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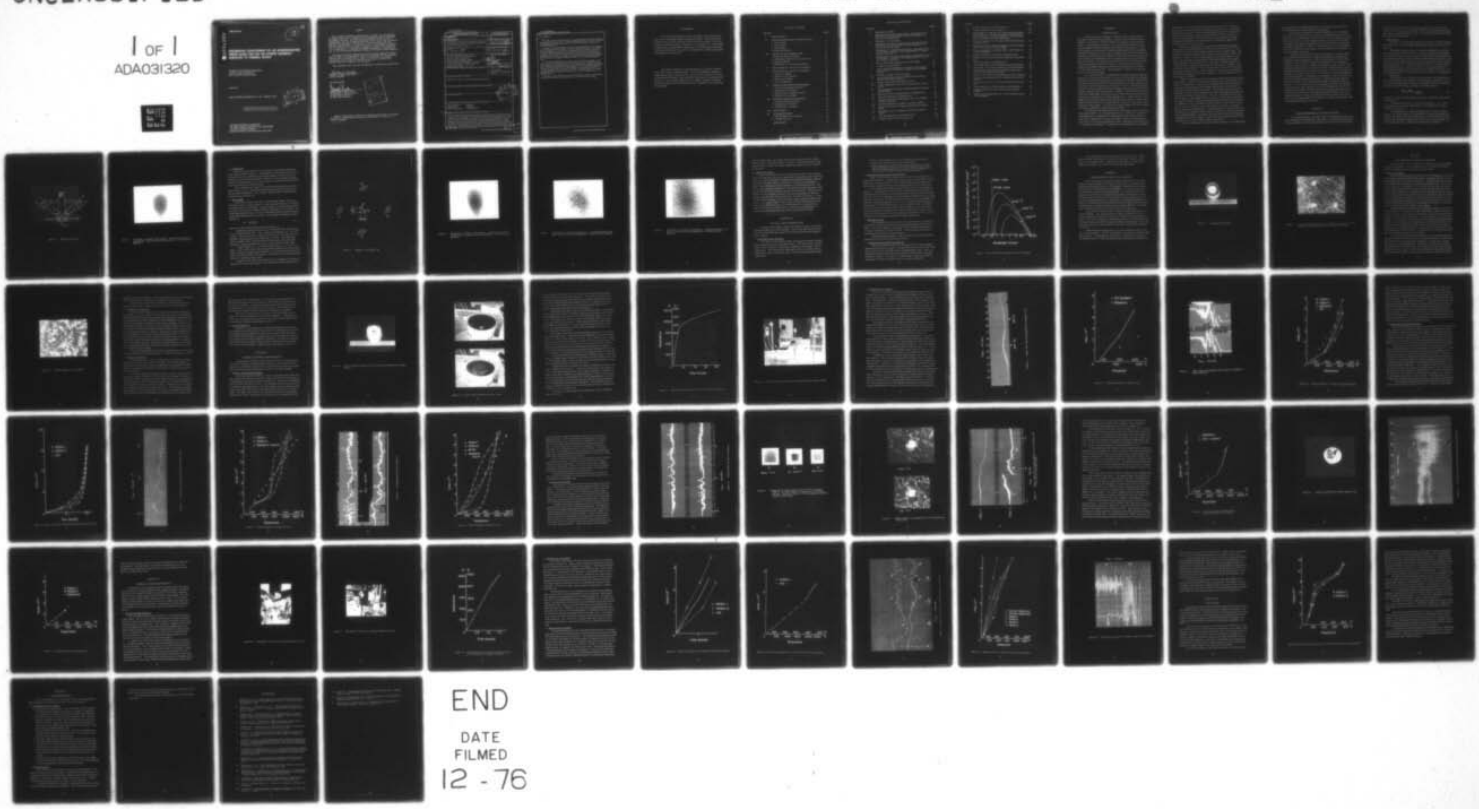

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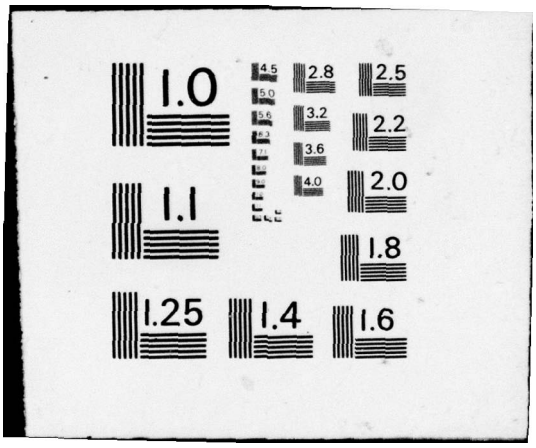
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PRELIMINARY DEVELOPMENT OF AN INTERFEROMETRIC STRAIN GAGE FOR USE ON NOSETIP MATERIALS SUBJECTED TO THERMAL SHOCK

DIVISION OF ENGINEERING RESEARCH
MICHIGAN STATE UNIVERSITY
EAST LANSING, MICHIGAN 48824

JUNE 1976

FINAL TECHNICAL REPORT MAY 1, 1975 - MARCH 31, 1976

AIR FORCE MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
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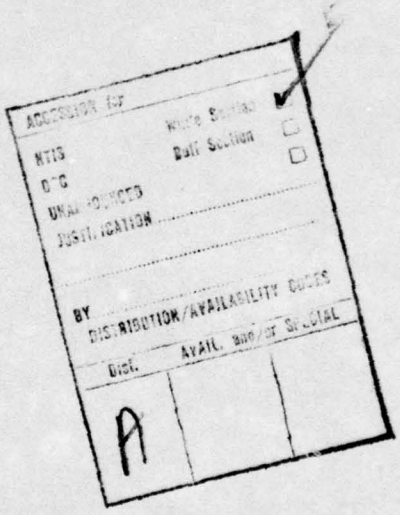

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This report describes the results of a limited effort to extend the capabilities of the interferometric strain gage (ISG) to high temperatures at high heating rates on graphite and tungsten. The specified temperature limit was 2500°F, which was to be reached in a time of approximately two seconds. Experiments were conducted using a specially constructed furnace at Michigan State University to evaluate various facets of the technique, and the high heating rate experiments were conducted at Southern Research Institute.

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20. Abstract

The ISG is based on the motion of laser-generated interference patterns from the specimen surface. If suitable reflecting surfaces can be made that will withstand the high temperature environment, then the technique has great potential.

Tungsten is easily polished, and reflecting indentations can be applied with a Vicker's hardness tester. However, tungsten oxidizes very rapidly and the technique can be used for heating tests only up to about 800°F in air. The temperature limit can be extended by using a less oxidizing atmosphere. The oxidation and background radiation at 2500°F would be the limiting factors on use of the ISG on tungsten, but the background radiation can be reduced with better interference filters over the photomultiplier tubes.

Reflecting indentations cannot be formed directly in graphite, so another material must be attached to the specimen surface and the indentations applied to it. Platinum/10-percent-rhodium was evaluated and found to be an excellent material for this purpose. It does not oxidize enough to destroy its reflections under short exposure (ten minutes) to 2500°F. A ceramic adhesive was evaluated as a means of attaching Pt/10% Rh tabs to graphite and was found to be satisfactory for slow heating, but not for thermal shock.

Details of the techniques and instrumentation, as well as descriptions of the various evaluative experiments, are included in this report.

FOREWORD

This report describes the efforts to develop a laser-based strain gage for use on graphite subjected to thermal shock. The work was performed under Air Force Contract F 33615-75-C-5230 with Lt. Terry Hinnerichs as technical monitor. Professor W. N. Sharpe, Jr., of the College of Engineering, Michigan State University, was the principal investigator. The project period was 1 May 1975 to 31 March 1976, and the draft report was submitted 17 March 1976.

The author would like to acknowledge the very capable assistance of Mr. Rick Tonda, graduate student. Dr. Glenn Hollenberg originally suggested the general research area, and Lt. Terry Hinnerichs has been an interested and helpful contract monitor; their interest is gratefully appreciated. The excellent cooperation of a number of people at Southern Research Institute under the direction of Mr. S. Starrett is also gratefully acknowledged.

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SECTION I

INTRODUCTION

Thermostructural technology of reentry vehicle nosetips requires knowledge of the material properties, computer codes for predicting the response of structures, and test facilities for validation of the design procedures. Experimental techniques for measuring deformation enter into this technology in both the property measurement and test phases. Strain or deformation measurements for property determination are considerably less difficult than those for structural testing because one has the option of choosing a convenient specimen shape. In both cases the measurement problem is made more difficult because of the high temperatures and high heating rates involved. The purpose of this research is to develop and evaluate a laser-based interferometric technique for measuring biaxial strain on tungsten and graphite specimens subjected to temperatures up to 2500°F for very short periods of time. This technique, if feasible, has potential for use in structural testing.

In a recent survey[1] of techniques for measuring strain at high temperature for long times (100 hours or more), it was concluded that suitable techniques for use at temperatures above 1100°F do not exist. Considerable research is under way on this kind of measurement, and some progress has been made since the preparation of that report in 1973. The difficulty with long time measurements is the drift of the gage with time; the drift is caused by phase changes of the gage material, oxidation, and other thermal effects. These same difficulties are not as severe for short time measurements, but problems of response time, thermal expansion, etc., become severe. The gages examined in that report mainly fall into three classes: extensometers (both electromechanical and electro-optical), resistance strain gages, and capacitance strain gages. It is useful to consider gages in the same classes that may be applicable to high-heating-rate testing.

Electromechanical extensometers are not usually used for temperatures above 1800°F. The transducing element (an LVDT or resistance strain gage) that is attached to the extensometer arms must be kept at a much lower temperature for accurate results. However, extensometers have been used to measure deformation inside a nosetip as it was subjected to a rapid thermal loading.[2] Probably the most applicable electro-optical

extensometer is that developed by Babcock and Hochstein.[3] They used an argon laser whose beam was rotated across a gage section. The edges of the gage section were defined by sputtering a thin film of refractory material onto the specimen. Strain measurements on ATJ-S graphite to 5500°F at heating rates of 100°F/second were made with this technique. It would be very difficult, if not impossible, to extend this approach to strain measurement on structures.

Resistance strain gages have been successfully used to measure static strains at temperatures up to 1500°F for at least one hour.[4] Presumably they could be used at higher temperatures for shorter times. The bonding of resistance strain gages becomes a problem at these higher temperatures; ceramic cement or spotwelding of encapsulated gages is often used. The relatively new capacitance strain gages appear promising for high temperature strain measurement. One kind, the Hughes gage,[5] is usable for static strain measurement up to 1750°F and for transient strain measurement up to 2000°F. It has a gage-length of only 1/2 inch and may be attached by spotwelding, ceramic adhesive, or flame-spray techniques. None of the resistance or capacitance gages are at present capable of measuring strain at 2500°F.

Moiré techniques can be used to measure whole-field strains at high temperatures. Durelli[6] describes moiré techniques used at 1100°F for times up to 1000 hours. However, the techniques developed by Neudecker[7] are much more applicable to thermostructural problems of nosetips; he measured strains at 2500°F on graphite. The specimen grid was formed by gluing a commercially available nickel grid to the graphite specimen and curing it at an elevated temperature. The cure caused the nickel grid to form carbides on the graphite specimen surface which produced a grid that was usable to 2500°F. He illuminated the specimen with a 1 watt argon laser (4880 A°), but he also obtained visible fringes at 2370°F with a 1 milliwatt He-Ne laser (6328 A°). His specimen was enclosed in a resistance furnace with an argon atmosphere. He measured the coefficient of thermal expansion of ATJ graphite and the strains at 2200°F in a diametrally loaded ring of H4LM graphites. Neudecker did not continue with the development and application of this technique, but his work demonstrates that the moiré technique can be used at 2500°F on plane specimens.

The laser-based speckle interferometry technique is currently under

investigation for use at high temperatures.[2] Many of the procedures used by Neudecker would be useful in this approach.

None of these techniques is entirely satisfactory; it is expecting too much to hope that strain measurements at high temperature will ever be as easy as room temperature measurements with the ubiquitous foil gage. This report describes a first attempt to extend a novel laser-based interferometric strain gage to high temperatures measurements--specifically on graphite and tungsten at 2500°F. From this report one should expect more of an indication of the potential of the technique than a description of a well-developed strain gage.

The basic theory underlying the interferometric strain gage (ISG) is described briefly in Section II. Expected problems in extending this technique to high temperatures are discussed in Section III. Strain measurements on two materials--tungsten and graphite--are investigated. Tungsten is a fine-grained, isotropic, homogeneous metal, similar to the materials the ISG has been used for previously, and the problems associated with high temperature strain measurement are discussed in Section IV. Graphite is a large-grained material that requires a large gage length for useful measurements. Furthermore, the graphite is not as reflective and this causes another significant problem. A description of the approach taken to overcome these measurement problems in graphite is given in Section V. The first tests were fairly straightforward thermal expansion measurements on both materials; these are described in Section VI. The critical measurements--of biaxial strain on thermally loaded specimens of both kinds--were performed at Southern Research Institute and are presented in Section VII. These are preliminary measurements, and conclusions as to the applicability of the technique are drawn in Section VIII, with recommendations given in Section IX.

SECTION II

THE INTERFEROMETRIC STRAIN GAGE (ISG)

The interferometric strain gage (ISG) is a unique laser-based technique developed at Michigan State University. Its main features are that it is noncontacting, has a short gage length, and can operate in a hostile

environment. Its small size has made it useful for the measurement of displacements near fatigue crack tips,[8] while its noncontacting characteristic offers a potential solution to some of the strain measurement problems discussed in the Introduction.

1. Basic Theory

The ISG can be understood with the aid of Figure 1 and the following brief discussion. A more complete description of it is given in a review paper (9).

Two wedge-shaped grooves or pyramidal indentations are pressed into the specimen surface at A and B. These two grooves or two indentations form the "gage," and each one has two sides that reflect the incident laser light onto screens L and R located symmetrically at an angle α_o. The grooves are fairly shallow (on the order of 5 microns deep), thus the light reflected from the sides is spread by diffraction effects, with the zeroth order appearing at α_o. Because A and B are close enough together for their diffraction patterns to overlap and the laser light is coherent, interference fringe patterns are formed at L and R. Figure 2 is a photograph of a fringe pattern.

If the distance, d_o, between A and B changes, the fringe patterns at L and R will move with respect to fixed observation positions at L and R. If this fringe motion is measured as a fraction of the spacing between fringes and is labeled ΔM_L and ΔM_R, the simple equation:

$$\epsilon = \frac{\Delta M_L + \Delta M_R}{2} \frac{\lambda}{d_o \sin \alpha_o} \qquad (1)$$

in which λ is the wavelength of the laser light, relates the fringe motion to the strain ϵ.

