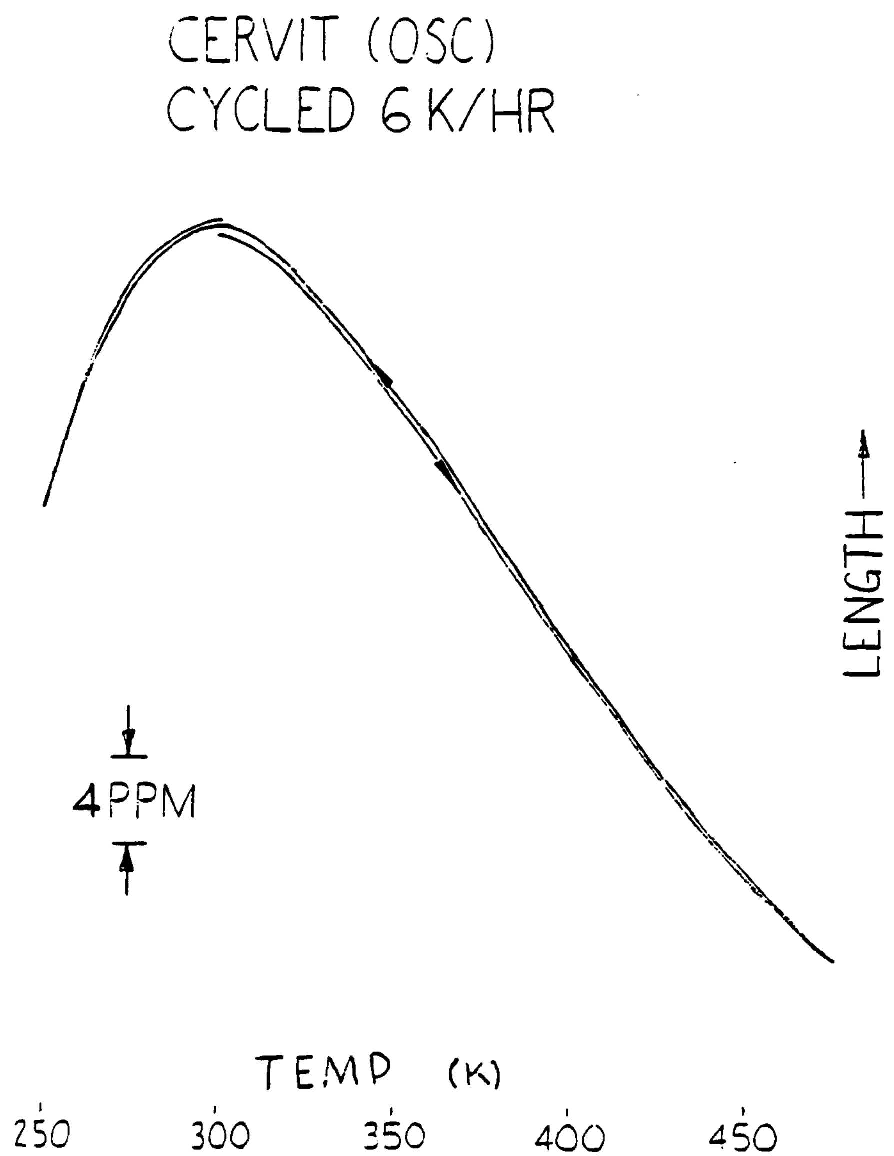


Fig. 6e. Concluded.

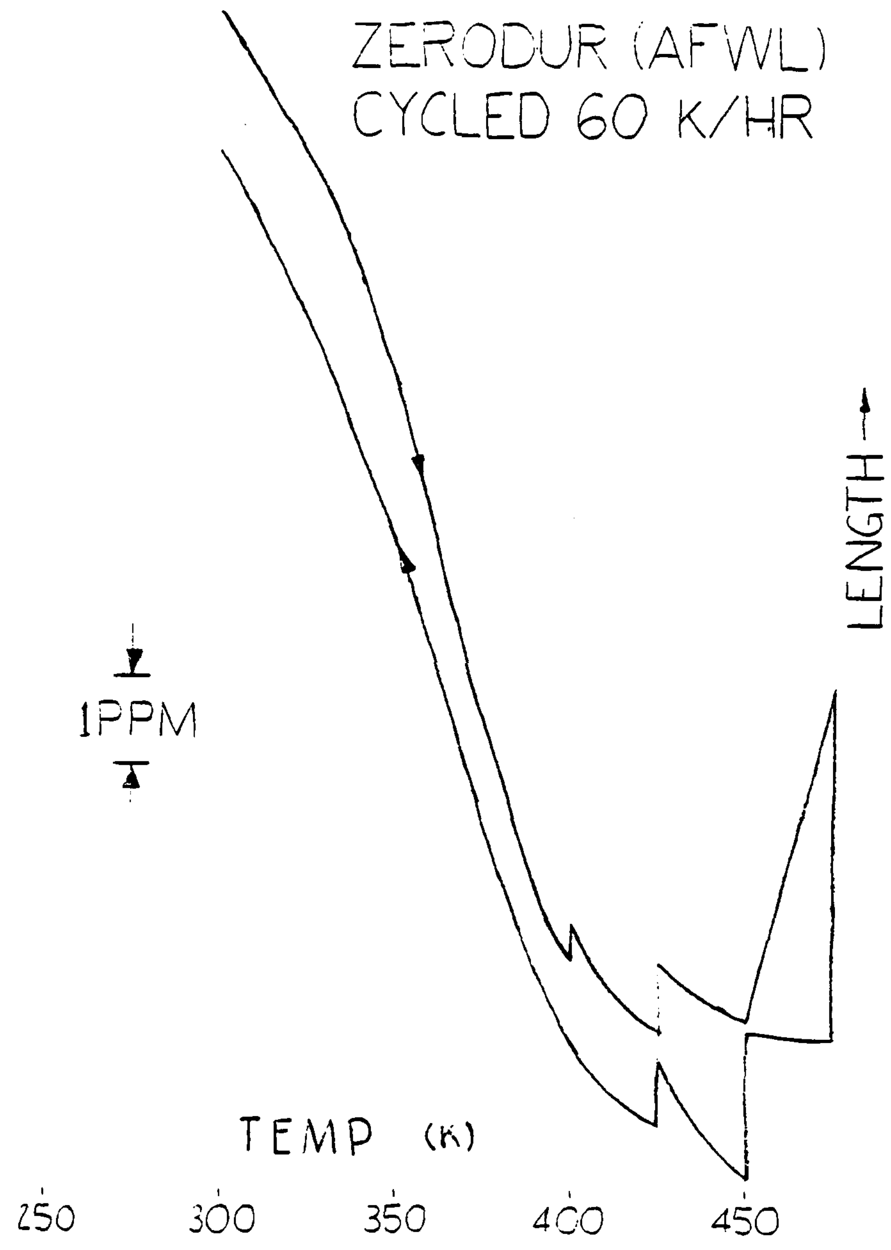


Fig. 7a. Dilatometer sample of Zerodur cycled 60 K/hr.

