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MEASUREMENT OF THERMAL EXPANSION IN SOLID PROPELLANTS

James A. McKinnis

Air Force Rocket Propulsion Laboratory
Edwards Air Force Base, California

March 1973

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TECHNICAL REPORT AFRPL-TR-72-109

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FOREWORD

This report was prepared by the Propellant Development Branch, Solid Rocket Division, Air Force Rocket Propulsion Laboratory (AFRPL), Edwards, California. The work documented by this report was conducted from July 1971 to July 1972 under Project 305910MA, "Solid Propellant Mechanical Behavior Investigation." The project engineer on this effort was Capt James A. McKinnis.

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This report has been reviewed and is approved.

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ABSTRACT

An analysis was performed to determine the sensitivity of solid rocket motor stress (strain) analyses to a variation in thermal coefficient of linear expansion (TCLE). For the wide range of TCLE values currently utilized in the propellant industry, the stress analysis predicted an approximately 60 percent change in the maximum inner bore strain. A laser holograph technique was therefore devised to more accurately measure propellant TCLE. The interferometric holographic technique provided an accurate means (resolution in the microinches) of measuring linear thermal expansion for small temperature variations (1.0 to 3.6°F). Irregularities resulted in the interference fringes when the temperature change was greater than 4.0°F. For the limited data taken, measured TCLE values were well within the currently acceptable range, but this effort did not narrow the range appreciably.

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INTRODUCTION

Based upon the recommendation of the 1970 JANNAF Mechanical Behavior Working Group, a solid propellant thermal coefficient of linear expansion (TCLE) study was undertaken in July of 1971. The purpose of this study was twofold; first, determine how sensitive stress analyses are to typical TCLE variations and secondly, if this sensitivity proved to be large, determine an accurate and repeatable means of measuring TCLE.

In the last few years, laser holography has become an extremely useful experimental tool for measuring very small displacements. By making use of a holographic double exposure technique, interference patterns can be obtained of displacements as small as 13 microinches. The average TCLE value for solid propellants is approximately 50 micro in/in/$^{\circ}$F. It was, therefore, decided that the holographic technique should be able to accurately and repeatably measure propellant TCLE. The only major limitation of this technique was the ability to resolve the number of interference fringes that appear on the propellant surface. This effectively limits the technique to measuring small expansions (typically, 400 to 500 microinch displacements) for the rough propellant surfaces.

SECTION II

SENSITIVITY ANALYSES

A. OBJECTIVE

Due to the inaccuracies in measuring techniques and to the interaction between thermal expansion and volume dilatation, there is a wide discrepancy in the measured values of TCLE for a given propellant. Typical TCLE measurements range from 3.5 to 7.5 x 10^{-5} in/in/°F. This variation is commonly cited as the cause of error between experimental results and analytical predictions (References 1 and 2). To determine just how sensitive these analyses are to the TCLE parameter, the following analytical study was undertaken.

B. TECHNIQUE

Both Rohm and Haas finite element (Reference 3) and Structural Design Handbooks (Reference 4) analyses were performed on a typical four star grain geometry shown in figure 1. The case and propellant material and physical properties used in these analyses are shown in Table I. Two different types of loads were imposed in each analysis. The first was a simple temperature differential of 100°F, while the second was a combined 100°F temperature differential plus a pressure load of 100 psi on the inner bore.