For typical values of d_o = 0.016 inch (0.4mm), α_o = 45°, and λ = 0.633 microns, an average fringe shift of 1 corresponds to a strain of 2.2×10^{-3} or 2,200 $\mu\epsilon$.

The indentations are typically applied with a Vicker's hardness tester. The fringe motions are monitored by mounting a photodetector covered by a narrow slit in the fringe pattern. As the fringes pass over this slit, the intensity of light on the detector varies; this variation can be recorded and used to compute the fringe shifts in Equation 1.

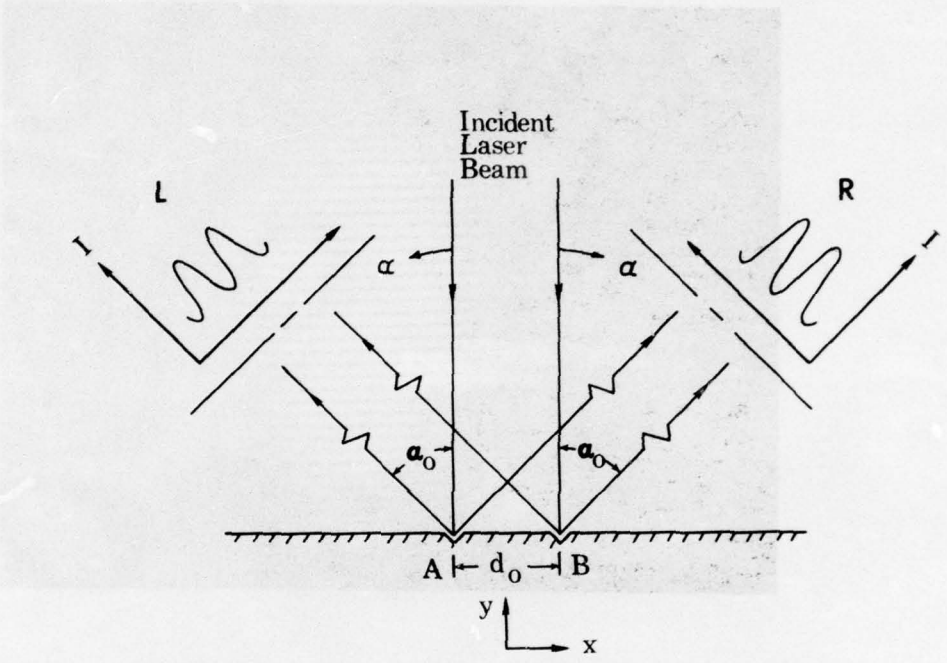


Figure 1. Schematic of the ISG.

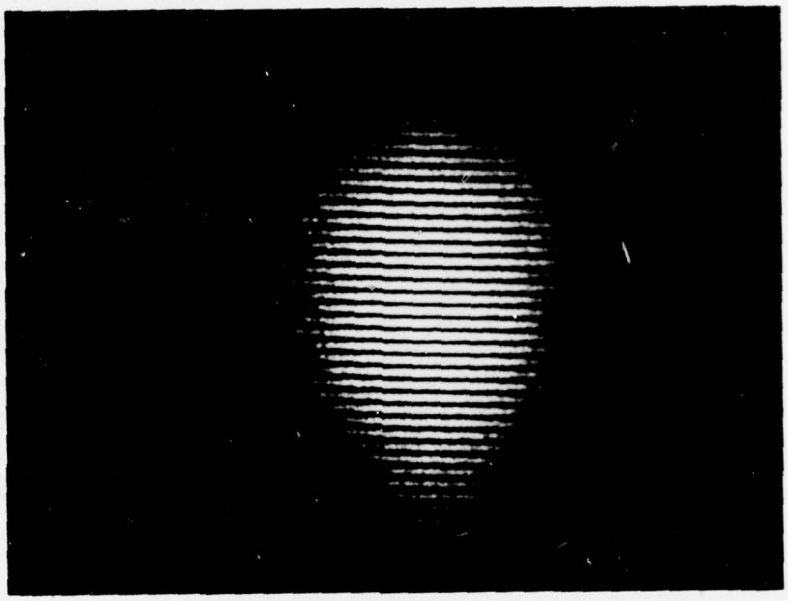


Figure 2. Photograph of uniaxial fringe pattern. Indentations were 400 microns apart, and pattern was photographed at 1.1 meter from the specimen.

2. A Biaxial ISG

Strain in two directions can be measured by arranging indentations in the manner shown in Figure 3. Photographs of fringe patterns are given in Figure 4 and it is seen that the continuous fringes are broken up by interference from the third indentation. However, if one positions the photodetector slit perpendicular to the strain direction, fringe motion (and strain) will only be measured in that direction.

The configuration of Figure 3 is useful if the principle strain direction is known. If not, a general biaxial strain gage can be constructed using six-sided indentations. This concept, along with other considerations, is discussed in reference 10.

3. Gage Length

One of the features of the ISG is its short gage length. There are situations, such as measurements on a coarse-grained material, when that is a disadvantage. In principle, the gage length can be made approximately as large as the coherence length of the laser (typically 1 cm or larger). But as the gage length increases, the spacing between fringes becomes smaller; fringes then become harder to resolve. The equation governing the fringe spacing is:

$$\Delta\alpha = \frac{\lambda}{d_o \cos \alpha_o} \qquad (2)$$

where $\Delta\alpha$ is the angle between fringes observed at α_o.

Indentations produced by the Vicker's hardness tester diamond have an α_o of approximately 45°. If d_o = 0.016 inch (0.400 mm), $\Delta\alpha = 2.2 \times 10^{-3}$ radians. This means that at a distance of 20 inches (51 cm) from the specimen the fringe spacing will be 0.044 inches (1.1 mm). This spacing is quite acceptable since a slit 0.010 inch (0.25 mm) wide lets enough light through to a typical photodetector to give a good signal-to-noise ratio.

A gage length of d_o = 0.25 inches (6.4 mm) has $\Delta\alpha = 1.4 \times 10^{-4}$ radians. At 20 inches (51 cm), the spacing will be 0.0028 inches (70 microns), which is barely resolvable to the naked eye. One must then extend the distance from the specimen to the detector. This reduces the intensity into the detector, requiring a larger laser.

Figures 5 and 6 show fringe patterns from indentations 0.2 inch (5.1 mm) and 0.1 inch (2.5 mm) apart. This brings up another problem with

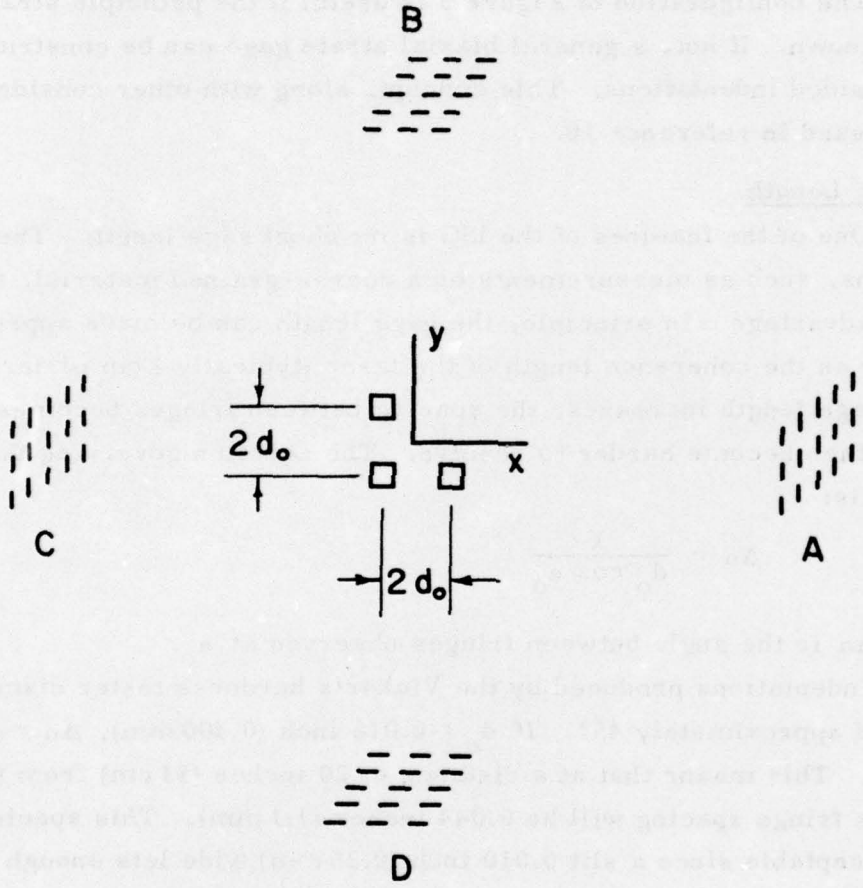


Figure 3. Schematic of a biaxial ISG.

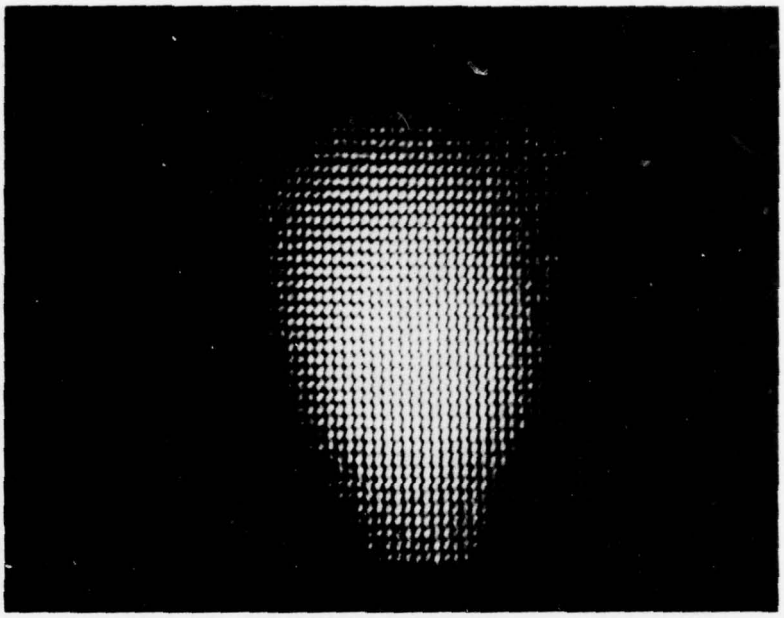


Figure 4. Photograph of a biaxial fringe pattern. Indentations were 400 microns apart, and pattern was photographed at 1.1 meter from the specimen.

Figure 5. Photograph of a uniaxial fringe pattern. Indentations were 5.1 mm apart, and pattern was photographed at 2 meters from the specimen.

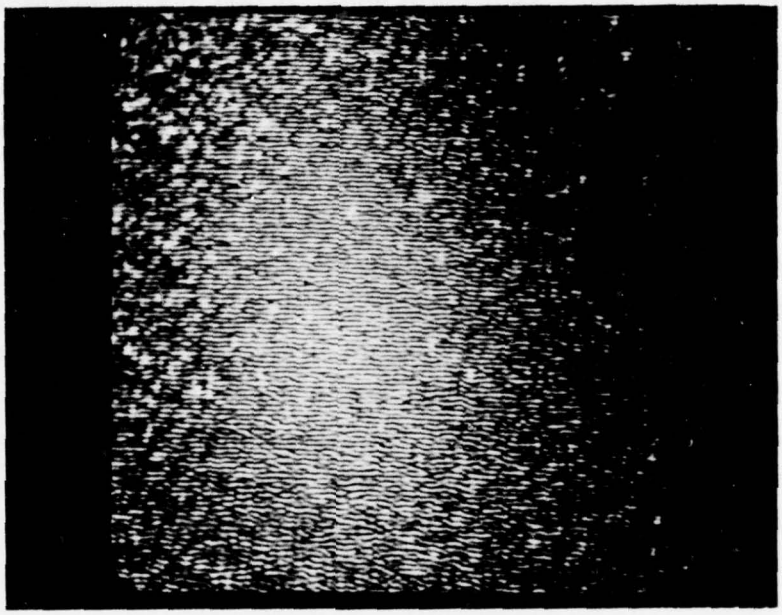


Figure 6. Photograph of a uniaxial fringe pattern. Indentations were 2.5 mm apart, and patterns were photographed at 2.3 meters from the specimen.

increased gage length; the fringes become small and on the order of the laser speckles as the gage length is increased. Measurements were made at both of these lengths, but a gage length larger than 0.2 inch (5.1 mm) is not practical.

4. Rigid Body Motions

If the fringe patterns move as the specimen strains, they will also move if the specimen moves as a rigid body. A discussion of the rigid body motion is given in reference 9, but the important points should be mentioned. Averaging the fringe shifts as in Equation 1 cancels any contribution to fringe motion of displacements in the plane of the specimen surface. However, displacements perpendicular to the specimen surface generate fringe motions that are not canceled out and thus lead to errors. If the specimen surface moves vertically enough to cause the fringe pattern to shift one spacing, then the ΔM values in Equation 1 would be in error by 1. If only a small amount of strain is to be measured, this could lead to intolerable errors. For a gage length of 0.016 inches (0.4mm), the specimen would have to move 0.044 inches/cos 45° (0.062 inches or 1.5mm) to cause a fringe shift error of 1 at detectors 1.1 meters from the specimen. In most laboratory experiments, this much motion would not occur.

SECTION III

THE ISG AT HIGH TEMPERATURE

In spite of its potential, the ISG has not been used for even moderately high temperature measurements. This section discusses the problems associated with high temperature strain measurement and suggests some solutions.

1. Reflecting Surface Oxidation

The "gage" of the interferometric strain gage is the pair of indentations impressed into the specimen surface. The sides of these indentations must be reflective; indentations in bakelite, for example, are not satisfactory. Most metals have sufficiently reflective surfaces when indented with a Vicker's hardness tester, but all oxidize with time at high temperature.

There are two possible ways for solving this oxidation problem:

1) Protect the specimen with an inert atmosphere.

2) Attach some type of nonoxidizing tabs to the specimen and then put indentations in the tabs. The latter solution is more desirable if a suitable material can be found, small enough tabs can be made, and a suitable method of attachment devised.

2. Background Radiation and Laser Selection

Figure 7 is a plot of the radiation from a black body heated to the temperature indicated. As the body becomes hotter, not only does the magnitude of the radiation increase, but it shifts to shorter wave lengths and becomes more of a problem in the visible region between 0.4 and 0.7 microns.