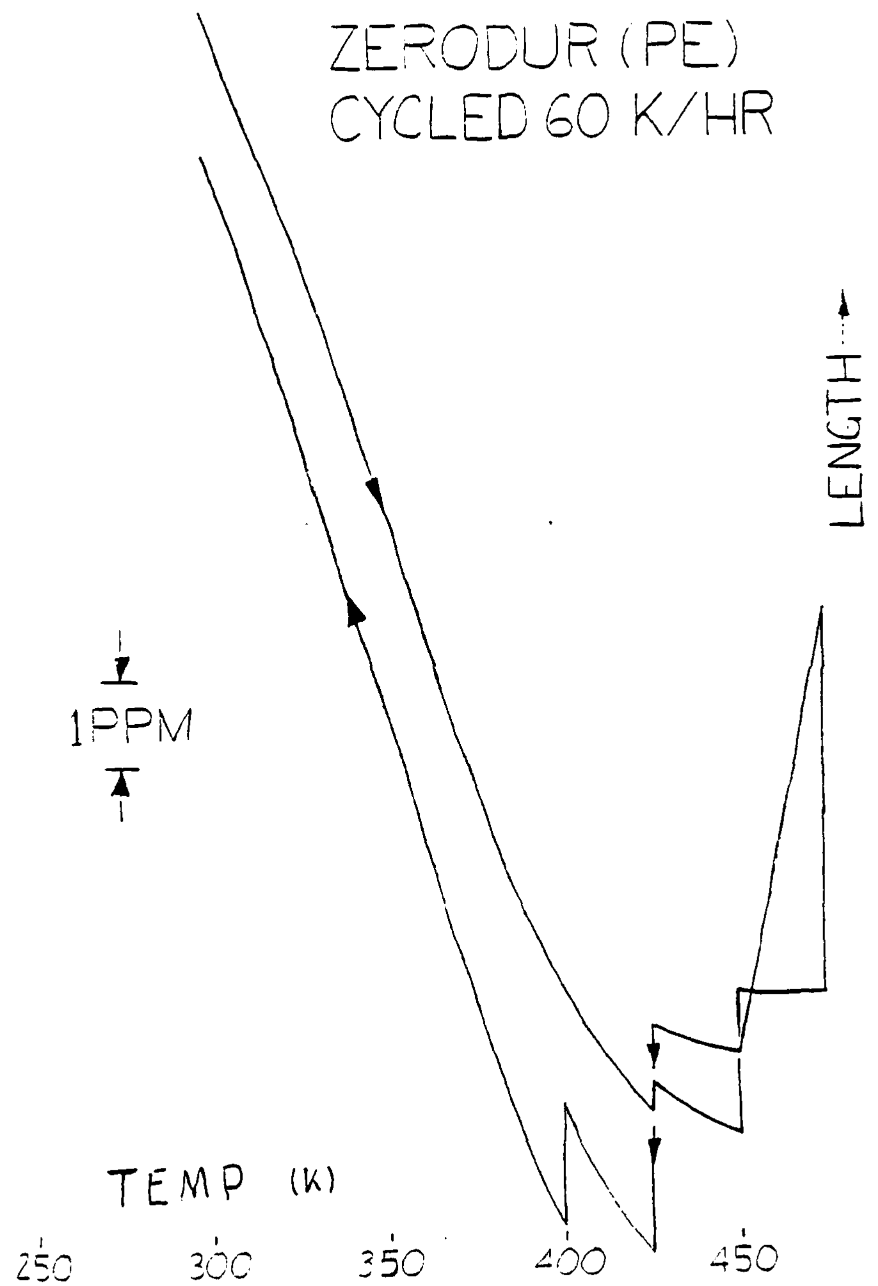


Fig. 7b. Continued.

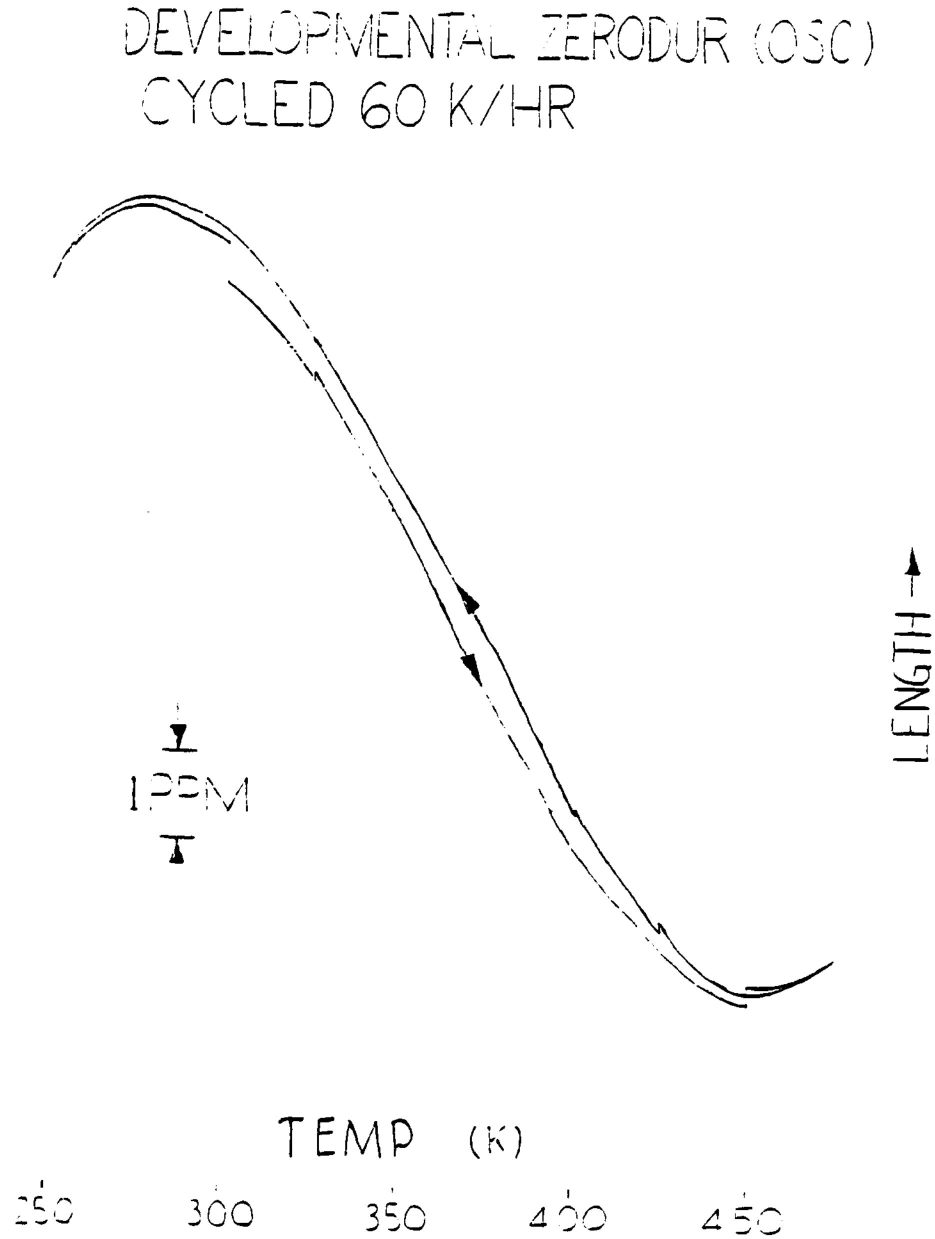


Fig. 7c. Continued.

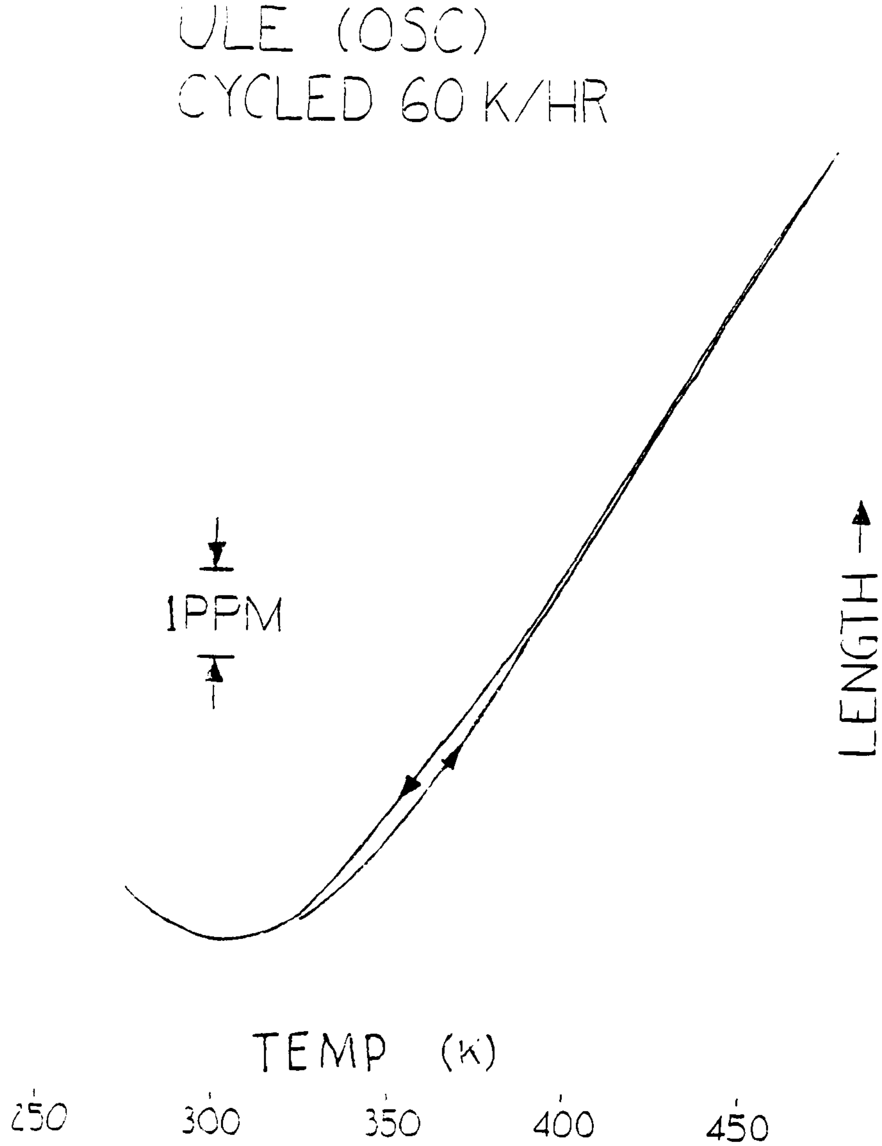


Fig. 7d. Continued.