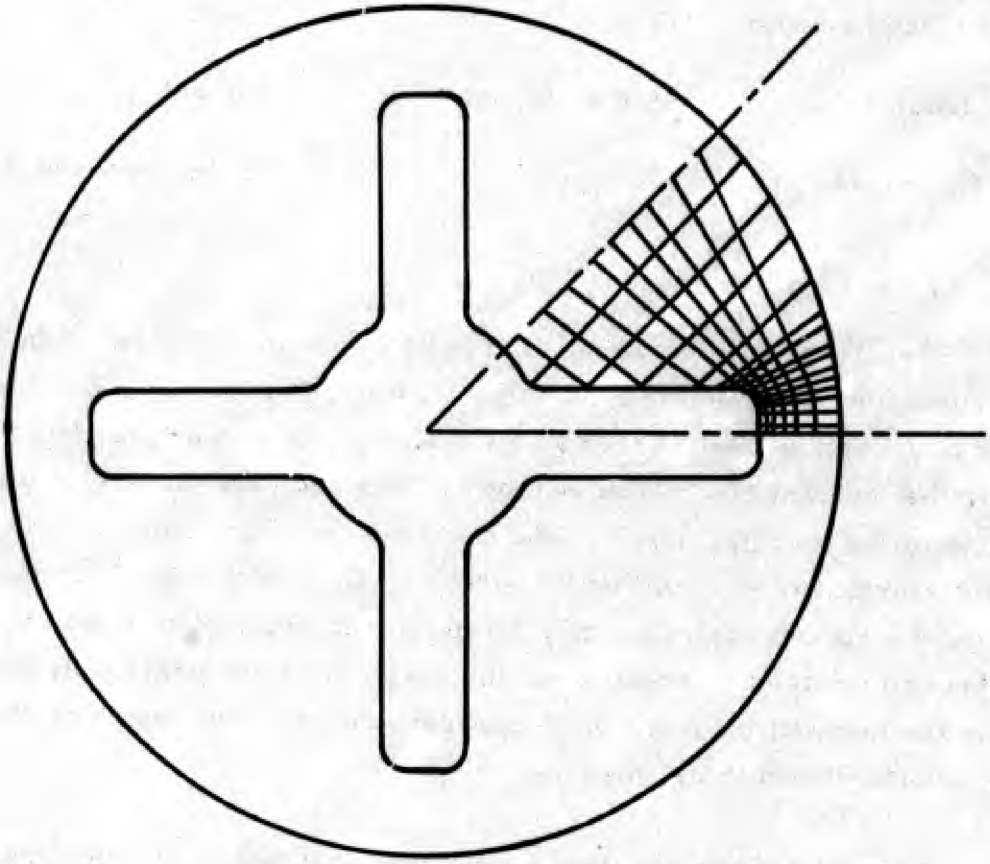

Figure 1. Finite Element Grid for Four Star Grain.

TABLE I. CASE AND PROPELLANT MATERIAL AND PHYSICAL PROPERTIES

	Case Properties	Propellant Properties
TCLE	6.0×10^{-6} in/in/°F	3.5×10^{-5} in/in/°F 5.5×10^{-5} in/in/°F 7.5×10^{-5} in/in/°F
Poisson's Ratio	0.3	0.495
Modulus	1.0×10^{6} psi	100 psi
Thickness	0.1 inch	75 percent (web fraction)

C. RESULTS

Results from both these analyses are shown in figure 2. In this figure, maximum inner bore strain is plotted versus TCLE for each analysis. As can be seen, there was a discrepancy between the two analyses for the thermal load. The reason for this anomaly is generally due to the size of the star grain web. The two analyses lose some validity when the web fraction is as small as 20 percent. Even with this discrepancy, figure 2 clearly indicates that the maximum inner bore strain varies up to approximately 60 percent (over the range of TCLE previously indicated) for the thermal loading. A 35 percent variation was predicted for the pressure plus thermal loading.

From these results it was concluded that linear elastic stress (strain) analyses are quite sensitive to the range of TCLE values studied and that a more accurate and repeatable means of measuring TCLE should be found.