Wave lengths of two of the common types of lasers are indicated in Figure 7. The ubiquitous He-Ne laser has a wavelength of 0.633 microns and argon lasers have their more powerful lines at 0.514 microns and 0.488 microns. From a consideration of the background radiation alone, one would select an argon laser. Interference filters with very narrow bandwidths are available, and in principle one can filter most of the background radiation. Neudecker (7) viewed moiré fringes at 2370°F (1300°C) with a 1 milliwatt He-Ne laser and interference filters, but changed to a 1.1 watt (at 0.488 microns) argon laser to obtain fringe patterns with better contrast. Argon lasers are more powerful (and more expensive) than He-Ne lasers and clearly are the best choice for the ISG at high temperatures.

3. Specimen Heating

If the specimen is to be heated in an enclosed furnace, then optical ports for the incident laser beam and exiting fringe patterns must be provided. This is no problem as quartz ports with a melting temperature on the order of 4000°F are easily available. An enclosed furnace offers the possibility of providing an inert atmosphere.

If high-frequency heating is used, then the coils must be located to provide for the laser beam and fringe patterns.

4. Photodetectors and Recording Systems

With a 5 milliwatt He-Ne laser, the intensity of the fringe pattern passing through a slit in a typical arrangement is on the order of 10^{-9} watts. This is easily in the sensitivity range of photomultiplier tubes. Silicon photodiodes will measure intensities this small, but not with a very high frequency response. If one goes to a higher-power argon laser, then the photodiodes are quite satisfactory from a sensitivity viewpoint.

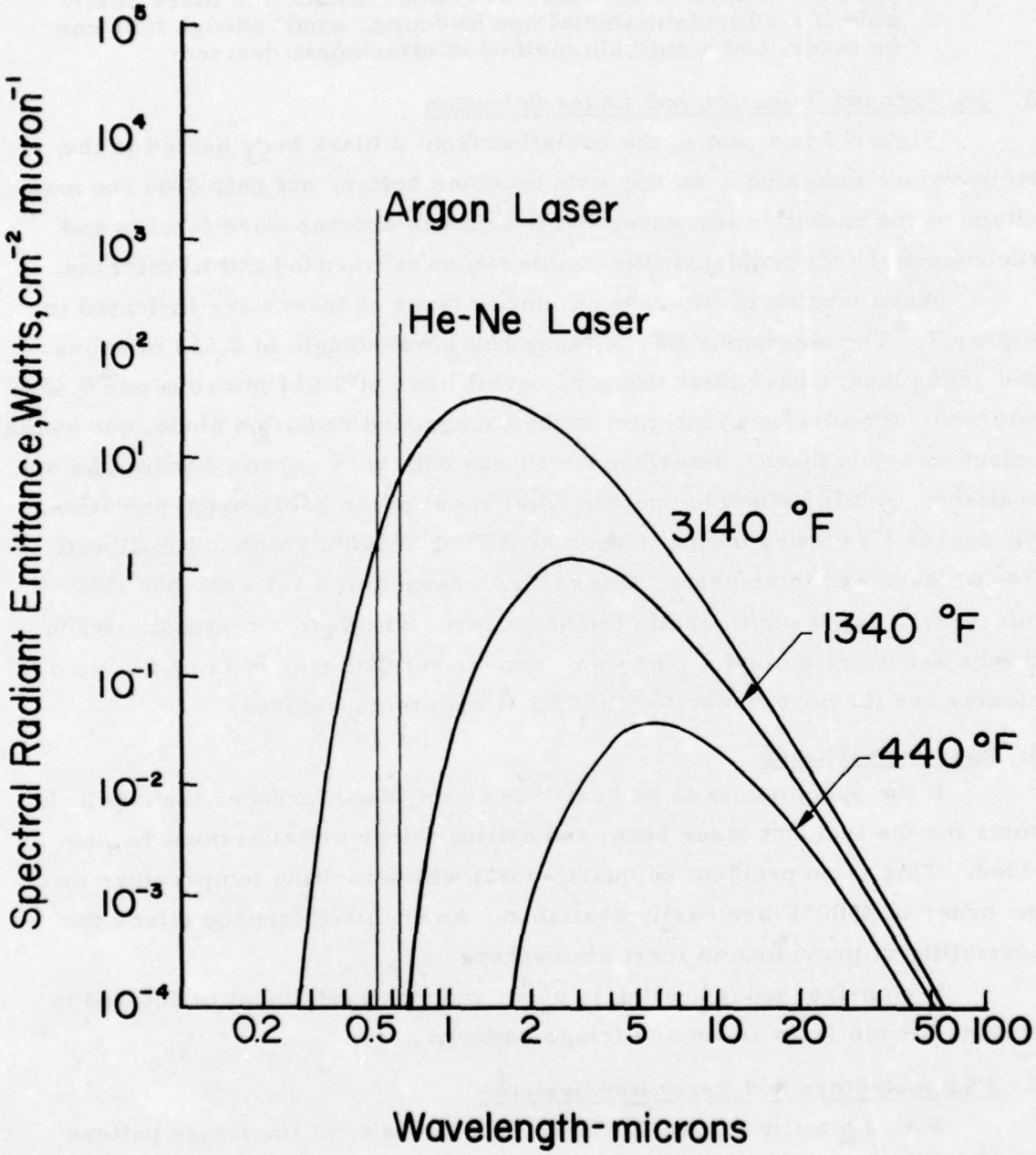


Figure 7. Plot of black-body radiation versus wavelength.

The recording system requirements are not very severe. Strip-chart recorders with frequency responses on the order of 40 Hz are adequate for most experiments. Light beam oscillographs, or other higher frequency recorders, are required for transient experiments.

SECTION IV

APPLICATION OF THE ISG TO TUNGSTEN

It is very easy to use the ISG on tungsten specimens at room temperature because tungsten is a reflective metal that can be polished and indented by standard procedures. The tungsten specimens were polished with 320, 400, and then 600 grit wet sandpaper. The last sanding process was followed by a polish with diamond paste. It is necessary to get the specimen surface fairly smooth before applying the indentations in order to prevent stray reflections of the laser beam into the fringe patterns, but the surface does not need to be "scratch free." Polishing flat cylindrical disks for the thermal stress measurements had to be done entirely by hand. A photograph of a tungsten specimen is presented in Figure 8; the indentations are inside the black circle in the center of the specimen.

The problem in using the ISG on tungsten at high temperature is of course oxidation. The vapor pressure of oxygen in tungsten oxide is so low that sophisticated vacuum chambers are required to prevent oxide formation. The rate of oxidation is controlled by the rate of temperature rise. The usable limit of the ISG on tungsten at different heating rates was a question to be answered by this research; the results are presented in Sections VI and VII.

Indentations were applied with a Leitz microhardness tester with a Vicker's type diamond. For the hard tungsten, the heaviest standard weight was required. A photograph of a biaxial set of indentations in tungsten is presented in Figure 9. These indentations produce high quality fringe patterns.

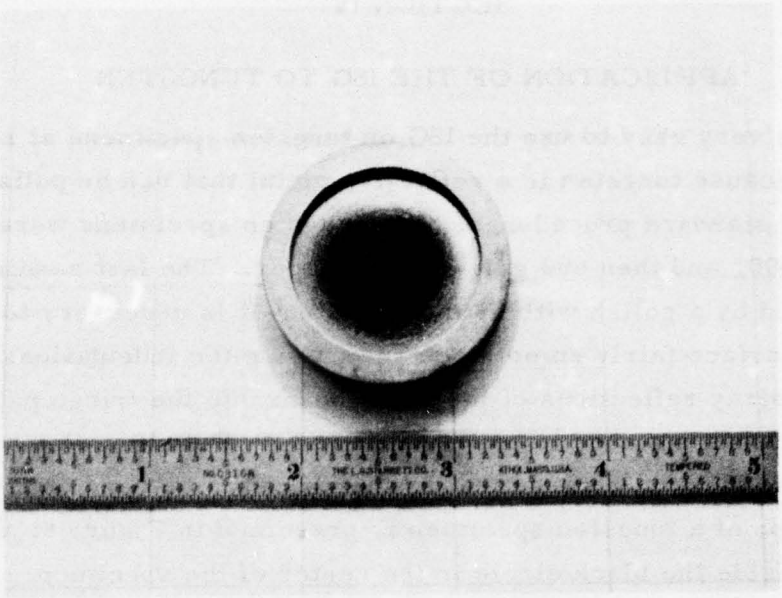


Figure 8. A tungsten disk specimen.

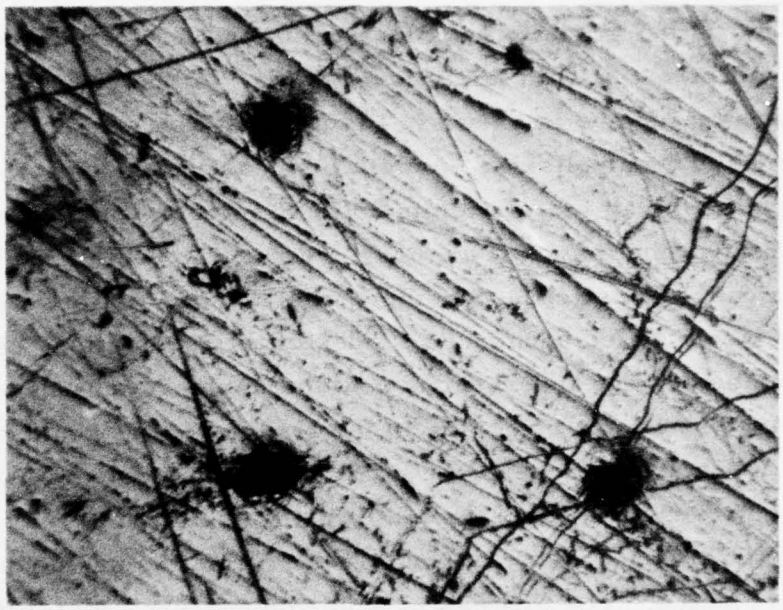


Figure 9. A set of biaxial indentations in tungsten. Good quality fringe patterns are observed in spite of the scratched surface.

SECTION V

APPLICATION OF THE ISG TO GRAPHITE

The ISG has been used on plastics (11), but not on a low reflectivity nonmetal like graphite. Use of the ISG on such a material requires that suitable reflecting surfaces be formed on the specimen.

1. Nature of Graphite

The ATJ-S graphite used for specimens is a homogeneous but coarse-grained soft material with fairly large voids. A photomicrograph of a polished graphite surface is shown in Figure 10. The specimen was prepared by sanding and then polishing on metallurgical polishing cloth. Since a typical dimension for an indentation is 25 to 40 microns on a side (see Figure 9), the voids in the material are large enough to prohibit the production of smooth-sided indentations. Attempts to make indentations with the Vicker's hardness tester failed because the sides of the indentations simply crumbled. Even if smooth-sided indentations could be formed, it is doubtful that ordinary laboratory lasers operating in the visible region would reflect enough from the indentations to generate a fringe pattern. Clearly, some kind of reflective material must be applied to the graphite.

Several schemes for producing the reflecting surfaces were considered but rejected. The process of vapor depositing a metallic (and thus reflective) coating onto the surface of indentations formed in the graphite was rejected because smooth indentations could not be produced in the crumbly graphite. If one were to coat the surface of the graphite by depositing metal onto it, one could then make indentations in the reflective coating. But attempts to make indentations in thin metal foils showed that the coating would have to be quite thick (0.010 inch (250 microns) or more) before suitable indentations could be formed. A coating that thick would reinforce the specimen and give incorrect strain readings.

Neudecker (7) generated a moiré grid by vapor-depositing tungsten through a 500 line per inch screen onto a graphite specimen. He also found that nickel wires can be attached to graphite by a simple heat treatment which forms a nickel-carbide line. These lines are reflective and when illuminated by a laser would reflect the radiation in all directions perpendicular to the line. A pair of these lines would in principle generate suitable interference fringes, but since the laser light is scattered over 180°,

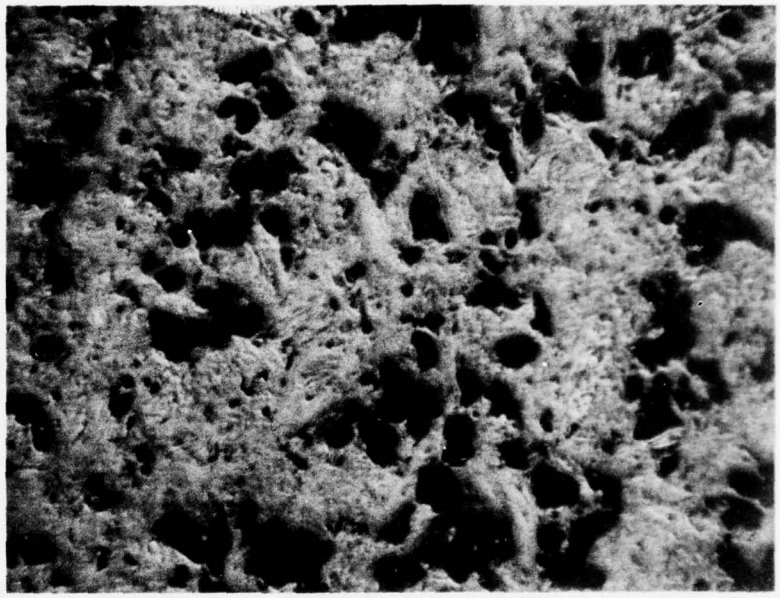


Figure 10. Photomicrograph of ATJ-S graphite.

the pattern would be quite dim. Because of this dimness and concern about the uniformity of the carbide lines, this approach was not tried.

The technique of attaching small tabs of a thin metal sheet to the specimen with ceramic adhesive was selected as the most promising.

2. Selection of Tab Material

The tab material needs to be reflective and resistant to oxidation. In addition, it must be available in thin sheets and easily cut and polished. Manufacturers of wire and foil resistance strain gages have examined many different alloys as to their suitability for high temperature gages. For resistance strain gages, the alloy must not only be oxidation resistant, but metallurgically stable to permit accurate resistance change measurements. Bertodo (12) concluded that platinum-tungsten alloys offered most promise as high temperature gages. However, based on the recommendations of a material supplier (13), platinum/10-percent-rhodium was selected as the tab material. This choice was supported by other references (14) which showed that Pt/10% Rh should be capable of standing 2500°F for eight minutes in air without oxidation. However, it was not known how much oxidation is permissible before the interference fringes deteriorate; this must be determined experimentally.

A 1 inch x 2 inch x 0.010 inch (2.5 in x 5 cm x 0.25 mm) sheet of Pt/10% Rh was purchased from Mathey-Bishop, Inc., Malvern, Pennsylvania, 19355.