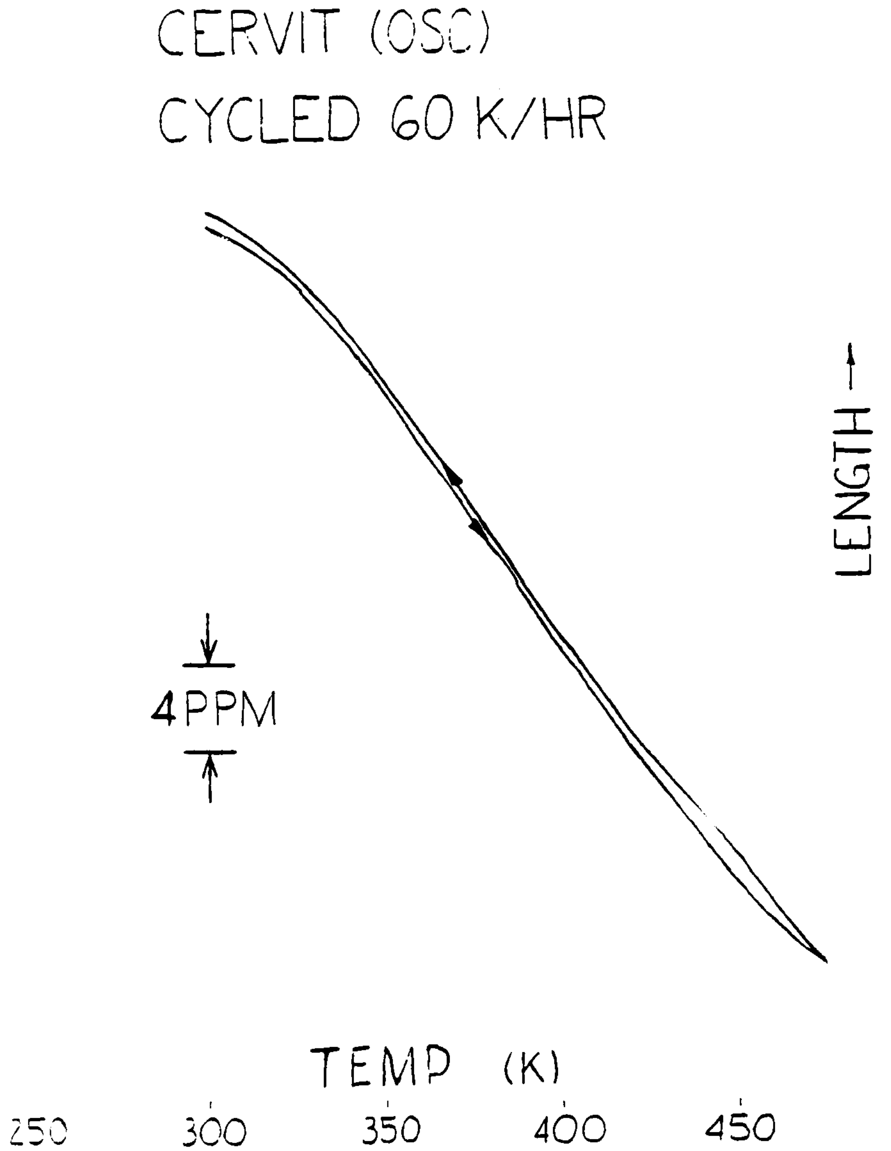


Fig. 7e. Concluded.

VII. SURFACE FIGURE MEASUREMENT METHOD

To characterize the surfaces, a real time Twyman-Green phase shifting interferometer was mounted on an air-suspended 8000 lb granite table. An entire underground room was dedicated to conducting these measurements. At early stages it became clear that to achieve data reproducibility, the measurement system has to be isolated. The effects of the system disturbing factors could be readily observed on the real time interference pattern. These effects of the system disturbing factors could be readily observed on the real time interference (RTI) pattern. These effects were vibrations of the fringe pattern, fringe drifting, and fringe distortion. They were mainly caused by building vibrations and sound, system temperature changes, and air turbulence.

To overcome these disturbing effects the massive granite table supporting the test setup had to be air suspended. The masses of the interferometer and mirror mounts were augmented to decrease their natural resonance frequency. To minimize thermal effects, a thermal insulation Styrofoam enclosure was constructed to cover the entire test setup, which also improved air stability of the optical testing path. The test instruments were left on throughout all 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 which led to data reproducibility with the required precision.

A reference mirror was incorporated to monitor the system performance and verify that for each measurement the test configuration remained the same (Fig. 8). As discussed in Section III, prior to each RTI measurement each surface was coated chemically with a thin film of silver to increase its reflectivity; the coating was later removed before thermal cycling. The effective aperture diameter was about 7 in. because edge diffraction effects introduced spurious data which led us to ignore the outermost rim.

Thirty data sets were taken for each sample before and after heat treatment. Averages were then computed and the differences of those averages were obtained to determine the change in optical figure. Referring to Figures 9 through 11, this change is presented as a surface contour for each sample in the left-hand column; the number given represents the root-mean-square (RMS) surface change (in visible wavelengths). Immediately following each sample measurement the same procedure was followed for the reference (sample removed). The change in reference figure is presented in the right-hand column of the Figures 9 through 11, along with RMS changes. The data processing was accomplished with WISP software from WYKO Corporation, Tucson, Arizona.

Each data set provides a surface contour obtained through phase-shifting interferometry. The set of figures that show the change in reference surface provide information about the system reproducibility as well as the system precision. These system characteristics are within the specifications required: (λ/50).

Surface contours are preferred for displaying global results, rather than simple numerical quantities which often fail to adequately describe information for this study of surface distortion. An estimate of the reliability of each measured change in sample figure may be obtained by forming a signal-to-noise ratio using the RMS change in sample figure as signal, and the RMS change in reference as noise.

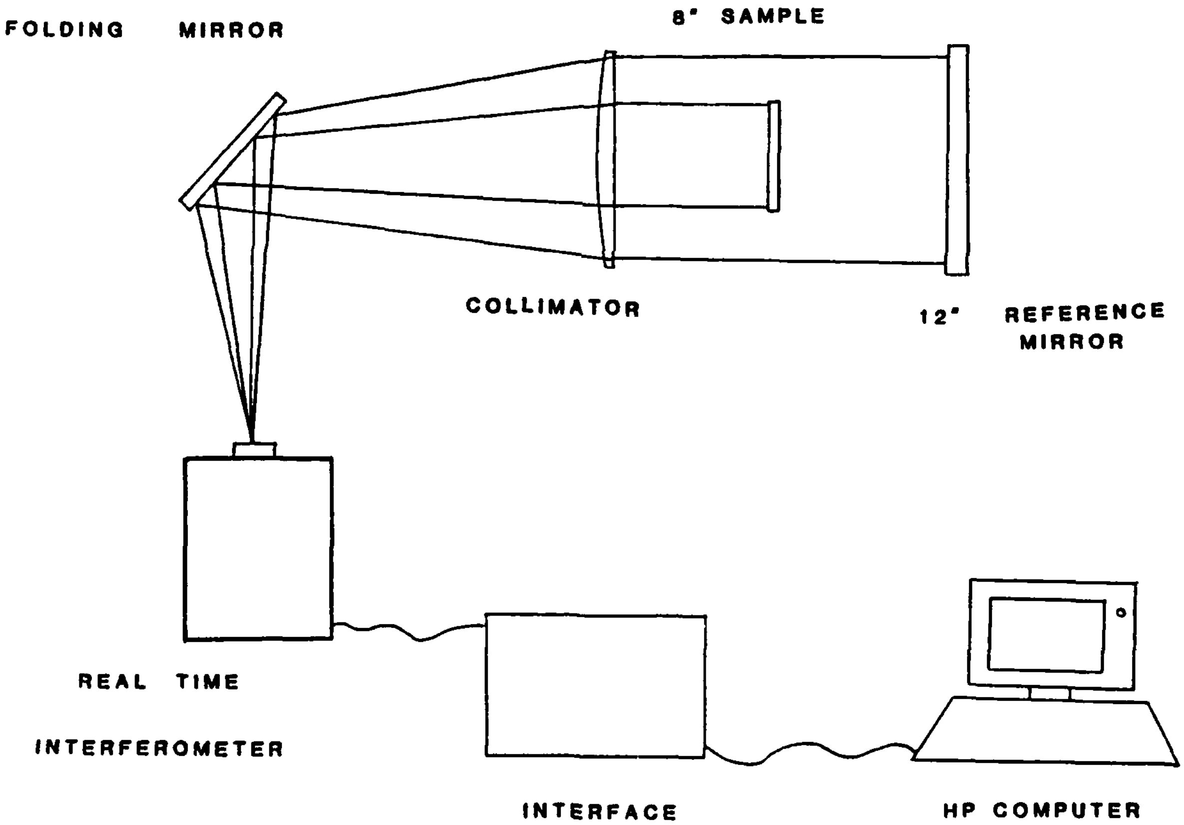


Fig. 8. RTI test arrangement.

VIII. SURFACE FIGURE RESULTS

1. Uniform heating - 60 K/hr (Figs. 9a and 9b).

The 8-in. discs of five materials were thermally cycled 300 to 475 K at 60 K/hr to determine figure changes. The left- and right-hand columns respectively show sample changes before versus after heating and changes in the reference (system reproducibility). Our general conclusion is that 60 K/hr uniform heating caused no significant change in surface figure.

2. Uniform heating - 6 K/hr (Figs. 10a and 10b).

We expected to see even less distortion at 6 K/hr than at 60 K/hr; this indeed was what we saw, except for the case of ULE, which showed quite a significant distortion. 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 had unusual strain in it. The conclusion is that, despite the ULE result, 6 K/hr uniform heating generally caused no significant change in surface figure.

3. Nonuniform heating - 90 K/hr (Figs. 11a and 11b).

Figure 3 shows the configuration of the vacuum coater used for nonuniform heating. The coater accommodated three 8-in. samples. Therefore, to maintain good mechanical balance, we added a second developmental Zerodur sample to the five. This resulted in two thermal cycling runs of three samples each.