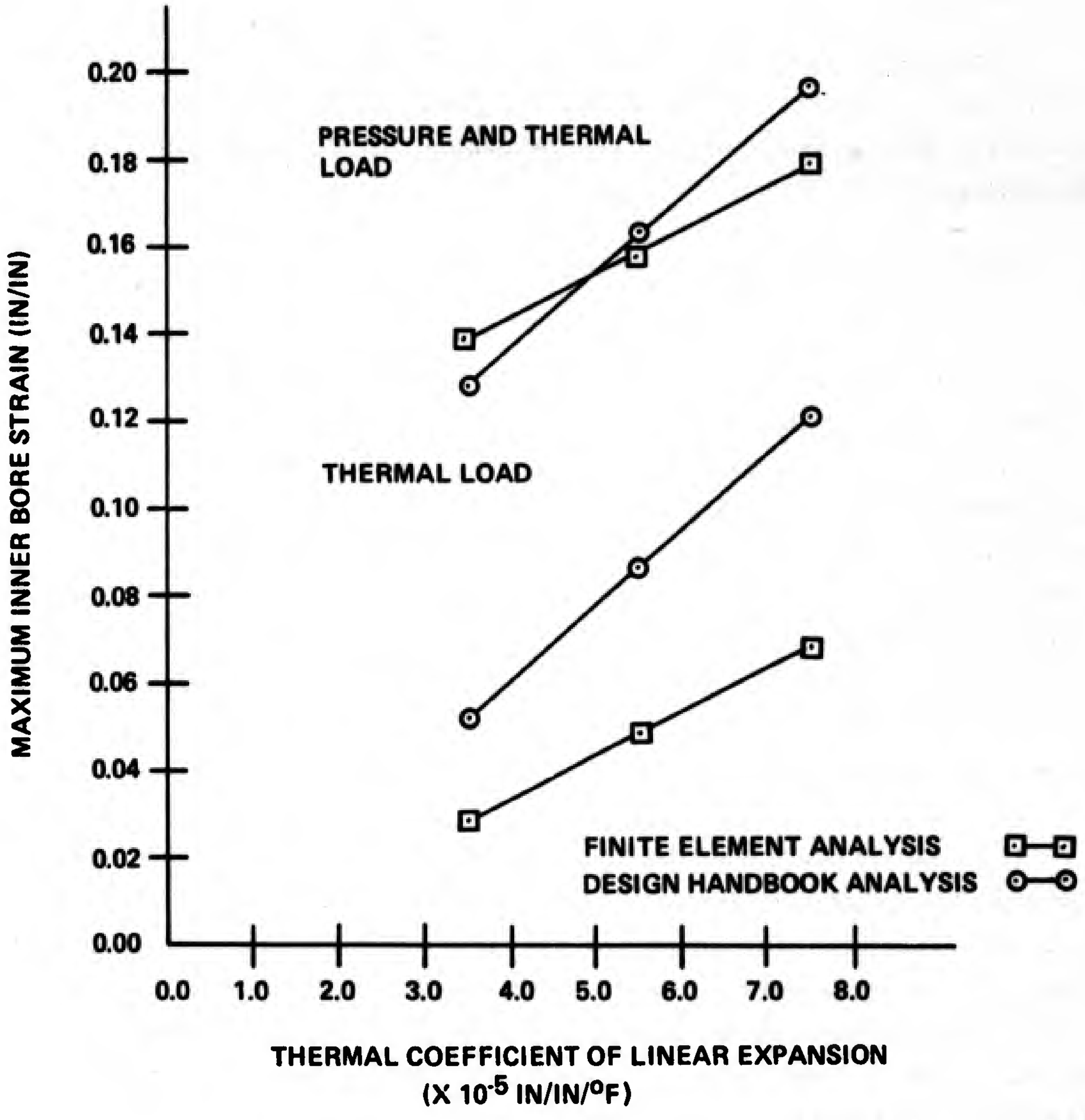

Figure 2. Maximum Inner Bore Strain versus TCLE.

SECTION III

EXPERIMENTAL STUDY

A. OBJECTIVE

Based upon the results of the sensitivity analysis (Section II), an experimental study was undertaken to determine if holographic interferometry could be used to accurately and repeatedly measure solid propellant TCLE.

B. TEST TECHNIQUE AND SETUP

An off-axis reflective holographic technique was used in this study as depicted in figure 3. To form a hologram of a propellant sample (figure 3a.), the light from a Helium Neon laser was split into two beams. One of these beams (reference beam) passed directly onto a holographic plate while the second beam (object beam) was reflected off the sample onto the plate. The interference of these two beams at the plate formed the hologram. After the hologram was exposed for the proper length of time (controlled by a shutter on the laser), it was developed and replaced in its original position. A three-dimensional image of the sample was reconstructed by illuminating the developed hologram with only the reference beam as shown in figure 3b.

If the object beam was allowed to illuminate the sample at the same time the holographic image was being viewed, the two images would coincide. The resulting image was the same as observed in figure 3b. as long as the sample had not moved. If the sample was displaced a small amount (figure 3c.), interference fringes appeared on (or near) the surface of the sample These fringes were spaced one half the wavelength of the laser beam apart (12.44 microinches) and thus gave an exact measurement of how far the sample had displaced.[1] This technique is referred to as real-time holography and has the advantage of displaying the actual sample