3. Selection of Adhesive

High temperature resistance strain gages are attached by ceramic adhesives, flame spraying, and spot-welding. The ceramic adhesive is the only practical one of the three for attaching tabs to graphite, but adhesives for resistance strain gages are intended for temperatures less than the desired 2500°F.

A ceramic adhesive that appeared ideal is Aremco Bond 503 manufactured by Aremco Products, Inc., P. O. Box 429, Ossining, New York. It is rated at 3000°F and specifically states that it can be used on graphite as well as other materials. It requires an initial cure to 250°F followed by a cure to 1000°F to reach maximum strength. It initially has the viscosity of an ordinary paint and so is quite easy to use. After obtaining a supply of the adhesive, a tab was attached to a test piece of graphite and the test-piece heated to 2500°F and held there for approximately ten minutes and

then furnace-cooled. Examination of the test-piece showed that the tab was still firmly attached even though all of the test-piece had been severely eroded by oxidation. The platinum/10-percent-rhodium tab had a pair of indentations in it, and fringes were still visible. This simple test indicated that the proposed ISG arrangement on graphite was feasible.

The Aremco Bond 503 has a shelf-life of several months, but its properties are quite dependent on the pH of the adhesive. Toward the end of the research program some bad bonds were made, but the adhesive was reconstituted by adding orthophosphoric acid to it.

4. Gage Configuration

Figure 11 is a photograph of a dished-disk graphite specimen with two platinum tabs attached with Aremco Bond 503. The light gray material surrounding the white adhesive is a thin precoat applied to the specimen. The platinum-rhodium tabs are pressed down flat on the graphite specimen and anchored by the ceramic adhesive around them. The tabs are approximately 0.050 inches (1.25 mm) square and the indentations on the two tabs are 0.10 inch (2.5 mm) apart. A biaxial gage would require a third tab.

SECTION VI

THERMAL EXPANSION MEASUREMENTS

Experiments in which the specimens were heated at relatively slow rates were conducted at Michigan State University. The purpose of these tests was to evaluate the various aspects of the technique before proceeding with the thermal loading experiments at Southern Research Institute.

1. Furnace and ISG Instrumentation

It was necessary to construct a furnace at Michigan State University that would permit entrance of the laser beam and exit of the fringe patterns. A photograph of the furnace is shown in Figure 12. Heating is accomplished by four Norton "Hotrods" in series. The top walls of the uncovered furnace are sloped at approximately 45°; when the specimen sits on a firebrick pedestal in the center of the furnace the fringe patterns just clear the furnace walls. The sides of the furnace are insulated with Kaowool covered with Alundum 485 cement. Temperature is measured by a sheathed Pt/10% Rh

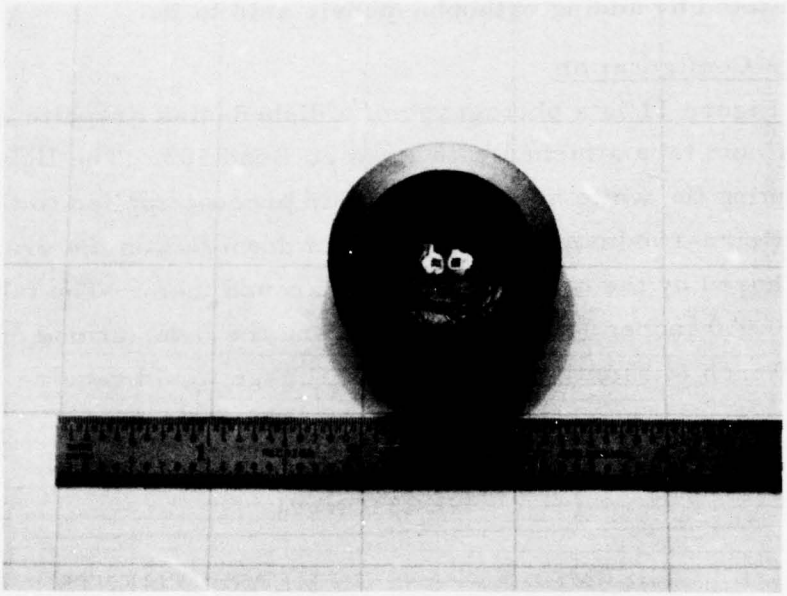


Figure 11. An ATJ-S graphite specimen with Pt-10% Rh Tabs attached with ceramic adhesive.

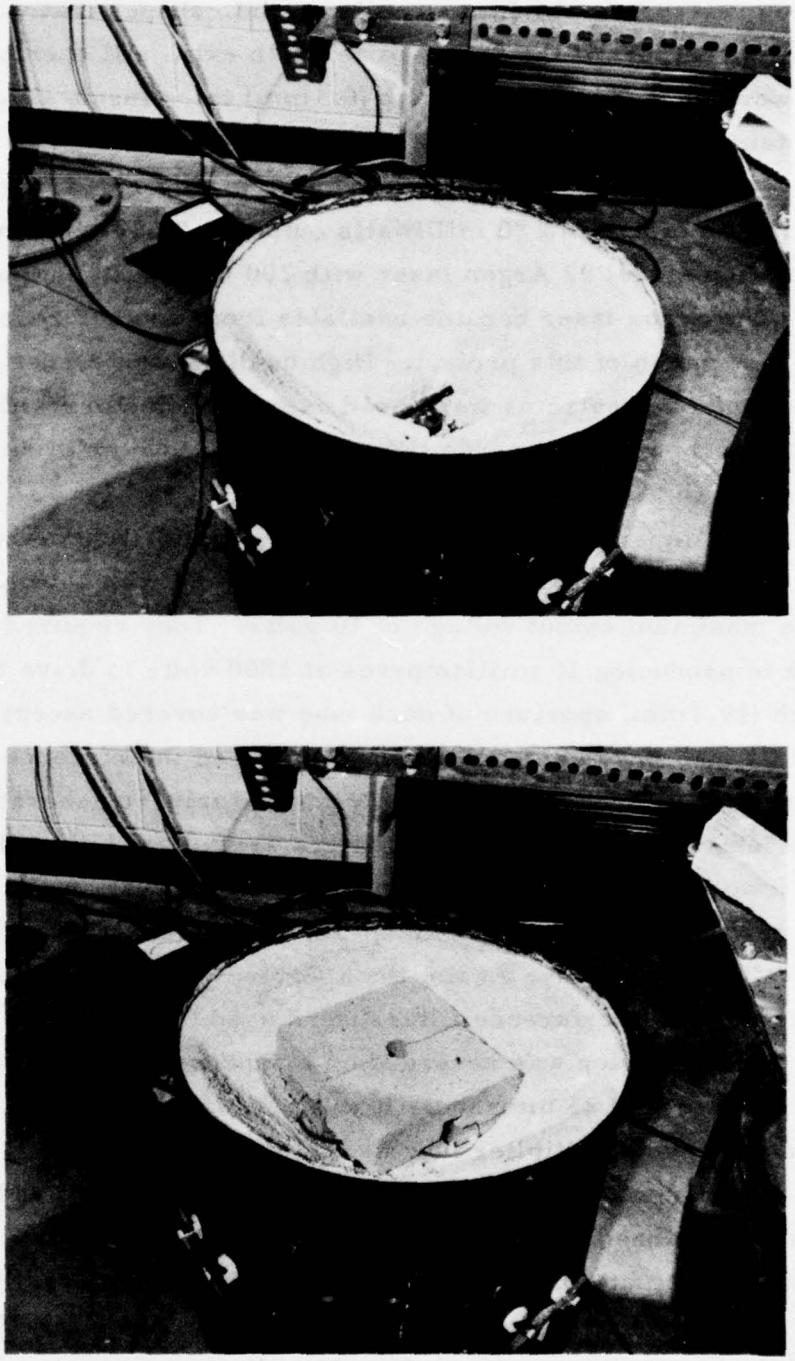


Figure 12. Furnace with and without firebrick cover.

thermocouple built into the side of the furnace cavity and by a small Pt/13% Rh thermocouple placed near the specimen. The furnace cover was constructed of firebrick which was easily cut into shapes that would fit the furnace walls and permit the fringe patterns to exit. All openings for laser beams were covered with 0.25 inch (6.4 mm) thick fused-silica parts. A typical temperature-time plot is shown in Figure 13.

Two lasers were used in the course of this research--a Jodon Model HN-20 He-Ne laser with 20 milliwatts output at 0.633 microns and a Coherent Radiation Model 52 Argon laser with 200 milliwatts output at 0.514 microns. The argon laser became available from another research project for the last month of this project. High quality interference filters with a bandwidth of 12 Angstroms were used over the detectors with the He-Ne laser and cheaper interference filters with a bandwidth of 80 Angstroms were used with the argon laser.

Two Amperex type XP-1117 photomultiplier tubes were used for the bulk of the experiments. These were terminated with a 10 K-ohm resistor giving a maximum output voltage of 10 volts. They required a power supply capable of producing 10 milliamperes at 1800 volts to drive both tubes. The 3/4 inch (19.2 mm) aperture of each tube was covered except for a slit of width 0.01 inch (0.25 mm) for the widely-spaced interference fringes or 0.003 inch (75 microns) for the finely-spaced fringes generated by the larger gage lengths. The interference filters were placed directly over the slits.

Two United Detector Type photodetectors with an aperture 0.040 inch (1 mm) in diameter and a built-in op-amp were also tried. They require only a ± 15 volt power supply. Again, the aperture was covered except for a narrow slit and the interference filters were used.

Fringe motion was recorded on a Sanborn strip-chart recorder running at a speed of 0.25 mm/sec. It was necessary to use an inverting circuit for each photomultiplier tube as the output is negative. Typically, the output of the tubes varied between approximately 0.5 volt and 1 volt as the pattern was scanned. The photodetector varied approximately 50 mv about a 1 volt dark output; this relatively large DC level was easily handled by the zero suppression of the Sanborn amplifiers. Since the ISG technique measures change of intensity rather than absolute intensity, setup and calibration for a test is rather easy.

Figure 14 is a photograph of the experimental setup at Michigan State University.

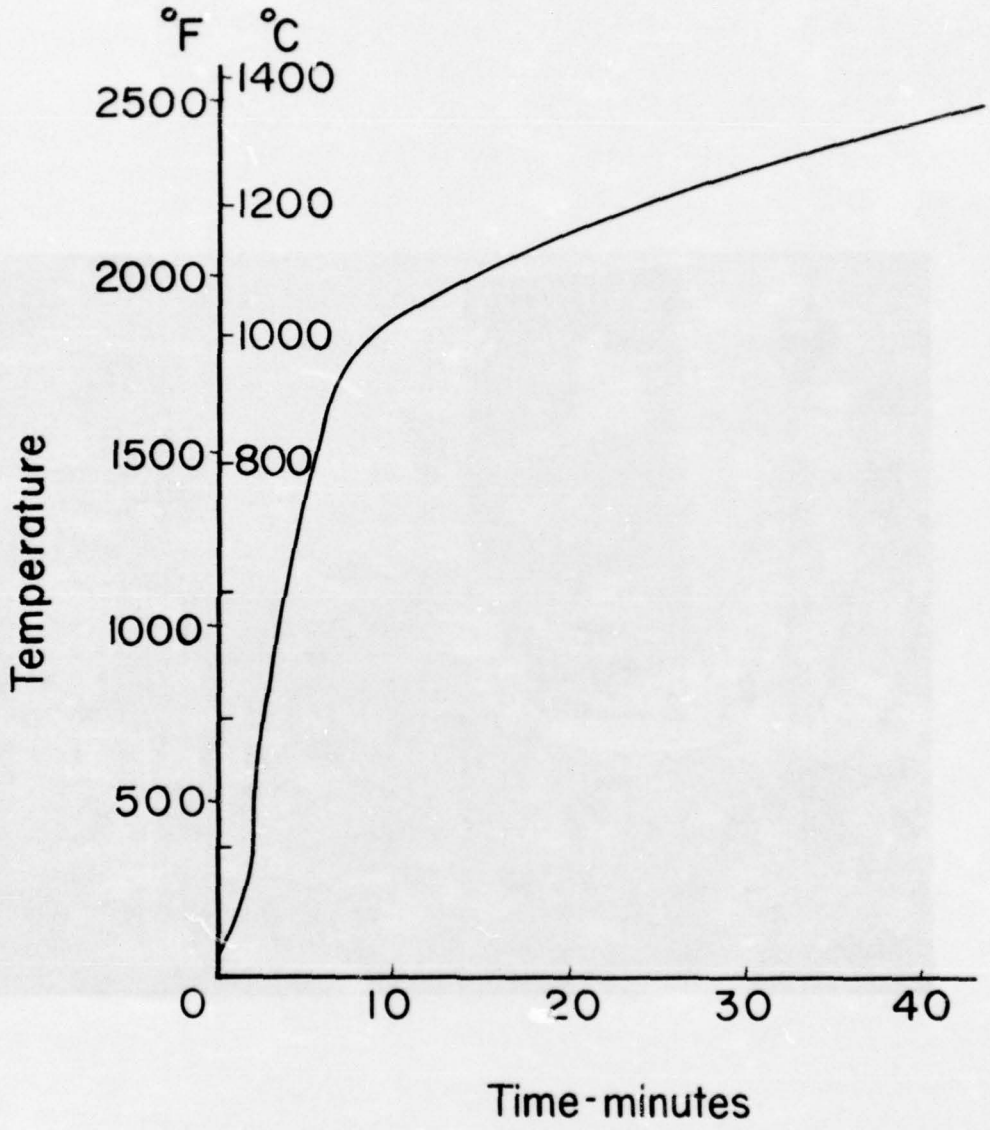


Figure 13. Typical temperature-time plot for the furnace of Figure 12.