The results are very striking. Both the Air Force Weapons Lab- and Perkin-Elmer-polished Zerodur showed significant distortion, while none of the others did. Especially noteworthy are the two samples of developmental Zerodur, Z'_{OSC}, which showed no significant distortion. (One was distorted a little more than the other, but far less than standard Zerodur).

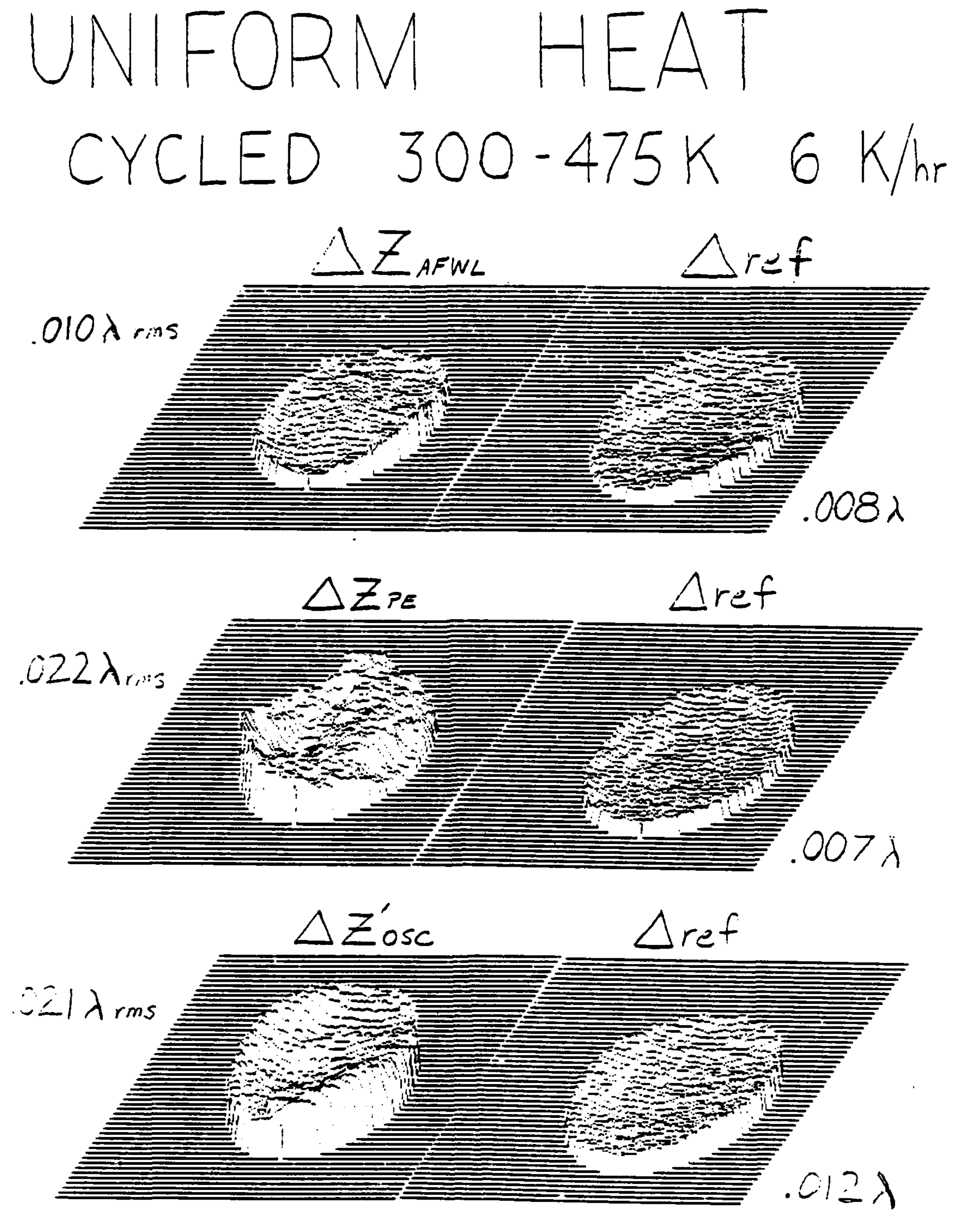


Fig. 9a. Uniform heat - 60 K/hr.

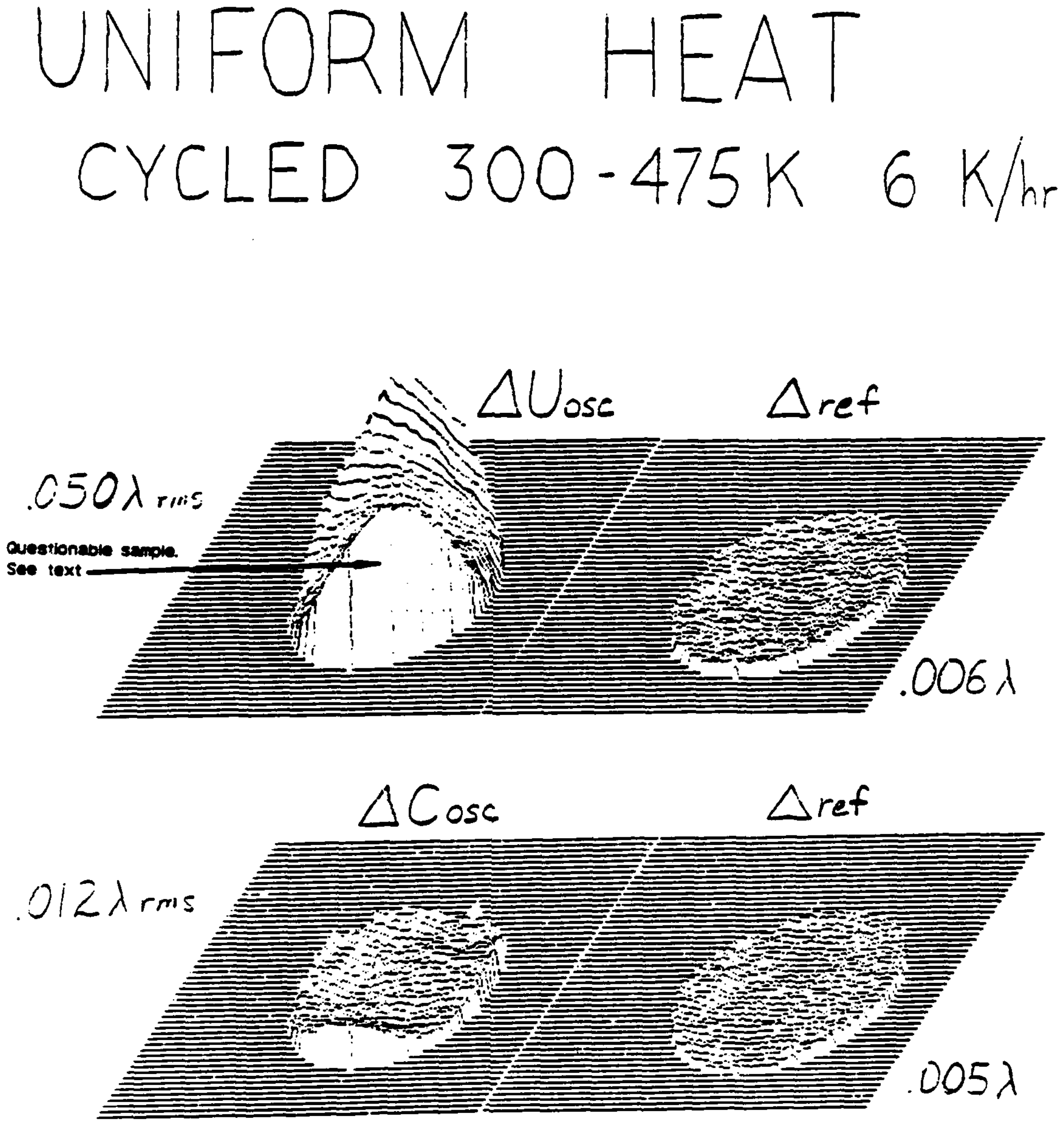


Fig. 9b. Concluded.