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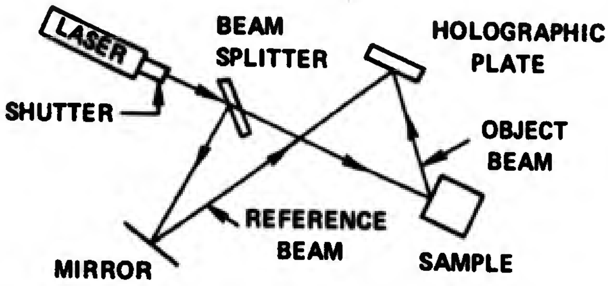
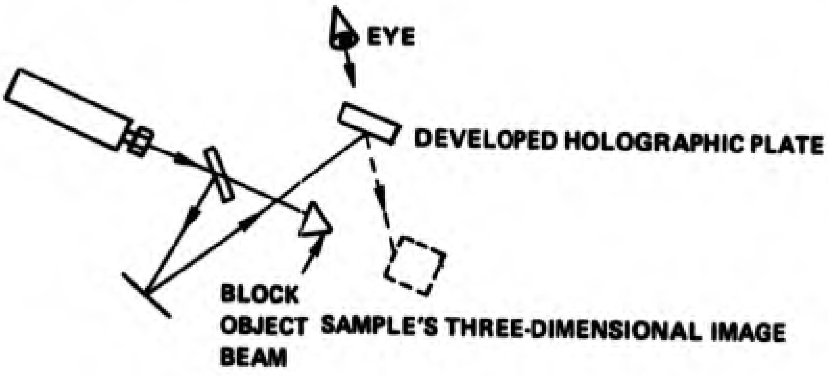
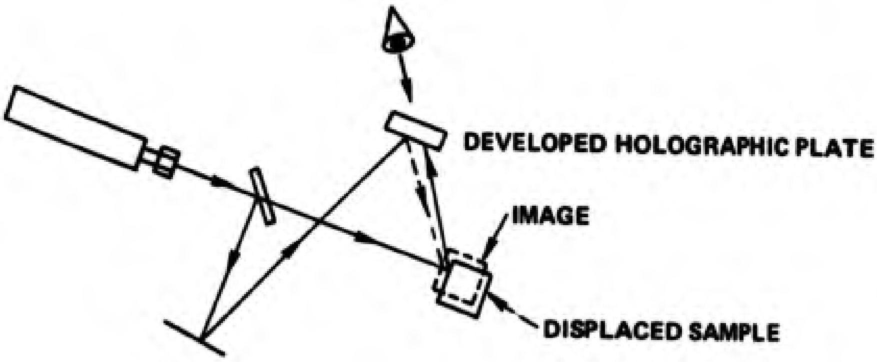
[1] References 5, 6, and 7.



(A) FORMING THE HOLOGRAM



(B) RECONSTRUCTING THE HOLOGRAM



(C) HOLOGRAPHIC INTERFEROMETRY

Figure 3. Holography and Interferometry Processes.

displacement at all times. In a similar technique called fixed-time holography, the first exposure was taken in the same manner explained above. Instead of developing the hologram plate at that time, the sample was displaced and a second exposure was taken on the same plate. The plate was then developed and replaced in its original position. The sample was removed (or object beam blocked off) and the hologram was viewed as before. A three-dimensional image of the sample was formed, but this time the displacement fringes were fixed on the sample image. Again, by counting fringes from a point on the sample that was stationary to a point that had moved, the relative displacement between the two points was calculated.

A schematic of the holographic interferometric equipment is shown in figure 4. A Spectra Physics Model 125 continuous wave laser was used for the coherent light source. Kodak 649F plates were mounted in a rigid plate holder so the developed plate could be repositioned for real-time viewing. The propellant samples (TPH-1011) were placed in a small chamber which was held at a constant temperature with heated nitrogen. One wall of this chamber contained a thin piece of glass through which the laser light was reflected off the sample. A small Iron-Constantan Thermocouple was attached to the side of the sample with a flexible silicone adhesive. A six window Hewlett Packard digital voltmeter was used to record the thermocouple output.

To eliminate external thermal and vibrational effects, the propellant sample was placed on a graphite block (figure 5). Silicone vacuum grease held the back of the sample to the graphite block. This allowed the sample to expand in the X, Y and Z directions while restricting the back surface to movement in the Y-Z plane only. Since the linear expansion of the sample was measured in the X direction, the graphite base served as a zero reference (the TCLE of graphite is approximately ten times smaller than that of the propellant).