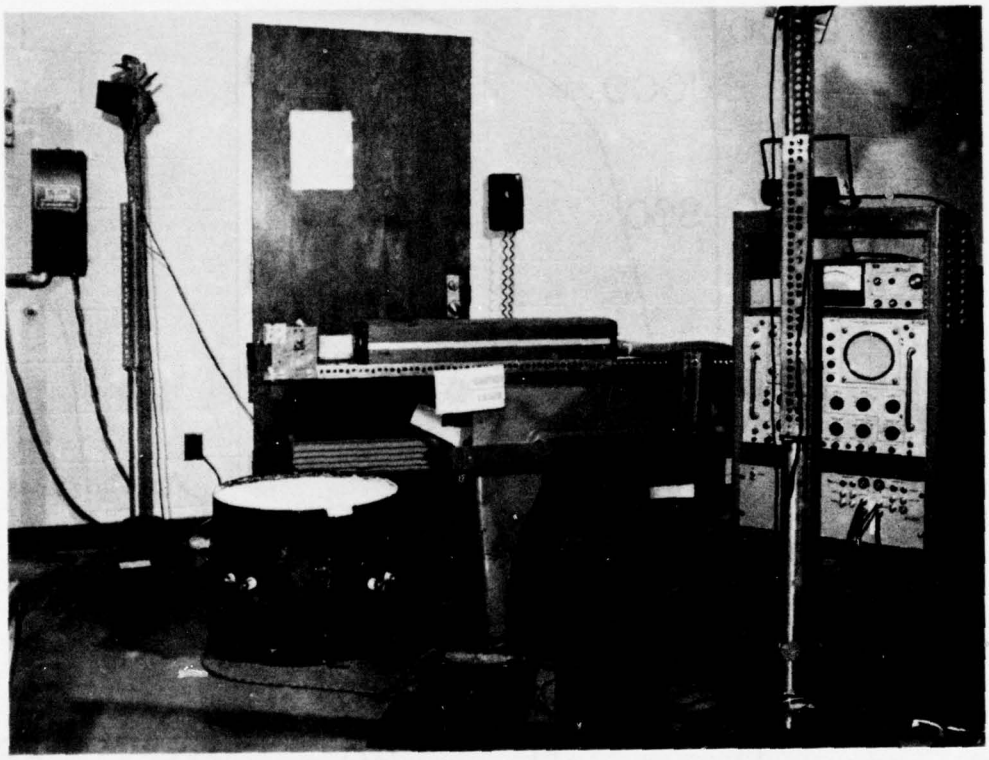


Figure 14. The experimental setup for thermal expansion measurements at MSU.

2. Experiments on Tungsten

One of the very first experiments run with the Michigan State University furnace was a thermal expansion test on tungsten with argon flowing over the specimen. The He-Ne laser was used along with the photodiodes. One photodiode malfunctioned during the test, but the output of the other is reproduced in Figure 15. It shows clearly the motion of one complete fringe shift and the decay of the signal as the specimen oxidizes. The rest of the signal (not included in Figure 15) shows no maxima or minima. Note the increased noise on the signal as the furnace begins to heat up; this is caused by increased motion of the dust particles in the air. This does demonstrate that a relatively inexpensive laser and photodiodes can be used; the voltage change of the signal is approximately 30 mv.

The temperature of the specimen was recorded originally by the large thermocouple in the furnace, but this was not satisfactory. A subsequent test in which the temperature was recorded by the small thermocouple placed next to the specimen as it was heated at the same power level was used to construct the strain-temperature curve of Figure 16.

In an attempt to reduce the oxidation of the tungsten, a specimen was enclosed in a fused quartz container which permitted the entrance and exit of the laser and fringe patterns. The container was then evacuated and purged with argon several times and finally sealed off. The fringe pattern traces (recorded by photomultiplier tubes) are given in Figure 17. Fringe shifts are easily identified, but again the motion of the air generates noise on the pattern. As the specimen oxidizes, the pattern intensity dies out.

Temperature was recorded by a thermocouple placed adjacent to the container. The strain-temperature plot of Figure 18 agrees roughly with the reference data (15); the difference is probably in the temperature measurement. Creating a relatively inert atmosphere around the tungsten extended the oxidation limit to approximately 1600°F, but a sophisticated vacuum system would be required for very high temperature measurements.

Because of the limited thermal expansion data obtained at Michigan State University, it was decided to subject one of the tungsten disk specimens to a "slow" heating rate at Southern Research Institute. The specimen was heated at a rate of approximately 17°F per second as measured by a thermocouple placed on the opposite side from the indentations and located approximately 0.2 inches (5 mm) from the center of the specimen.

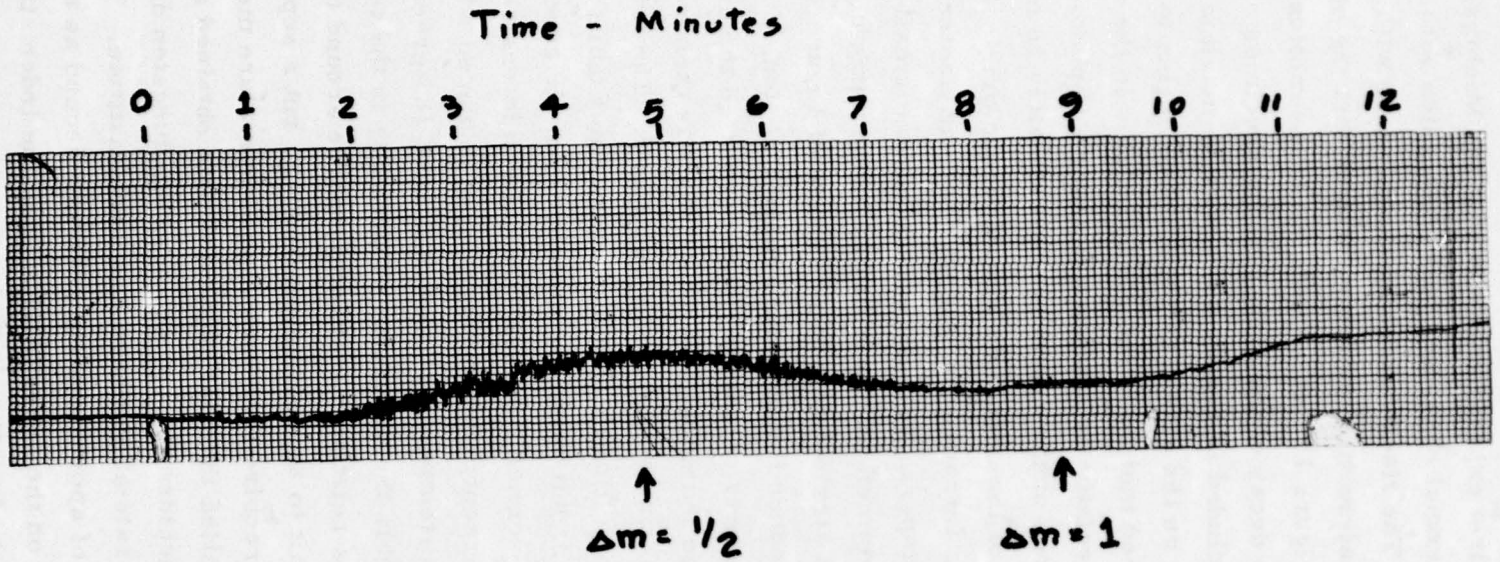


Figure 15. Signal trace from one photodiode for tungsten in air.

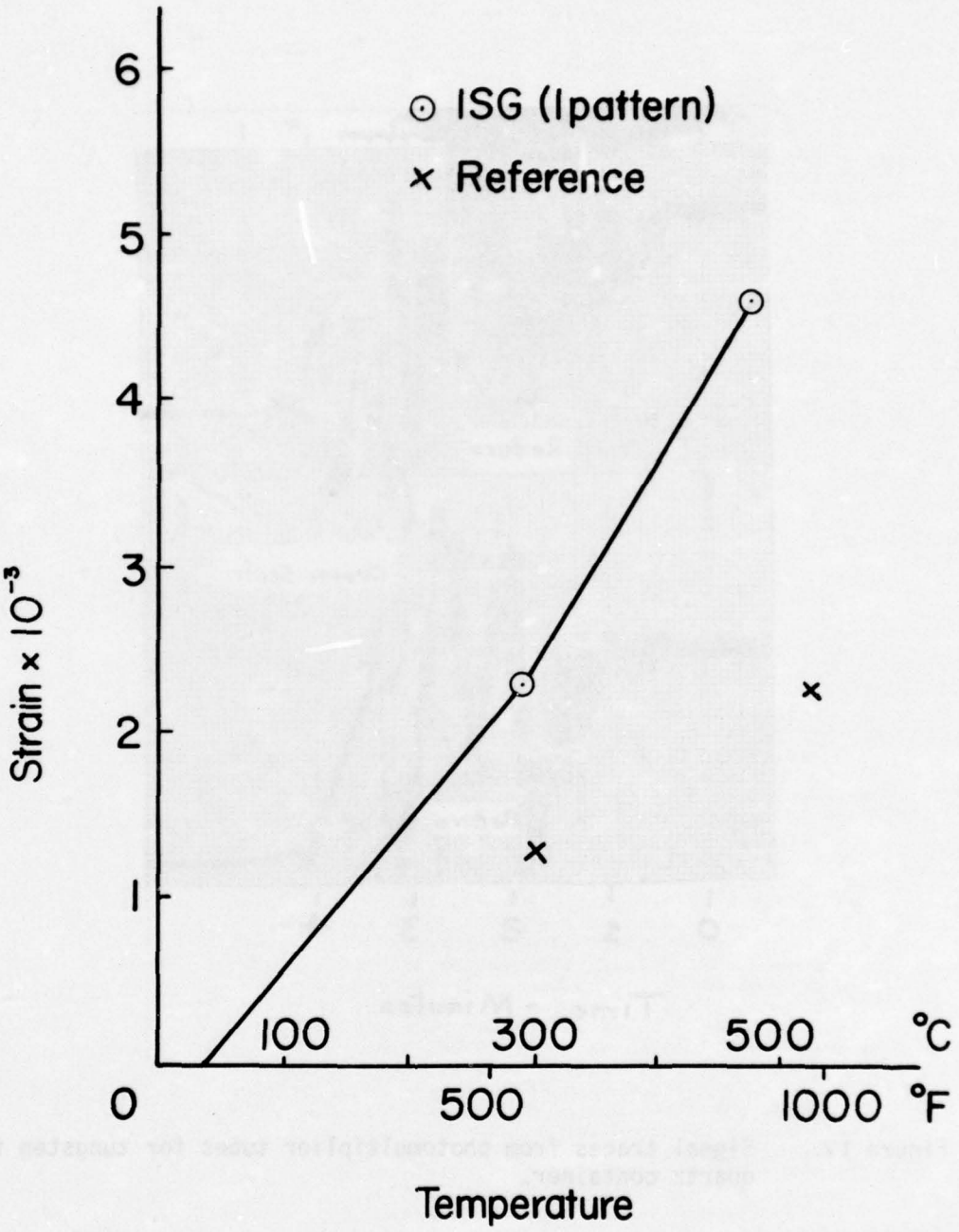


Figure 16. Thermal expansion of tungsten in air.

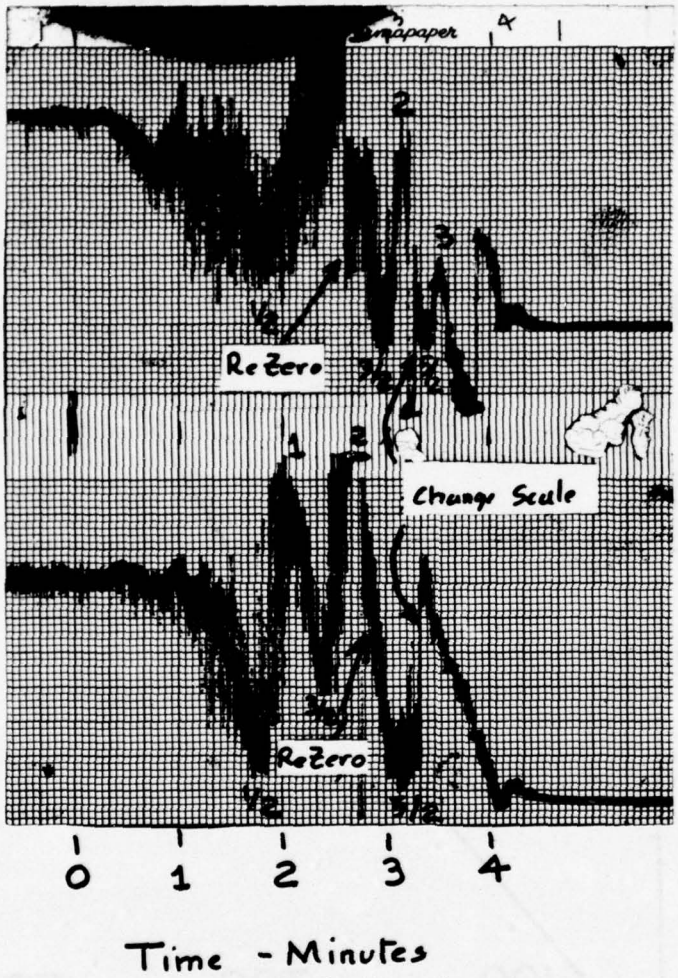


Figure 17. Signal traces from photomultiplier tubes for tungsten in quartz container.

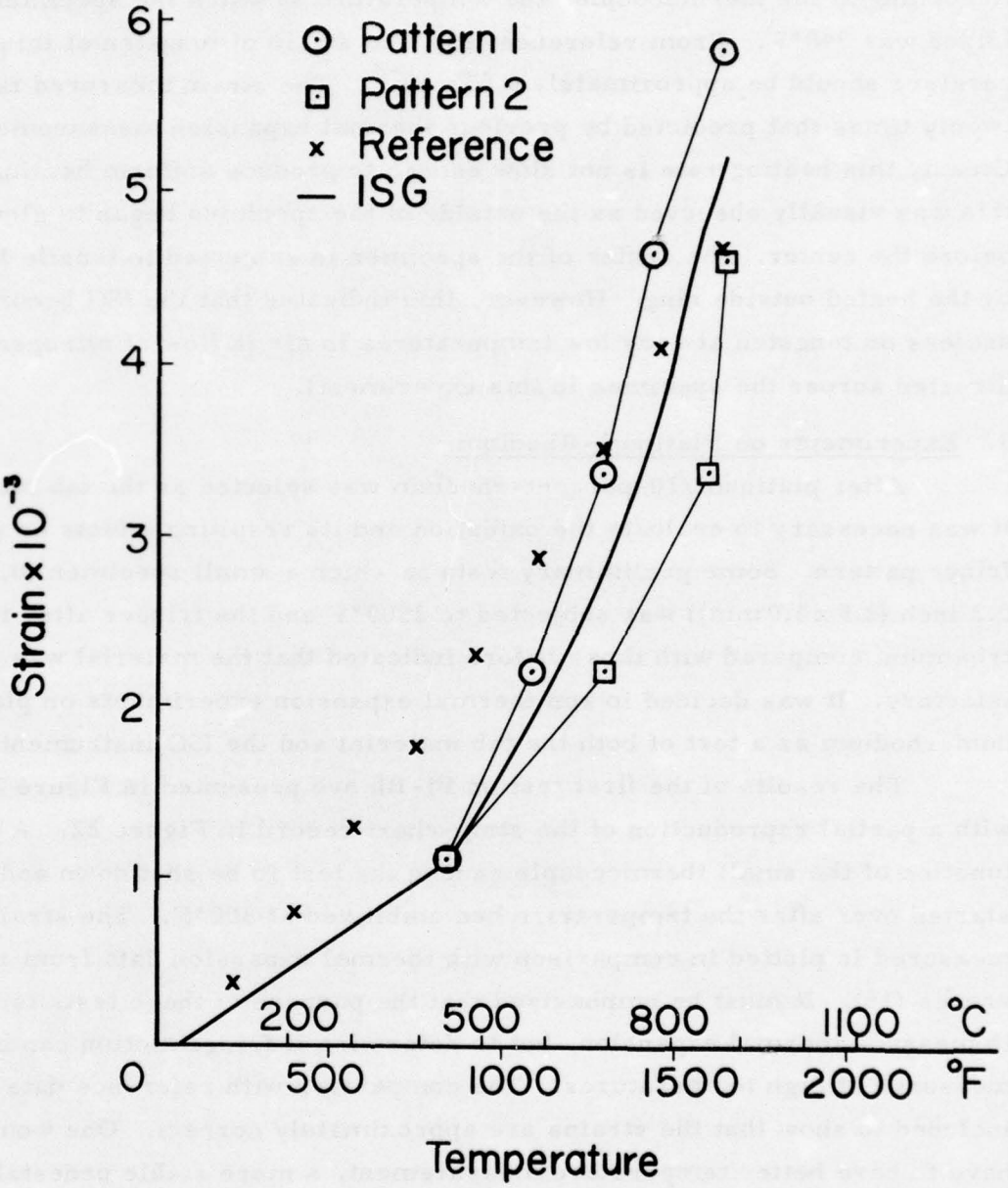


Figure 18. Thermal expansion of tungsten in quartz container.