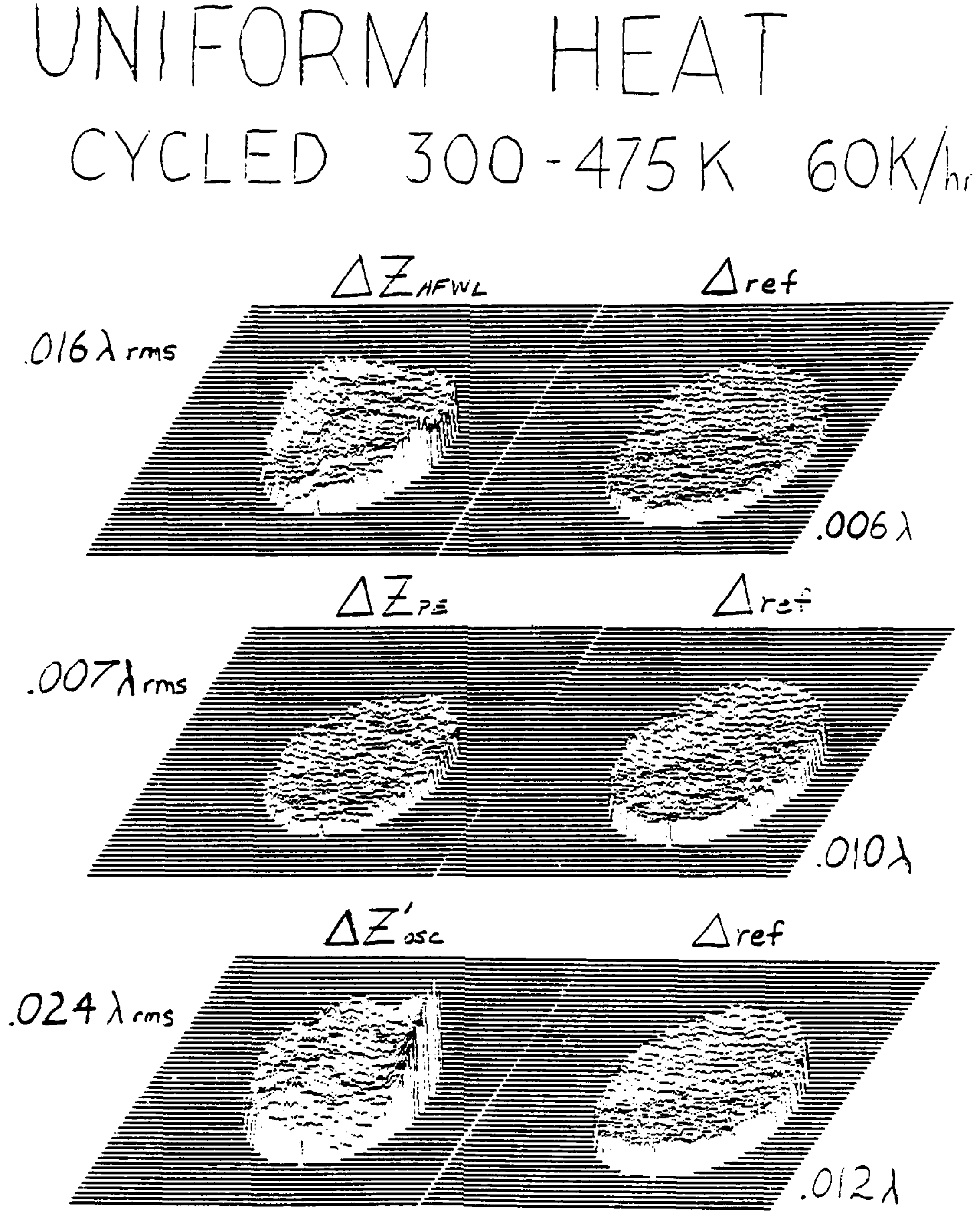
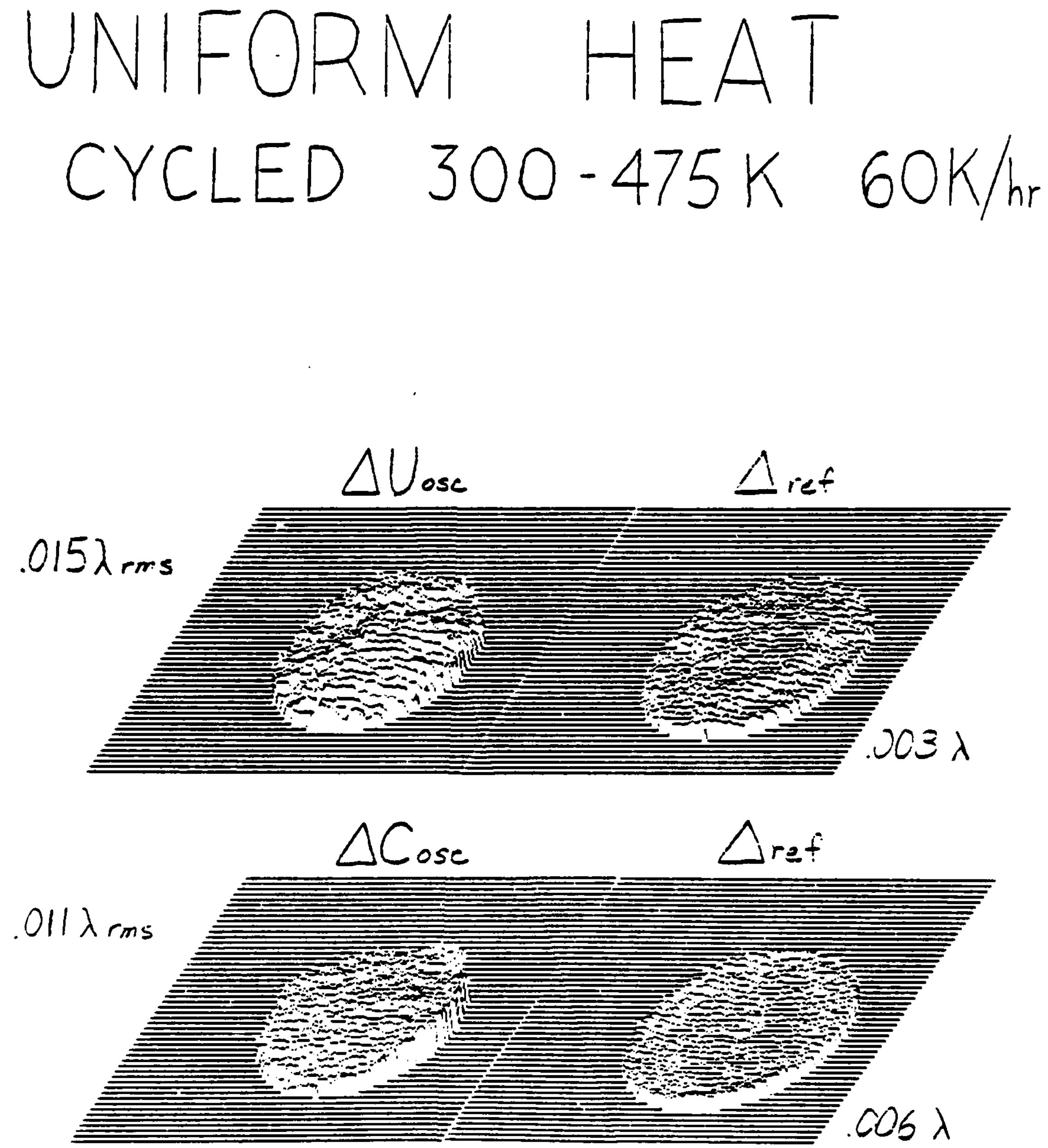


Fig. 10a. Uniform heat - 6 K/hr.



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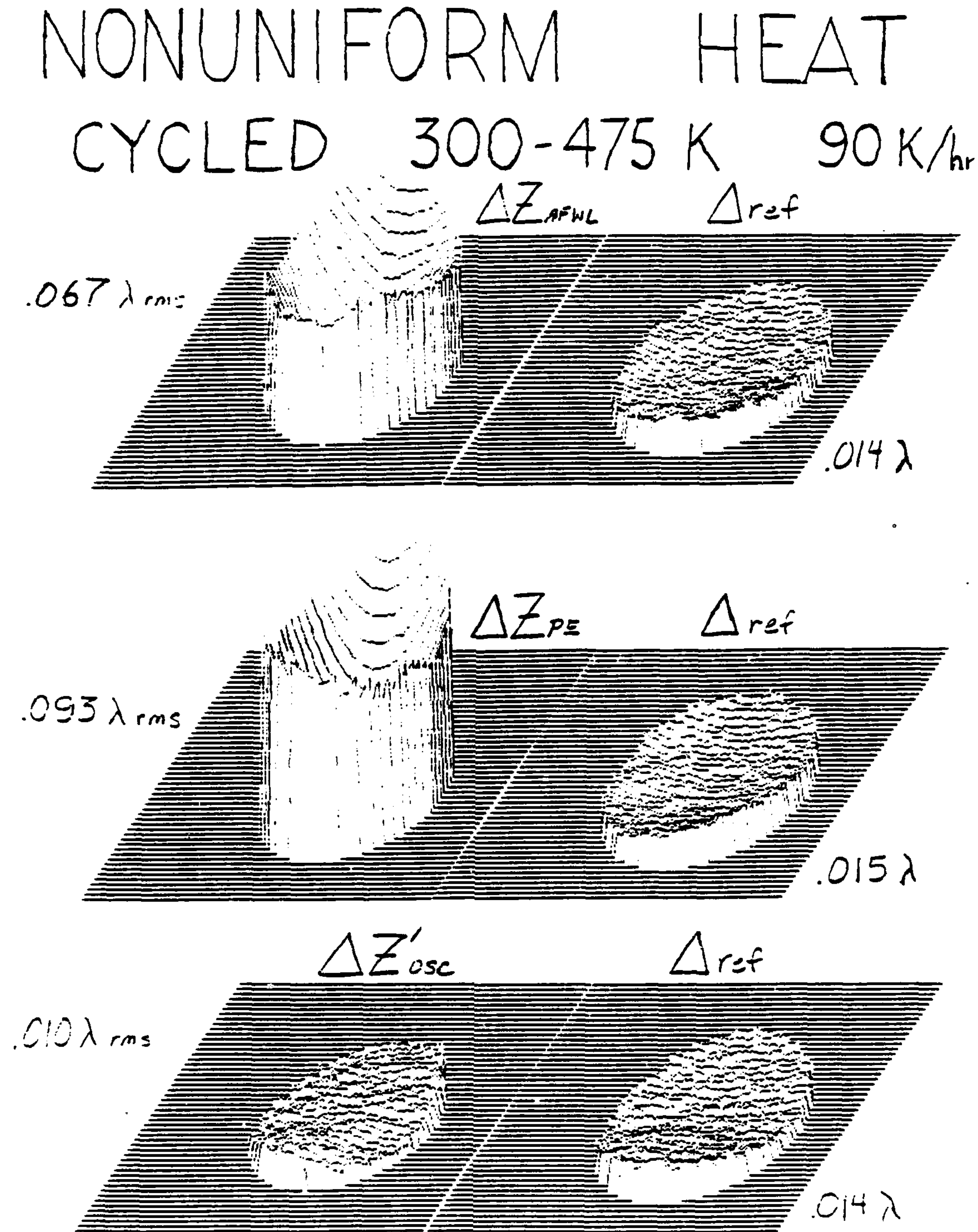

Fig. 11a. Nonuniform heating - 90 K/hr.