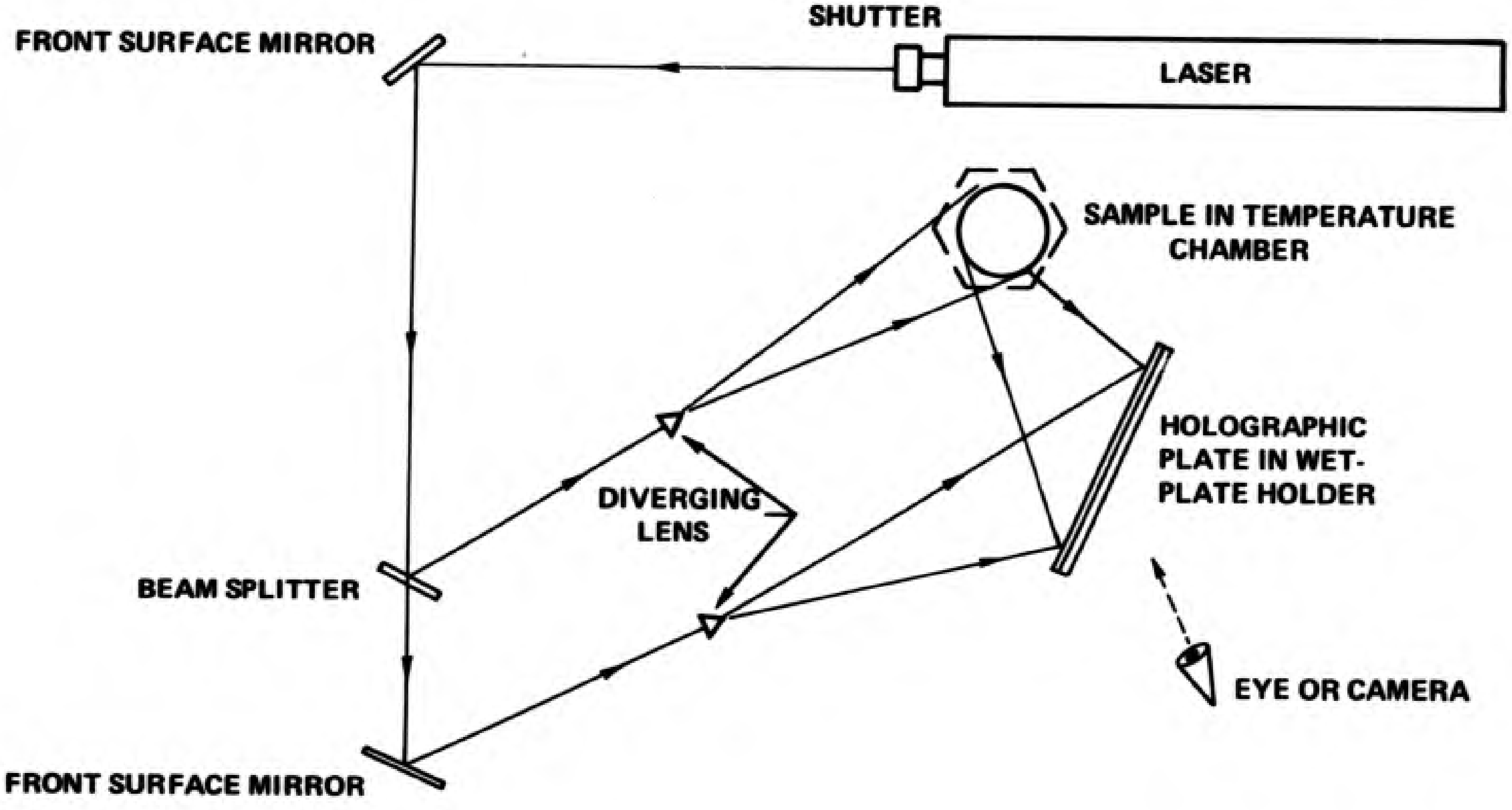


Figure 4. Holographic Interferometry Schematic.