A plot of the strain as a function of time is given in Figure 19 and a copy of the light beam oscillograph record of pattern 1 is shown in Figure 20. Figure 20 shows the decrease in fringe visibility as the specimen oxidizes. According to the thermocouple, the temperature at which the specimen oxidized was 340°F. From reference (15), the strain of tungsten at this temperature should be approximately 0.65×10^{-3}. The strain measured is over twenty times that predicted by previous thermal expansion measurements. Clearly this heating rate is not slow enough to produce uniform heating; this was visually observed as the outside of the specimen began to glow before the center. The center of the specimen is subjected to tensile loads by the heated outside ring. However, this indicates that the ISG becomes useless on tungsten at very low temperatures in air (a flow of nitrogen was directed across the specimen in this experiment).

3. Experiments on Platinum-Rhodium

After platinum/10-percent-rhodium was selected as the tab material, it was necessary to evaluate the oxidation and its resulting effects on the fringe pattern. Some preliminary tests in which a small specimen (0.1 x 0.2 inch (2.5 x 5.0 mm)) was subjected to 2500°F and the fringes after that treatment compared with those before indicated that the material was satisfactory. It was decided to run thermal expansion experiments on platinum/rhodium as a test of both the tab material and the ISG instrumentation.

The results of the first test on Pt-Rh are presented in Figure 21 with a partial reproduction of the strip-chart record in Figure 22. A malfunction of the small thermocouple caused the test to be shut down and started over after the temperature had stabilized at 300°F. The strain measured is plotted in comparison with thermal expansion data from reference (16). It must be emphasized that the purpose of these tests is not to measure thermal expansion, but to determine if fringe motion can be measured at high temperatures. The comparison with reference data is included to show that the strains are approximately correct. One would have to have better temperature measurement, a more stable pedestal for the specimen, corrections for small rigid body motions, etc., before accurate thermal strain data could be obtained. The temperature was recorded by the small thermocouple placed next to the specimen on the pedestal.

A second test was performed with improved experimental procedures, and its results are given in Figure 23. It shows results similar

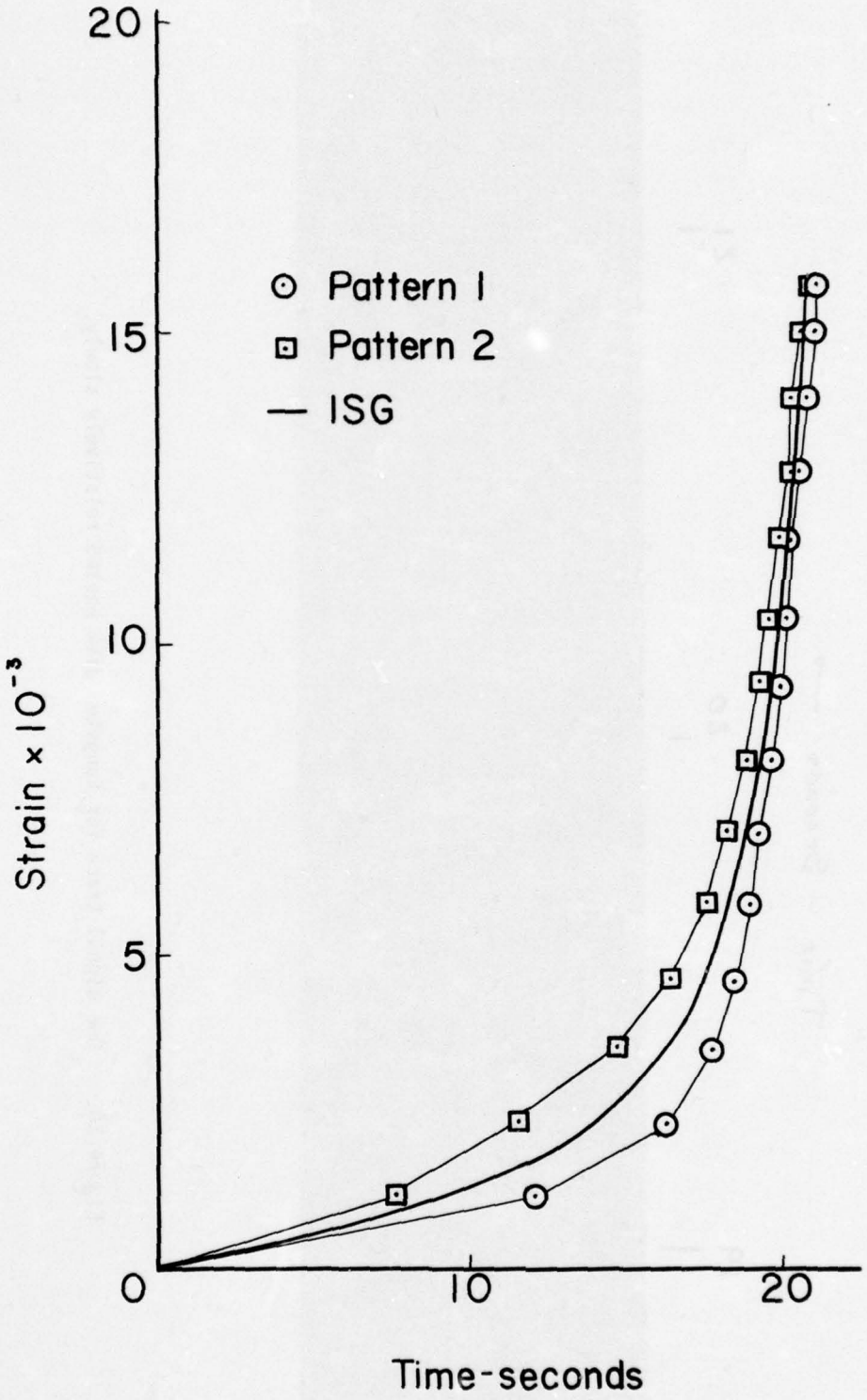


Figure 19. Strain versus time for tungsten disk heated relatively slowly.

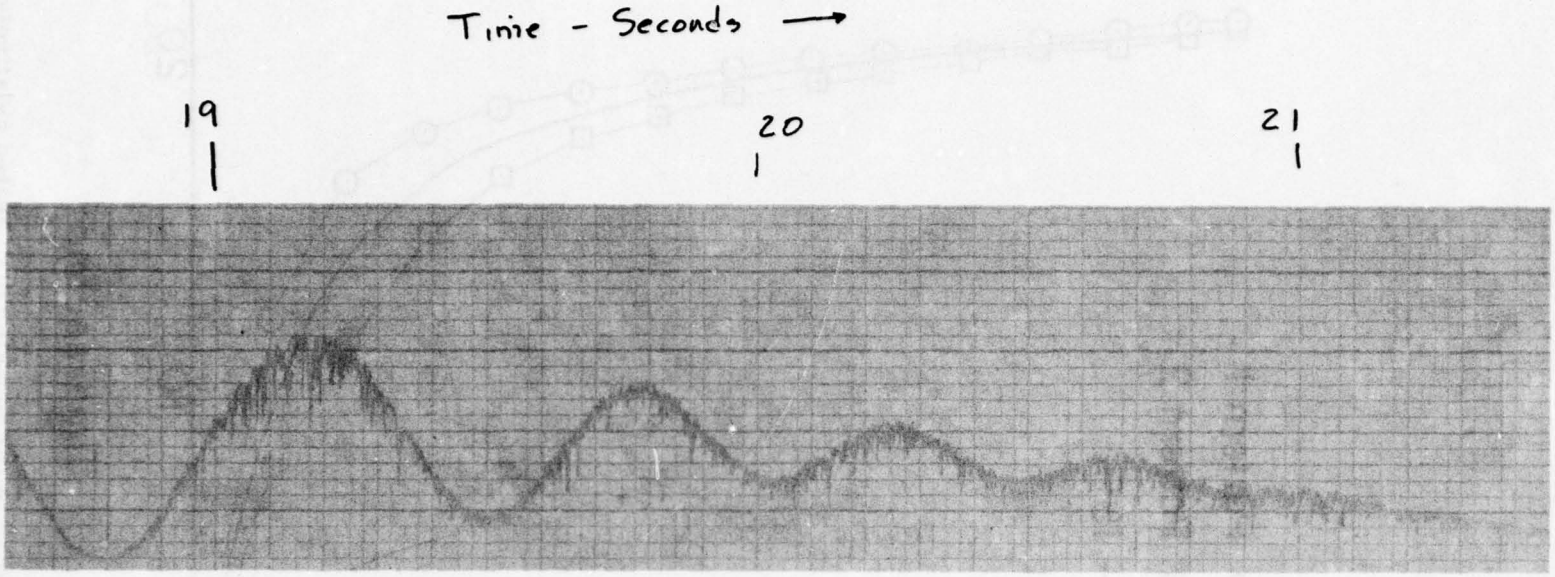


Figure 20. One signal trace for tungsten disk heated relatively slowly.

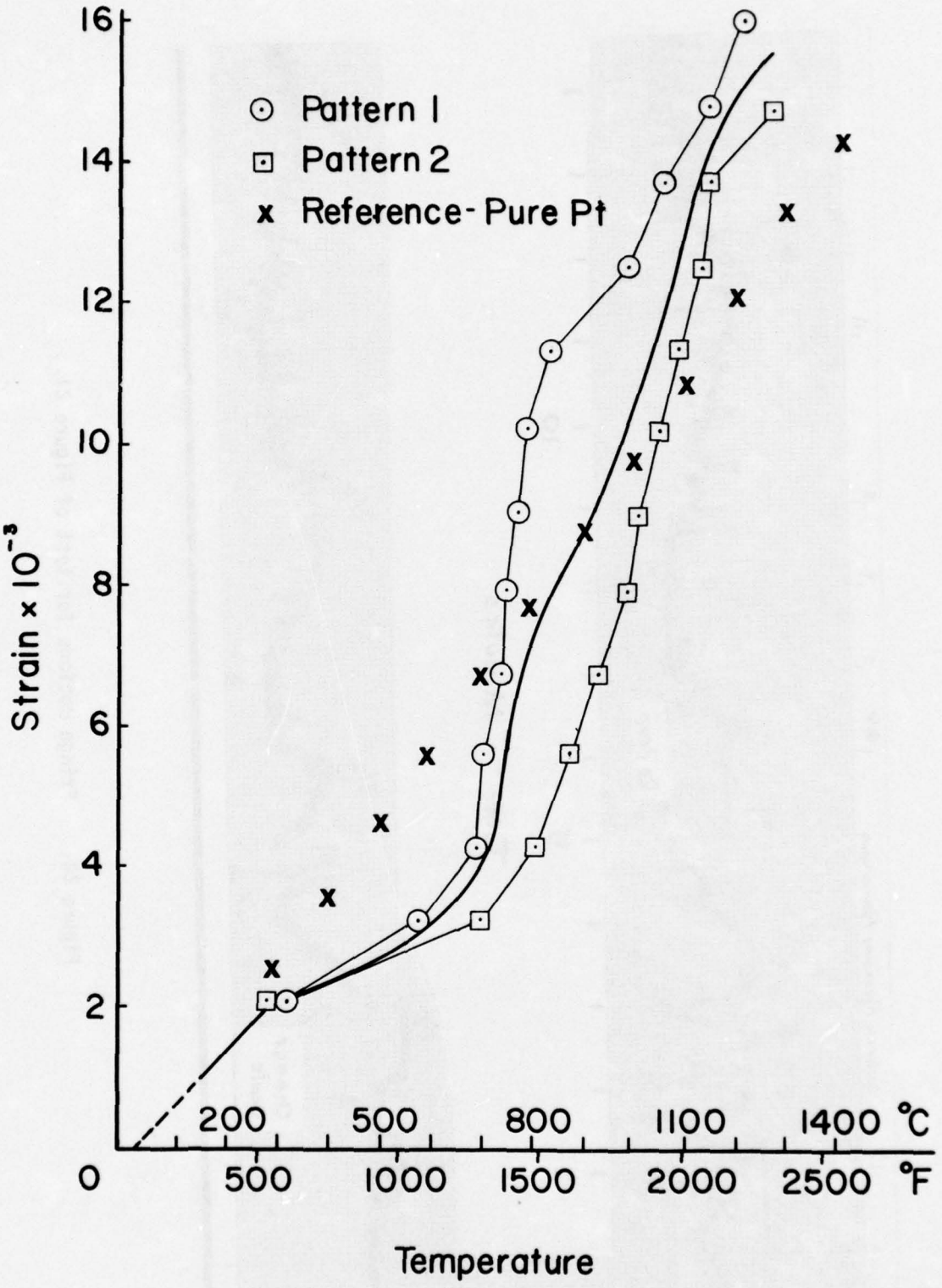


Figure 21. Thermal expansion of Pt/10% Rh in air.