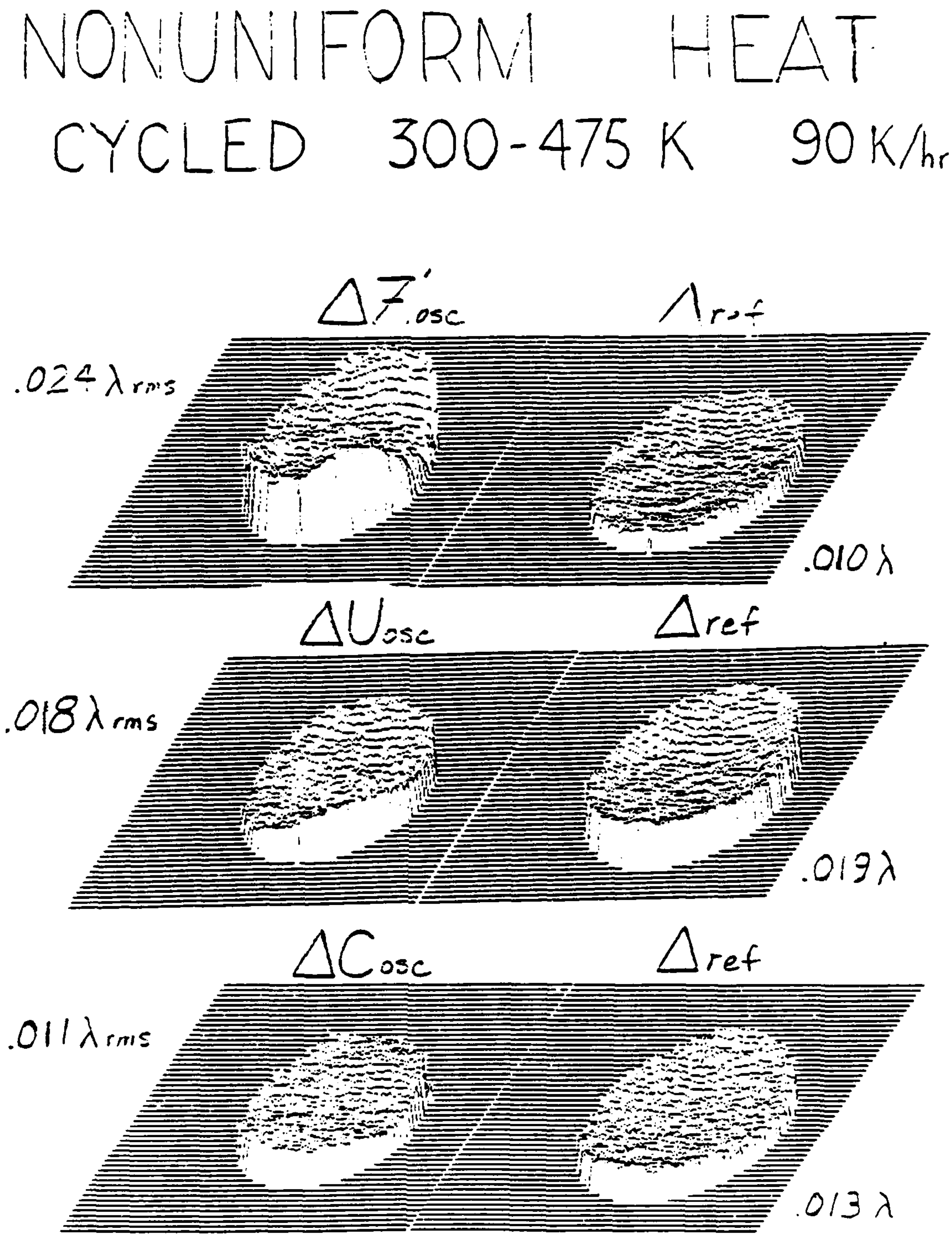

Fig. 11b. Concluded.

IX. COMPARISON OF DILATOMETER VERSUS SURFACE FIGURE MEASUREMENTS

Overview

In Figures 15 through 17 the dilatometer versus surface figure measurements were compared directly. To emphasize the correlation between hysteresis and figure distortion, we have deleted from the dilatometer curves many identification details, which were presented in Section VI.

In Figure 14, nonuniform heat, 90 K/hr, the dilatometer curves are the same as those for 60 K/hr. Thus the comparison is only approximate when it comes to cycling rate.

Details

Figures 9 through 11 and 12 through 14 present the same data as is shown in the overview, but without deletion of details, and grouped for direct comparison of surface data versus dilatometer curves.

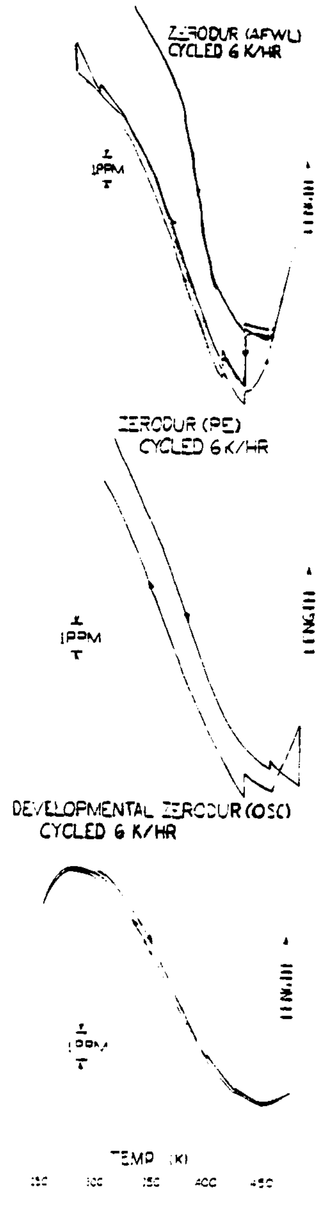

Fig. 12a. Uniform heat - 6 K/hr.

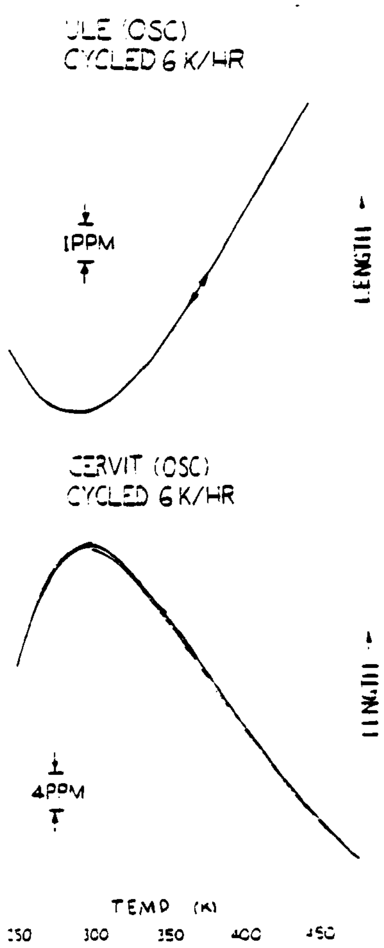

Fig. 12b. Concluded.

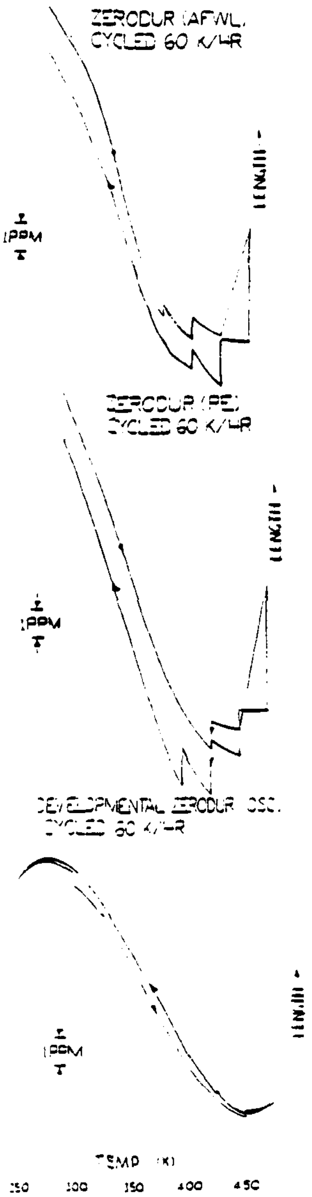

Fig. 13a. Uniform heat - 60 K/hr.