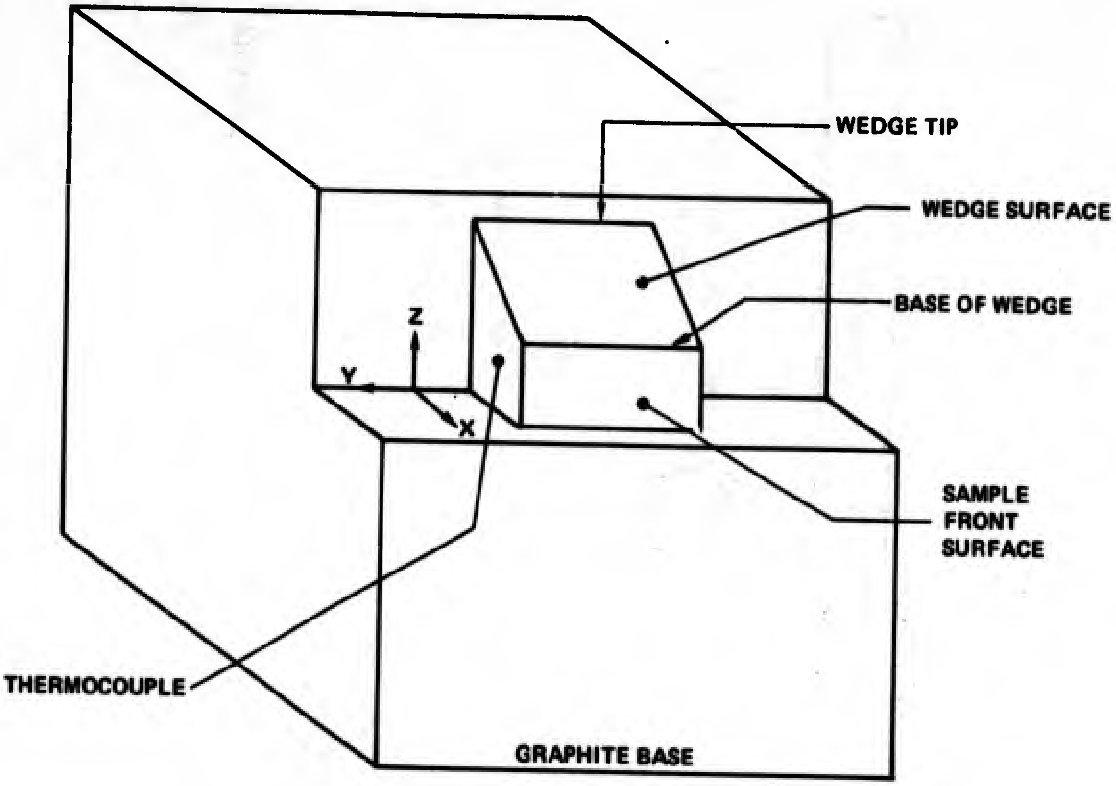


Figure 5. Propellant Sample and Base.

C. SAMPLE CONFIGURATION

As discussed previously, to utilize the holography interferometric technique to quantitatively measure an object's displacement,a zero reference point or plane must be established on that object (Reference 8). With this in mind, a wedge shaped sample was designed as shown in figure 5. By holding the back surface of the sample fixed (attached with grease to the graphite base) during expansion, the wedge tip did not move in the X direction since it has no mass to expand in that direction. The sample's thermal expansion in the X direction was therefore quantitatively measured by counting fringes along the face of the wedge (wedge tip to base of wedge) and multiplying this number by 12.44 microinches (the distance between fringes).

D. TEST PROCEDURES

The laser was allowed to warm up for at least 30 to 45 minutes prior to testing. This insured that the emitting beam was both monocromatic and coherent. At least an hour was allowed for the propellant to heat a few degrees above room temperature. The sample then equilibrated for an additional 30 to 45 minutes. This additional time was required because the sample would slump after it had been heated (or moved). This relaxation would produce several times the displacement fringes that a 3 or 4 degree temperature change would produce.

During the equilibration period, the optics were aligned and the holographic plate was placed in water for at least 5 minutes. The wetted plate was then placed in the plate holder which also contained water. The water neutralized the plate emulsion so that there would be no emulsion deformation during development. This deformation would produce unwanted fringes in the hologram image during reconstruction.

Once the sample had equilibrated, a two to three second exposure was taken of the sample and the thermocouple output was recorded. The temperature of the heated nitrogen was then reduced from between 1.0 to 3.6°F and the sample was left to equilibrate for at least another hour. At the end of this period, a second exposure was taken and the final temperature recorded. The hologram plate was then developed by standard techniques and replaced in the wet plate holder. The sample was covered so its image would not interfere with the hologram image and the hologram was reconstructed with the reference beam. If there were no more than one or two fringes on the front surface of the sample and several parallel fringes appeared on the wedge surface (figures 6 and 8), then data was taken from the hologram (number of fringes on wedge surface were counted). If several fringes appeared on the front surface or if the fringes on the wedge surface were not uniformly spaced (see figure 7), then the whole sample either rotated or had not heated uniformly (or both) and the resulting data were rejected (References 9 and 10).