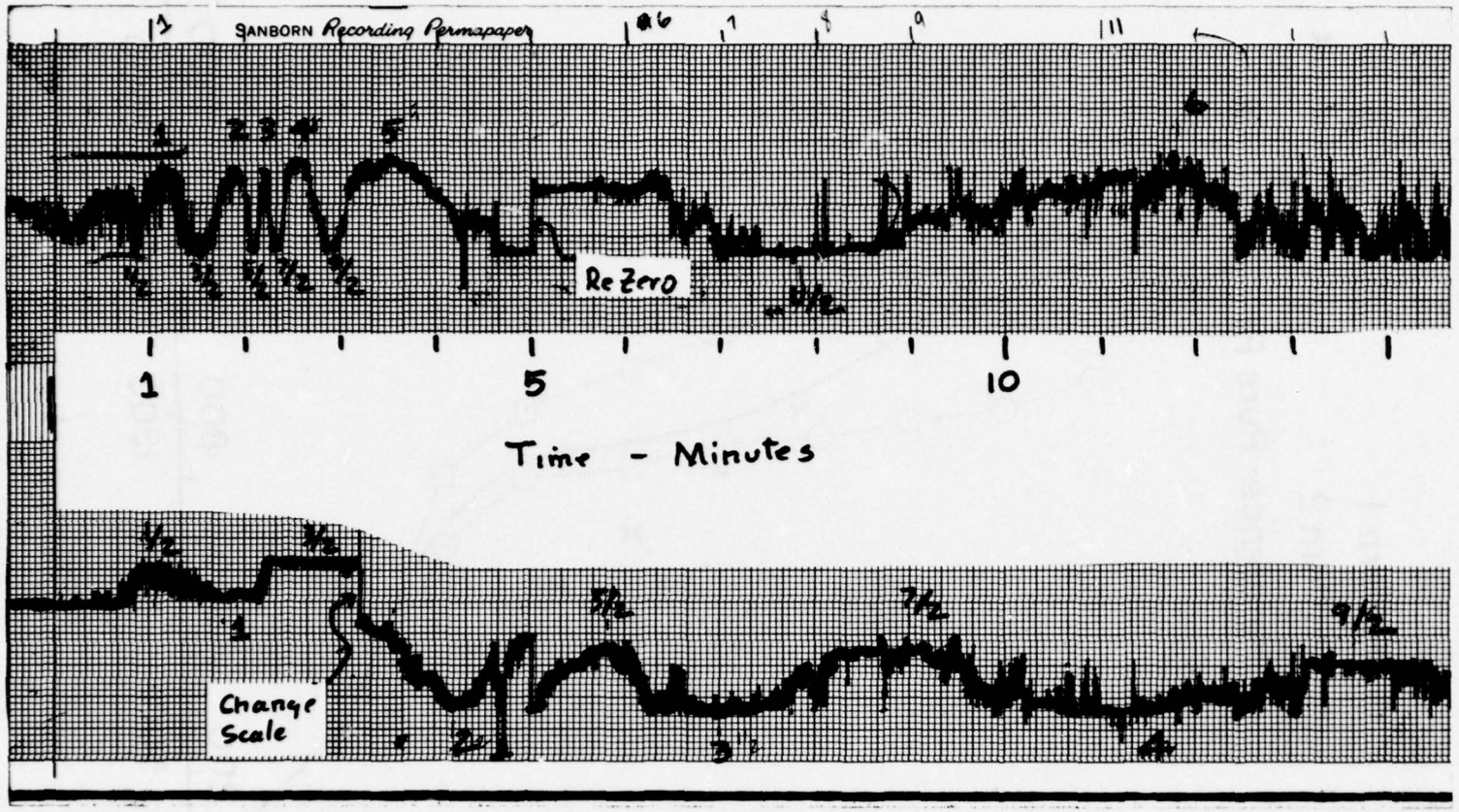


Figure 22. Fringe motions for test of Figure 21.

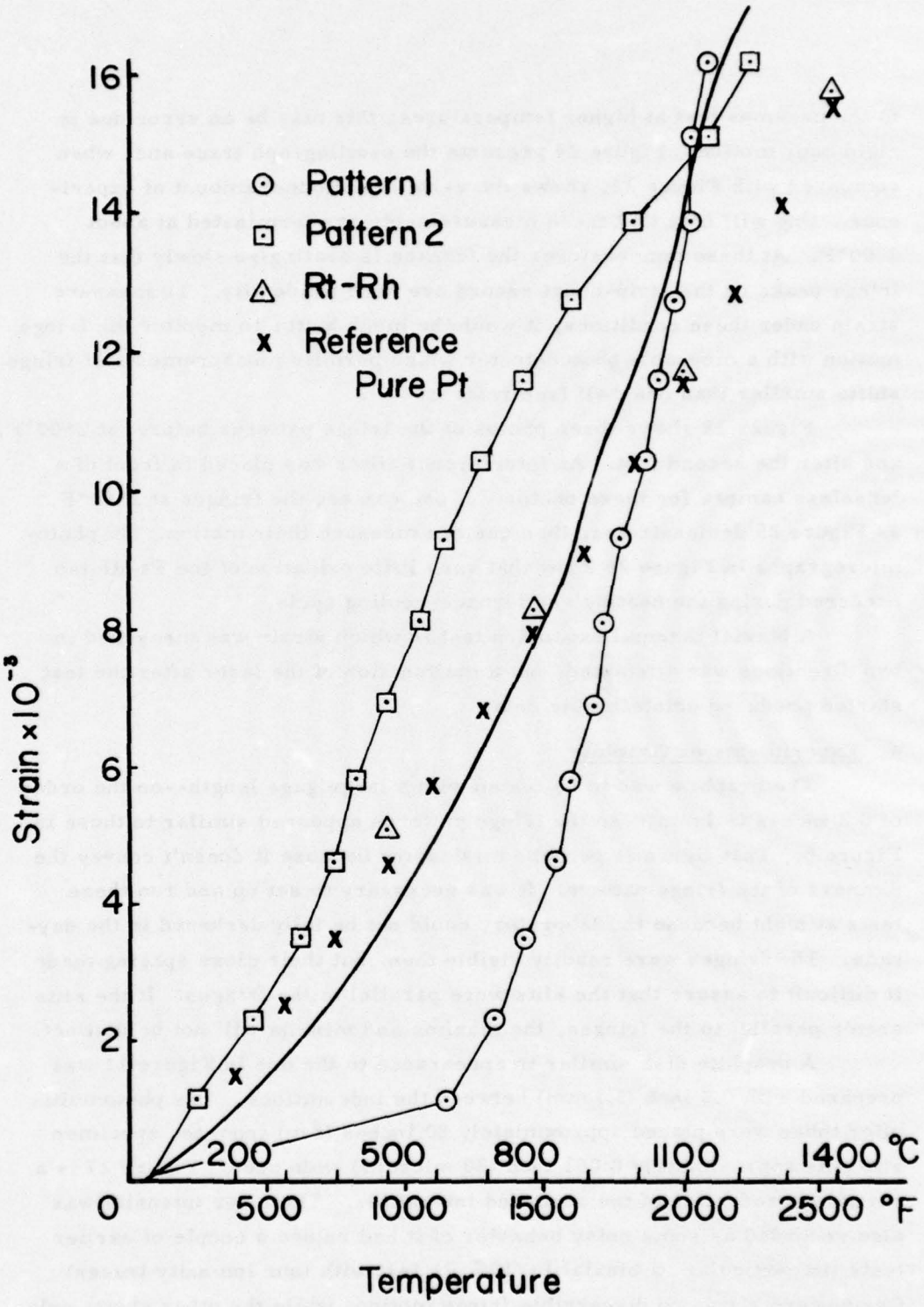


Figure 23. Thermal expansion of Pt/10% Rh in air.

to the previous test at higher temperatures; this may be an error due to rigid body motion. Figure 24 presents the oscillograph trace and, when compared with Figure 22, shows the value of a modest amount of experience. One will note that these measurements are terminated at about 2200°F. At these temperatures the furnace is heating so slowly that the fringe peaks on the strip-chart record are hard to identify. To measure strain under these conditions, it would be much better to monitor the fringe motion with a moveable photodetector which permits measurements of fringe shifts smaller than one-half (see reference 8).

Figure 25 shows three photos of the fringe patterns before, at 2500°F, and after the second test. An interference filter was placed in front of a lenseless camera for these photos. If one can see the fringes at 2500°F, as Figure 25 demonstrates, then one can measure their motion. The photomicrographs in Figure 26 show that very little oxidation of the Pt-Rh tab occurred during the heating and furnace-cooling cycle.

A biaxial thermal expansion test in which strain was measured in two directions was attempted, but a malfunction of the laser after the test started produced unintelligible data.

4. Experiments on Graphite

The graphite was to be tested with a large gage length--on the order of 0.2 inches (5.1 mm)--so the fringe patterns appeared similar to those in Figure 5. That figure is perhaps misleading because it doesn't convey the dimness of the fringe pattern. It was necessary to set up and run these tests at night because the laboratory could not be fully darkened in the daytime. The fringes were readily visible then, but their close spacing made it difficult to assure that the slits were parallel to the fringes. If the slits aren't parallel to the fringes, the maxima and minima will not be distinct.

A graphite disk similar in appearance to the one in Figure 11 was prepared with 0.2 inch (5.1 mm) between the indentations. The photomultiplier tubes were placed approximately 80 inches (2 m) from the specimen and slits approximately 0.003 inch (80 microns) wide used. Figure 27 is a partial reproduction of the recorded intensities. The laser intensity was also recorded as some noisy behavior of it had ruined a couple of earlier tests (in particular, a biaxial Pt/10% Rh test with four intensity traces). One pattern shows no discernible fringe motion, while the other shows only very small intensity variations. Clearly the signals of the latter pattern

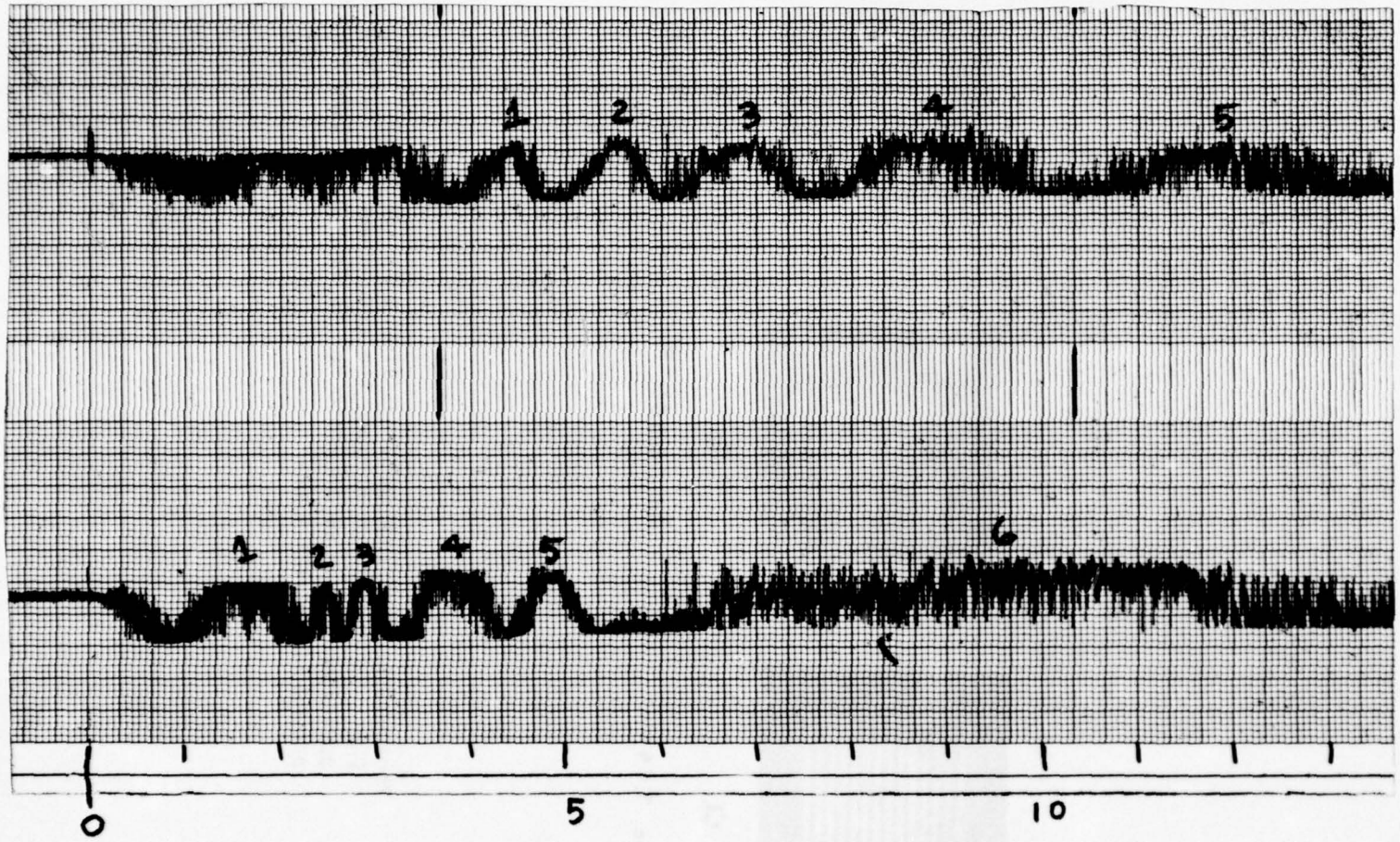


Figure 24. Fringe motions for test of Figure 23.