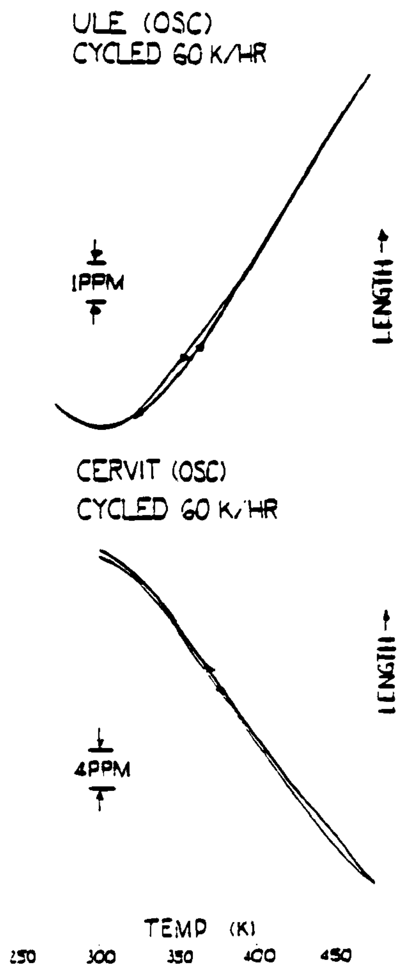

Fig. 13b. Concluded.

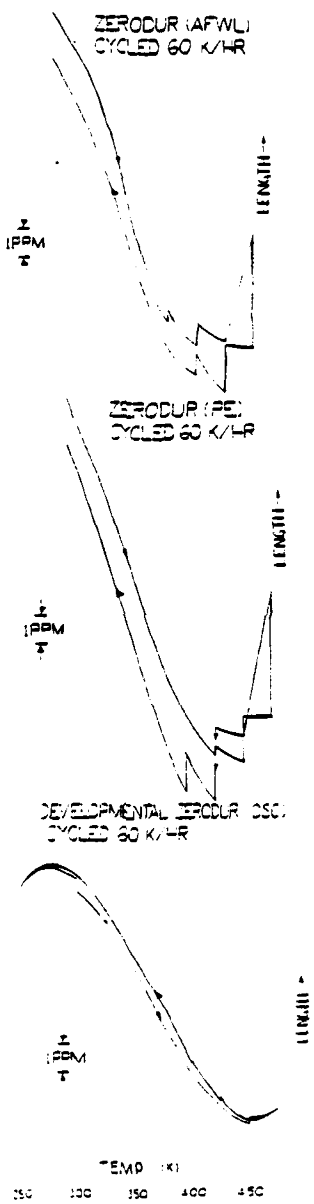

Fig. 14a. Nonuniform heating - 90 K/hr.

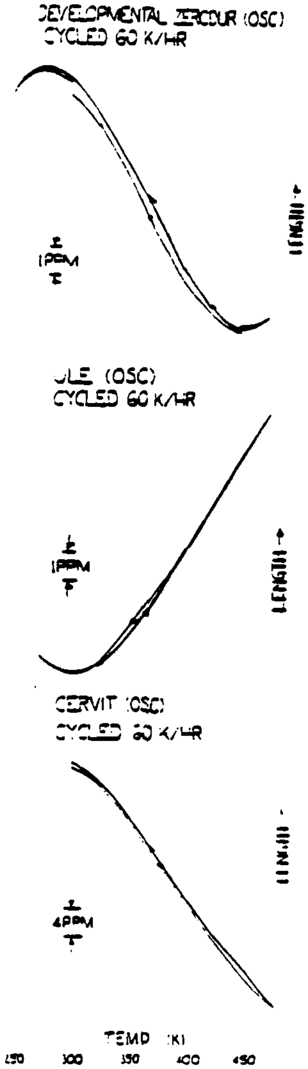

Fig. 14b. Concluded.

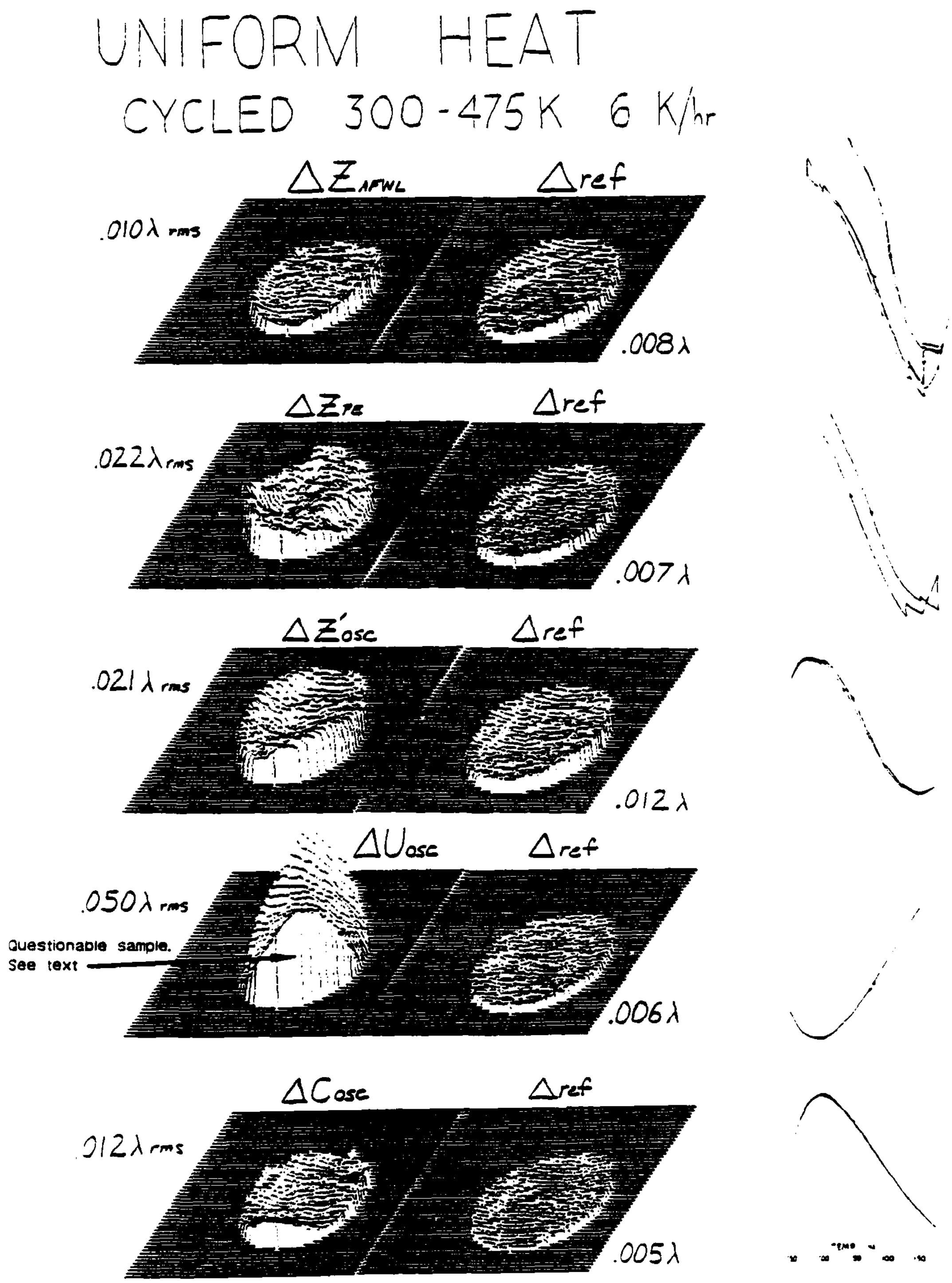

Fig. 15. Uniform heat 6 K/hr.