A wedge shaped aluminum sample (TCLE of 1.3×10^{-5} in/in/°F) was placed in the test chamber along with the propellant sample (see figures 6 and 7) and its temperature was monitored with a thermocouple. The fringe order and ΔT of this reference sample were recorded for each test along with the propellant sample data.

To obtain a standard two-dimensional picture of the sample's image and respective fringe pattern (figures 6, 7 and 8) a camera was placed in the plane of the viewing eye (see figure 4).

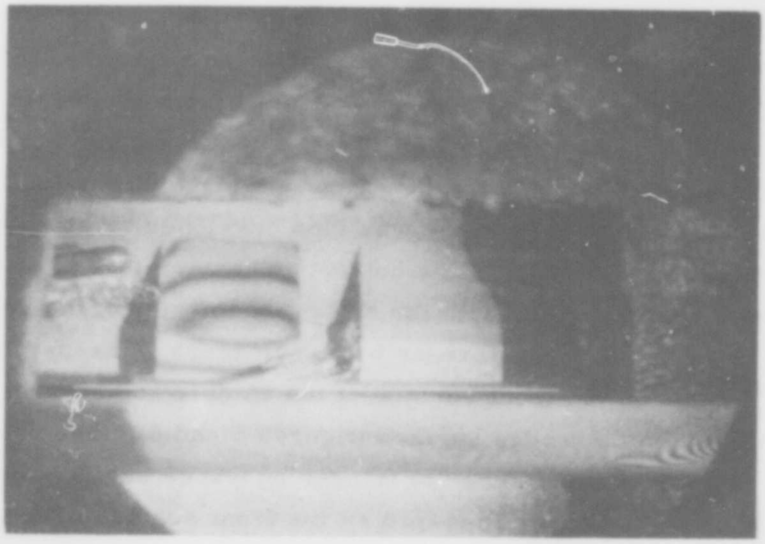


Figure 6. Fringe Pattern for Uniformly Expanded Sample

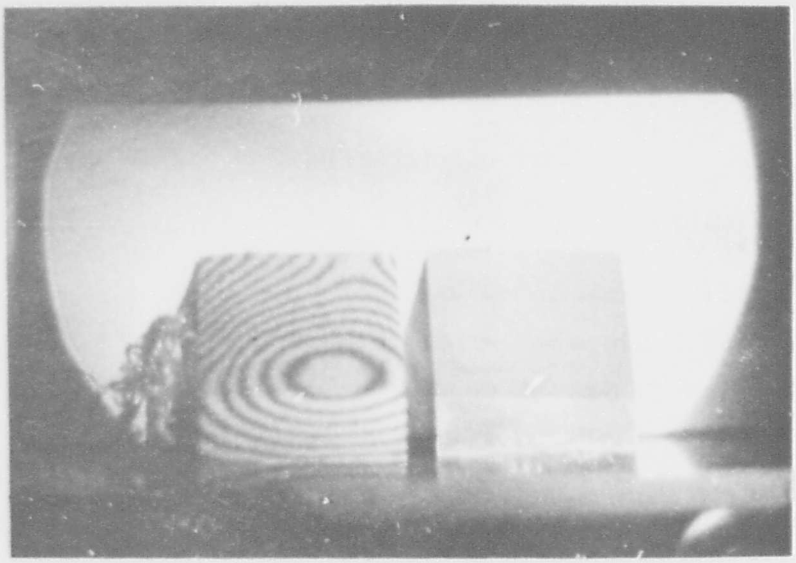


Figure 7. Fringe Pattern for Nonuniformly Expanded Sample.