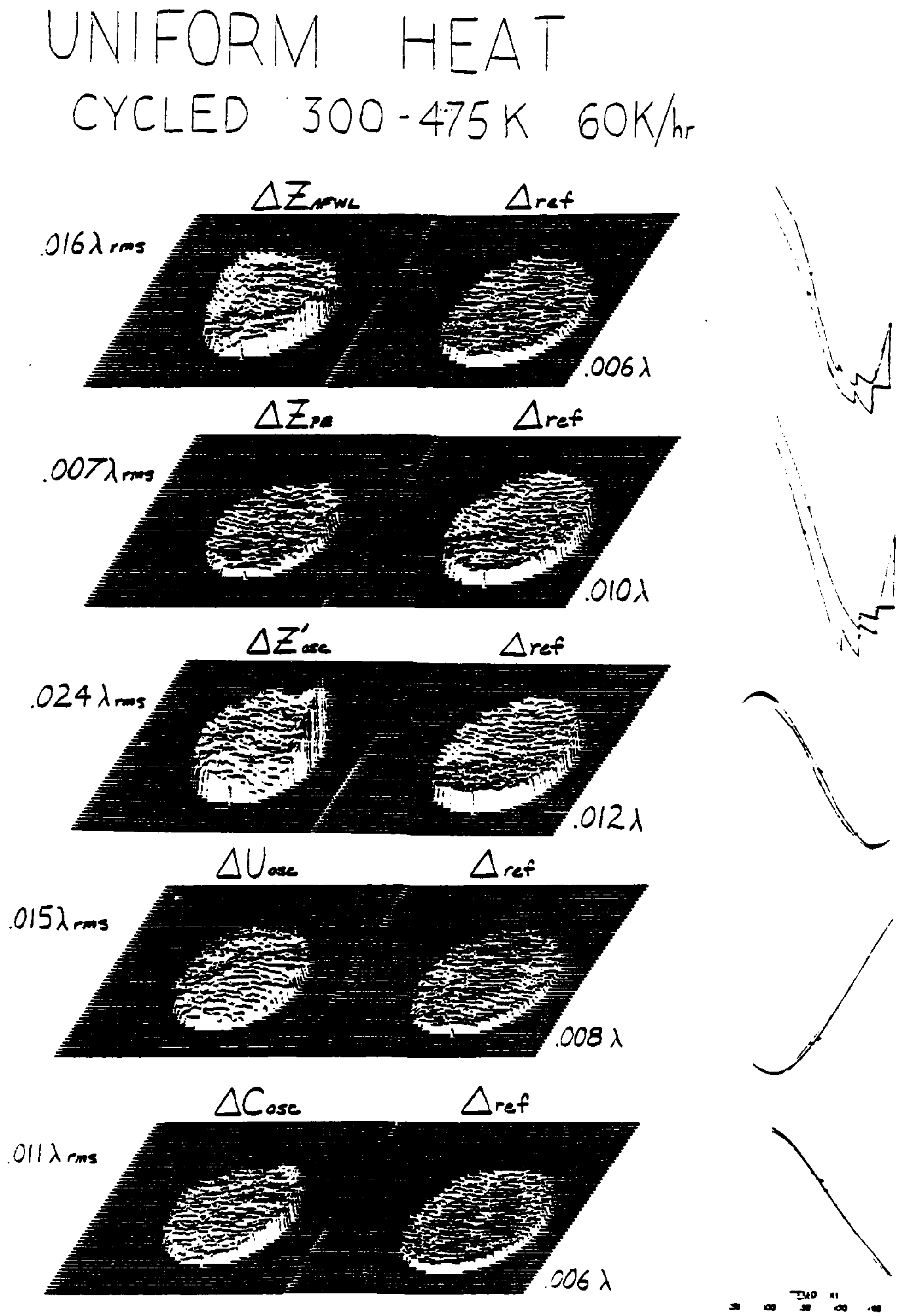

Fig. 16. Uniform heat 60 K/hr.

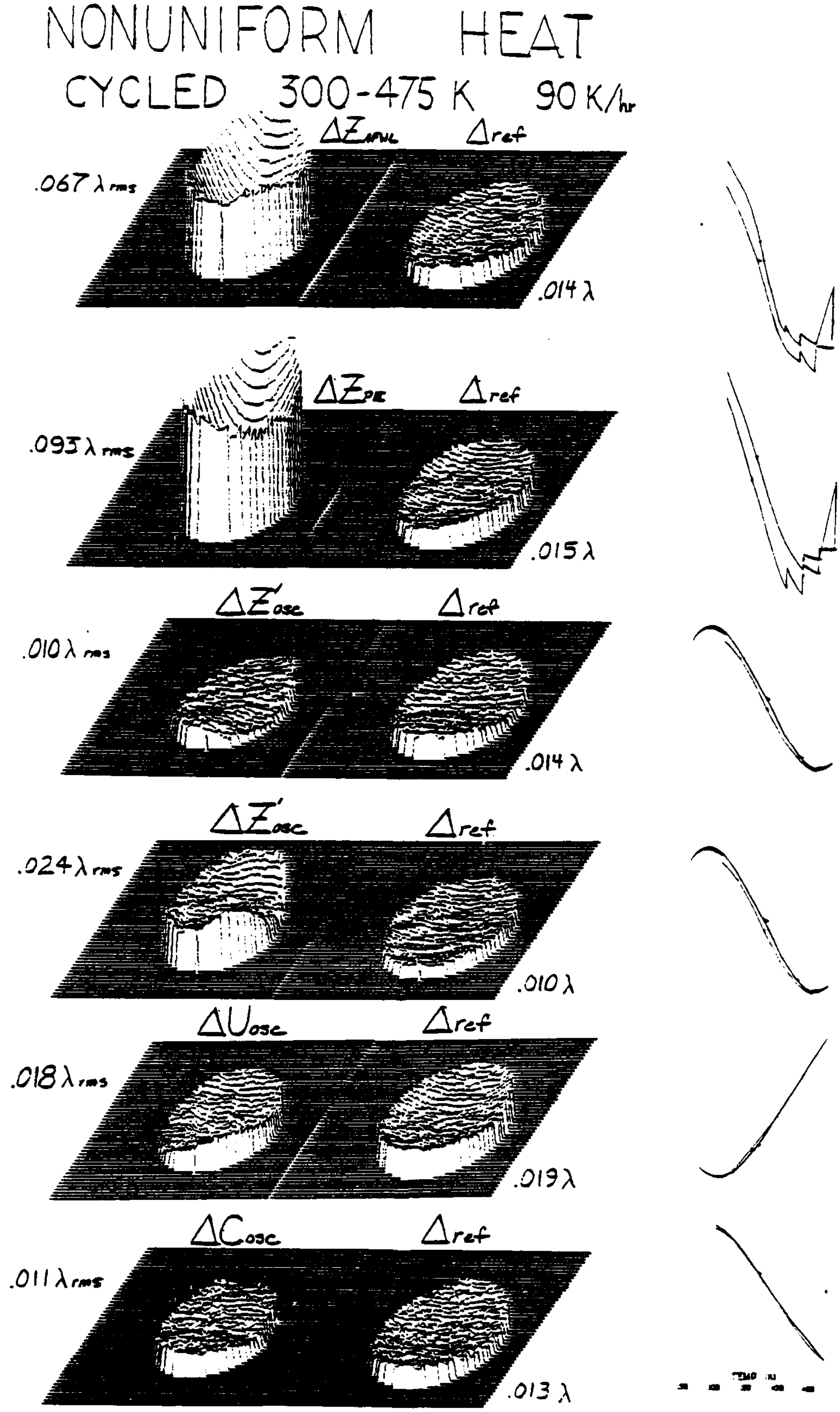

Fig. 17. Nonuniform heat 90 K/hr.

X. CONCLUSIONS

This investigation has been successful in answering many questions about the best low expansion mirror materials presently available. It 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.

We summarize below the findings of this work:

1. Cer-Vit and ULE exhibit no significant hysteresis figure distortion under the conditions of this study, even though the Cer-Vit showed obvious strain birefringence with crossed-polaroid examination.
2. In Standard Zerodur the hysteresis present appears to be responsible for surface figure distortion (about $\lambda/10$ across 8 in.). Additional strain (several ppm) is sometimes present which can be annealed out by thermal cycling. This strain may be due to thermal shock or machining procedures.
3. Uniform heating (300 to 475 K) causes no significant surface deformation in any of the materials studied here; however, *nonuniform* heating does cause surface deformation in materials which have 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 add to this the fact that distortion can take place at even lower temperatures (~450 K, where hysteresis sets in), but is due to *nonuniformity* of heating. This suggests that in the Bennett study the culprit was the quenching, which introduced nonuniformity into the cooling process. Our study agrees with and complements Bennett's finding that ULE exhibits no distortion.
4. Concerning effect of *rate* of cycling on hysteresis produced, we show that 60 K/hr generally causes more severe hysteresis than 6 K/hr in standard Zerodur.
5. The Perkin-Elmer treatment produced no significant reduction in standard Zerodur surface distortion, compared with standard Zerodur prepared by AFWL polishing procedures. The only difference observed was that the PE treated material was free from first-run irreversible characteristics. The PE treated material still had standard Zerodur hysteresis, but behaved as if it had already received some thermal cycling to relieve strains. In the present work we have demonstrated that thermal cycling can eliminate failure to return to length due to strain, but apparently it cannot eliminate hysteresis caused by structural components such as MgO.

6. Schott's new developmental material was virtually free of hysteresis and surface figure distortion under the conditions of these tests. This result indicates a connection between hysteresis and figure distortion. (No hysteresis; no figure distortion) Schott apparently anticipated this and removed the hysteresis-causing MgO from the recipe of the new material. Schott is to be congratulated on achieving this without sacrificing low expansion characteristics.

XI. RECOMMENDATIONS

Uniformity of thermal expansion is every bit as important as low thermal expansion in the performance of a telescope mirror. For this reason, the good news about Schott's experimental material must be followed up with evaluation of its thermal expansion uniformity.

We recommend that Schott make available samples from a large ingot of the new material, selected in such a way that the variation of CTE can be mapped and CTE gradient obtained in much the same way as was done for Heraeus TO8E fused quartz.[4]

Other characteristics which should be evaluated for the new material include its CTE at low temperatures and whether there is still hysteresis at low temperatures (~250 K) as Lindig et al. (Ref. 3) predicted there might be.

Two 8-in. polished flats of each material have been returned to AFWL for further testing.

XII. REFERENCES

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