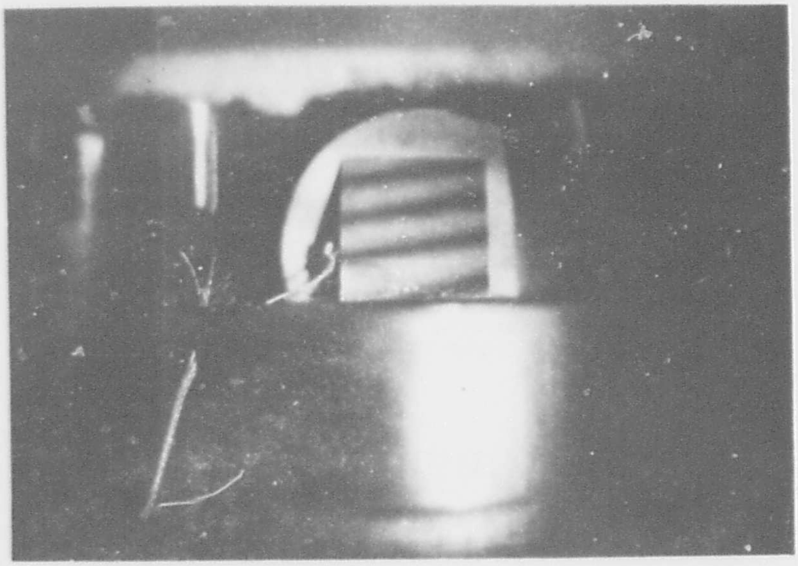


Figure 8. Fringe Pattern for Uniformly Expanded Sample.

E. DATA ANALYSIS

The change in an object's length due to a thermal change can be calculated by:

$$\Delta \ell = (\ell)(\alpha)(\Delta T)$$

where

$\Delta \ell$ = change in thickness in X direction.

ΔT = change in temperature.

α = TCLE.

In holographic interferometry, fringe patterns are produced if the object is displaced between exposures. This displacement is related to the fringe pattern by (References 6 and 11):

$$\Delta \ell = \frac{\eta \lambda}{2 \cos \theta}$$

where

η = Number of fringes (fringe order).

λ = Wavelength of laser light.

2θ = Angle between incident and reflected light rays (off object's surface).

By equating these two equations and solving for α, we find:

$$\alpha = \frac{\eta \lambda}{(\Delta T)(\ell)\, 2 \cos \theta}$$

where

$$\theta = 16 \text{ degrees } (\cos\theta = 0.96)$$

$$\ell = 0.5 \text{ inch}$$

$$\lambda = 6328\text{Å} = 24.88 \times 10^{-6} \text{ inches}$$

Putting these values into the equation for α we find:

$$\alpha = \frac{\eta}{\Delta T}\,(25.92 \times 10^{-6}) \text{ in/in/}^{\circ}\text{F}$$

Therefore, the thermal coefficient of linear expansion was directly related to the holographic fringe order and inversely related to the temperature change.

F. RESULTS

During the period covered by this report, only a limited number of holograms were taken. Eight of these holograms displayed uniform fringe patterns and were therefore used to obtain the data shown in Table II.

TABLE II. MEASURED TCLE VALUES FOR TPH-1011 PROPELLANT.

Hologram No.	TCLE (in./in./$^{\circ}$F x 10^{-5})
171	7.20
172	7.20
182	4.32
188	4.80
189	6.00
190	4.32
191	7.20
192	7.20

With the limited data shown in Table II, no definite conclusions were drawn. These measurements are within the range of TCLE values currently in use, but they too vary a great deal. They do indicate a trend toward a higher TCLE value (7.2 x 10^{-5}), but many additional tests need to be performed to determine the significance.

Since the temperature change was limited to 3.6 degrees Fahrenheit or less, (due to uneven fringe patterns at higher ΔT'S), the resulting thermal expansion and fringe order were quite small. The fringe order varied from two to six fringes for the respective temperature changes of 9.0 and 3.6°F. The errors introduced when resolving these smaller fringe orders are fairly large. A resolution of one-half a fringe for a fringe order of 6 would produce an error of approximately 15 percent for a ΔT of 3.5°F. The resolution was about one-half a fringe for the fringe order of six, but improved somewhat for the smaller fringe orders. The maximum resolution error introduced in the TCLE values listed in Table II was estimated to be approximately 15 percent.

Due to the small ΔT mentioned above, no meaningful fringes were observed on the aluminum wedge sample (ideally 1.08 fringes would form on this sample for the 3.6°F ΔT). As the temperature change was increased above 3.6°F, nonuniform fringes again appeared on the sample and rendered the resulting data useless.

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

Linear stress analyses are sensitive to variations in TCLE. Calculated maximum inner bore strains can vary up to 60 percent over the currently acceptable range of TCLE.

Laser Holographic Interferometry can measure solid propellant TCLE, but the technique is quite sensitive to propellant nonuniform heating and stress relaxation. TCLE values (for TPH-1011) measured with holography were well within the currently acceptable range, but the current effort did not narrow this range appreciably.

Additional work needs to be performed to verify the technique on metal samples that have known values of TCLE. In addition, the technique should be refined so that it can measure thermal expansions during larger temperature differentials (say ΔT'S of 10 to 15°F). This would improve the accuracy and I feel sure, the repeatability of this method.

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