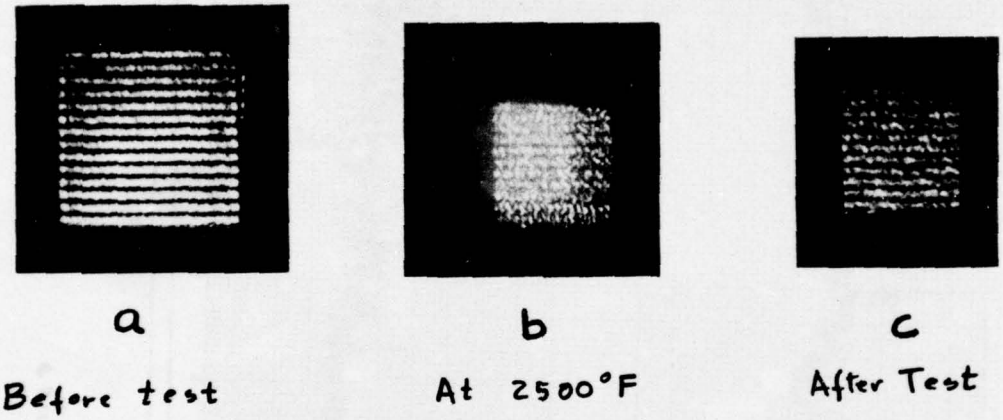
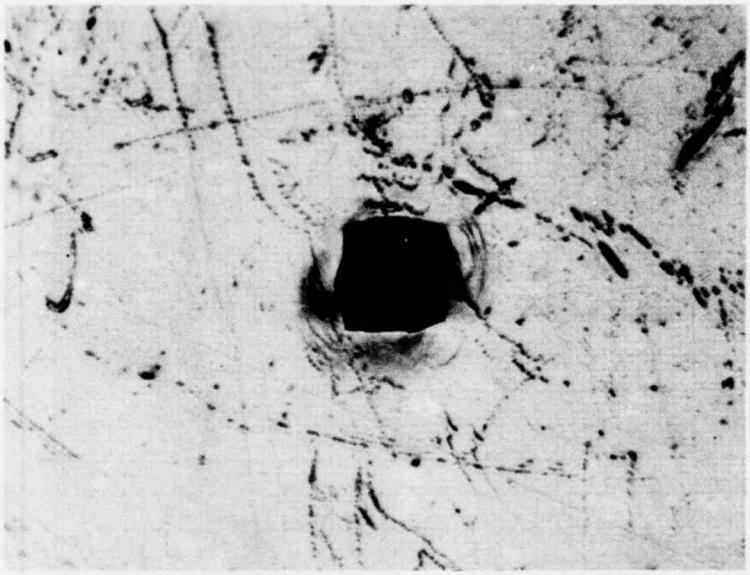
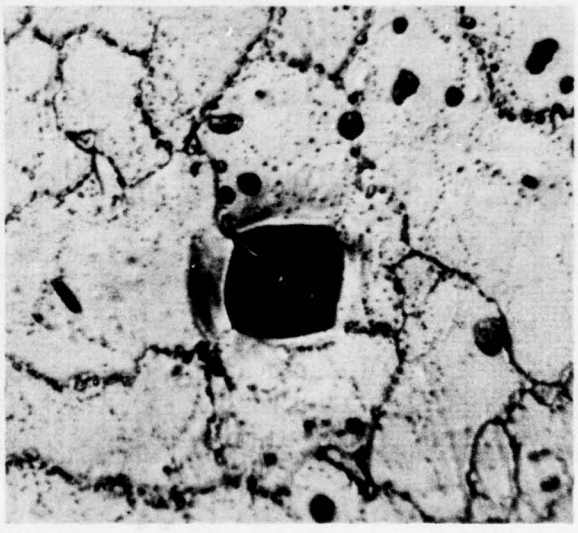


Figure 25. Photographs of fringe patterns from a Pt-10% Rh specimen. Photo "a" is larger because the camera was in a different position. The glow in photo "b" is from background radiation around the filter edge.



Before Test



After Test

Figure 26. Photomicrograph of an indentation on Pt-10% Rh before and after a test.

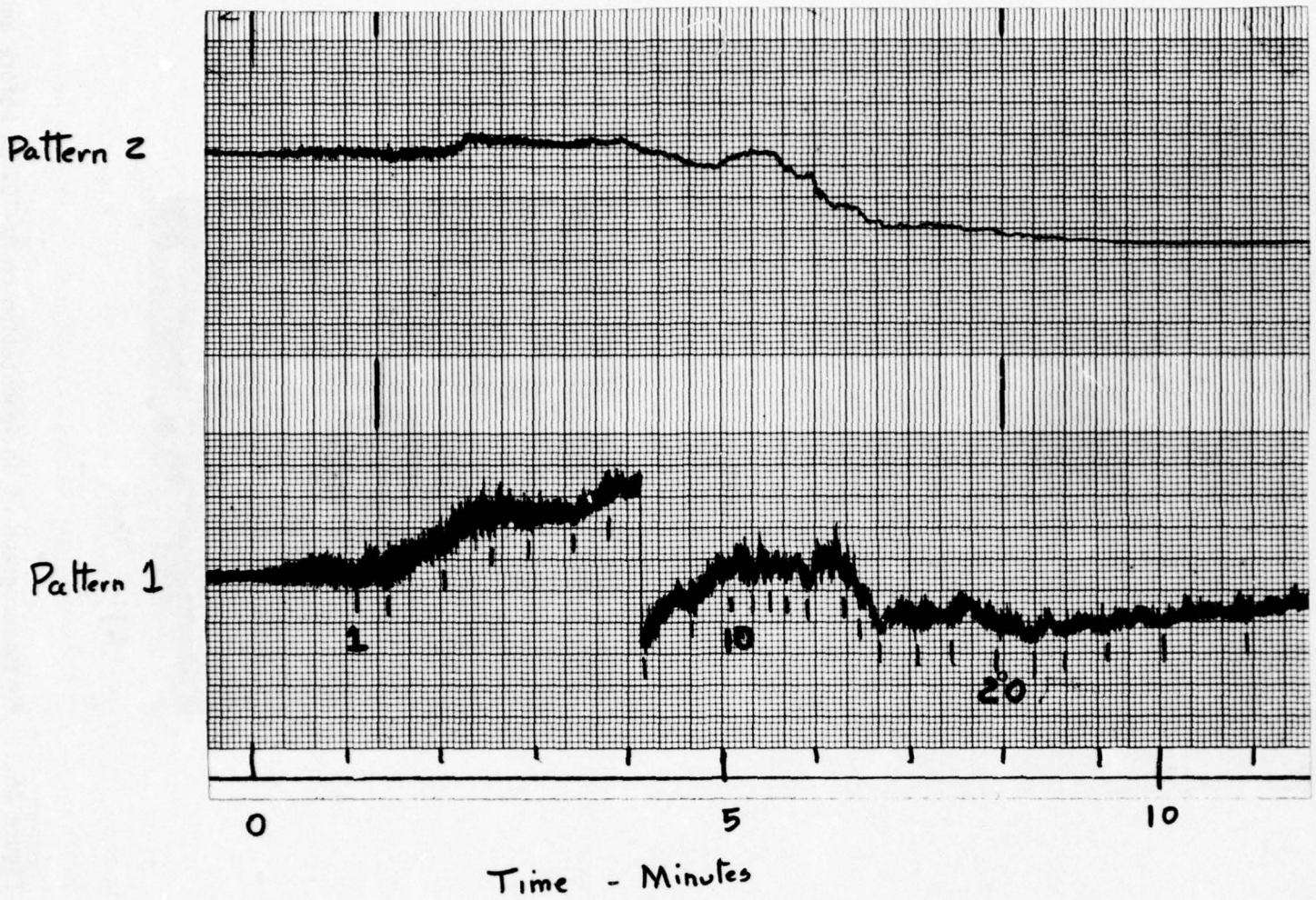


Figure 27. Signal traces from a thermal expansion test of graphite with a 0.2 inch (5.1 mm) gage length.

are not satisfactory, but if one assigns fringe shifts as indicated, the strain-temperature data of Figure 28 is obtained. This is roughly in agreement with thermal expansion data for ATJ-S graphite (17).

The specimen after the test is shown in Figure 29. It is very badly oxidized, and the tabs and adhesive have come loose from it. This failure of the adhesive is contradictory to the earlier test of the adhesive and is thought to be due to deterioration of the adhesive over time. After these tests, the pH of the adhesive was changed by adding orthophosphoric acid to it. In any event, the heavy oxidation of the graphite make it very difficult to measure thermal expansion at high temperatures in air.

A slow-heating thermal expansion test of graphite was attempted at Southern Research Institute in a manner similar to the tungsten experiment. The specimen had indentations located 0.076 inches (1.9mm) apart on the platinum-rhodium tabs. The adhesive had been subjected to only the first 250°F cure because it was thought desirable to compare the strength of the adhesive after the two curing procedures. The specimen was heated in the radio-frequency coil at a rate of approximately 10°F per second. Temperature was recorded by a chromel-constantan thermocouple mounted on the bottom of the disk.

The fringe motion traces are shown in Figure 30 and, although quite noisy, show definite fringe motion. The signal abruptly terminated at approximately seven seconds; the test was then shut down. Examination of the specimen showed that one tab had shifted. The heating had apparently released some of the water trapped in the adhesive and caused it to change shape. The specified cure to 1000°F requires heating up quite slowly--taking one hour to reach the cure temperature. This test shows that the 1000°F cure is essential. The strain computed from this limited amount of data is shown in Figure 31, but no conclusion as to the accuracy of the technique can be drawn from this; there simply is not enough data.

After the tab shifted, the specimen was no longer useful for ISG measurements. It was then heated to 2420°F at the same slow heating rate. When the graphite disks were prepared, a precoat of the adhesive had been applied to the graphite before attaching the tabs. This precoat had been scraped away on this specimen, leaving only the adhesive directly holding the platinum-rhodium tabs. At high temperatures--2000°F and above--the precoat adhesive that had penetrated the graphite rose to the specimen surface.

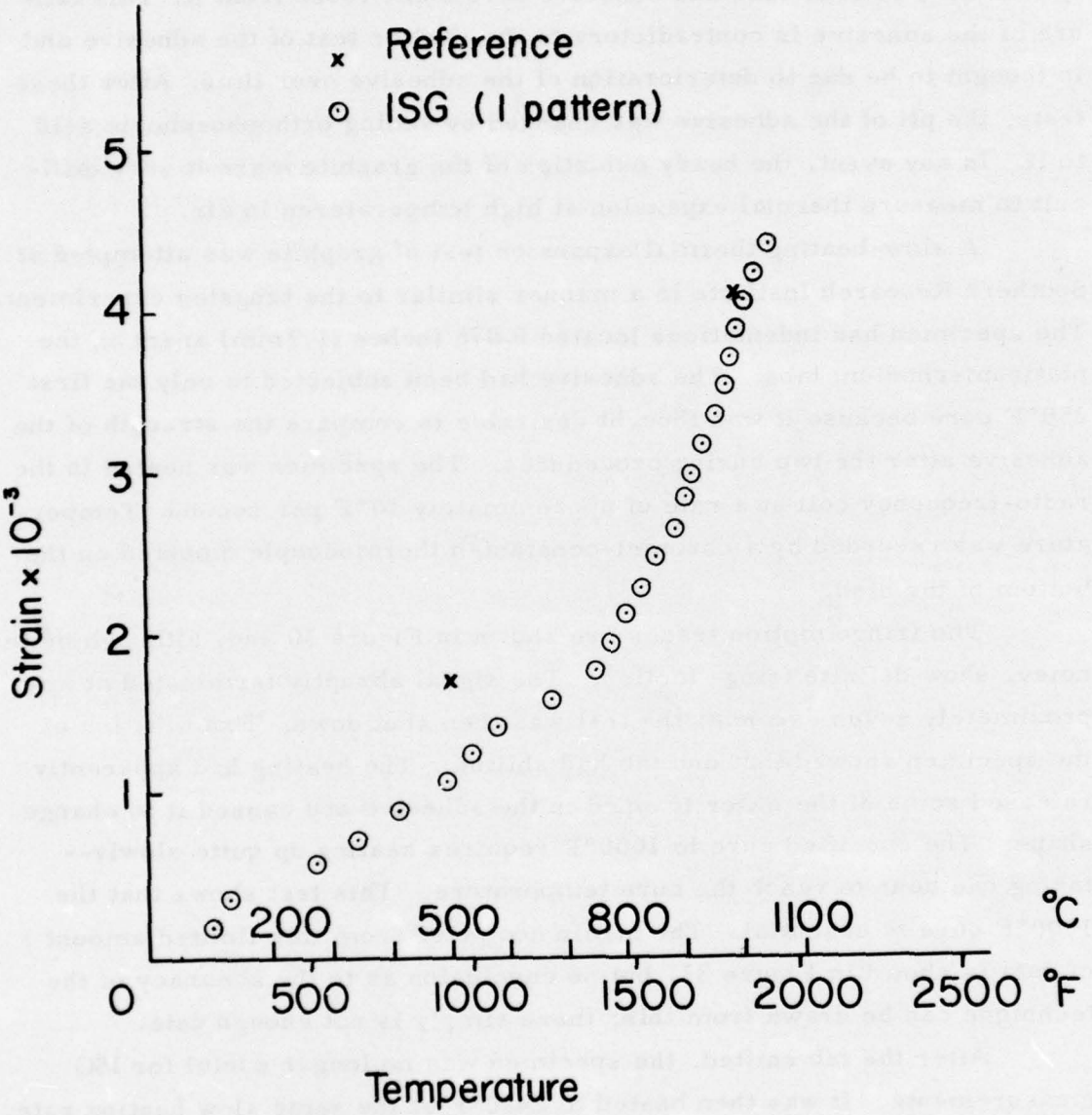


Figure 28. Thermal expansion of graphite based on the fringe motion of one pattern.

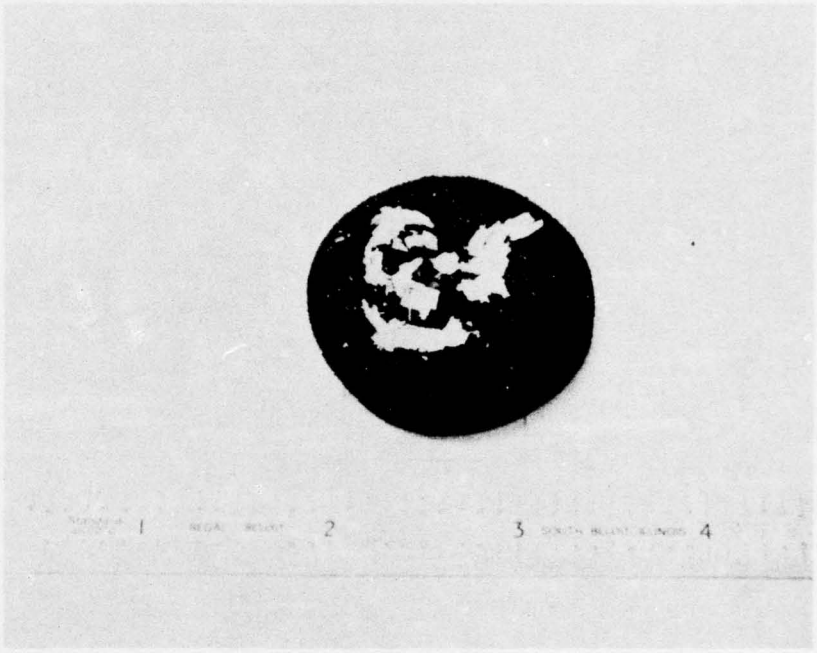


Figure 29. Graphite specimen after thermal expansion test.

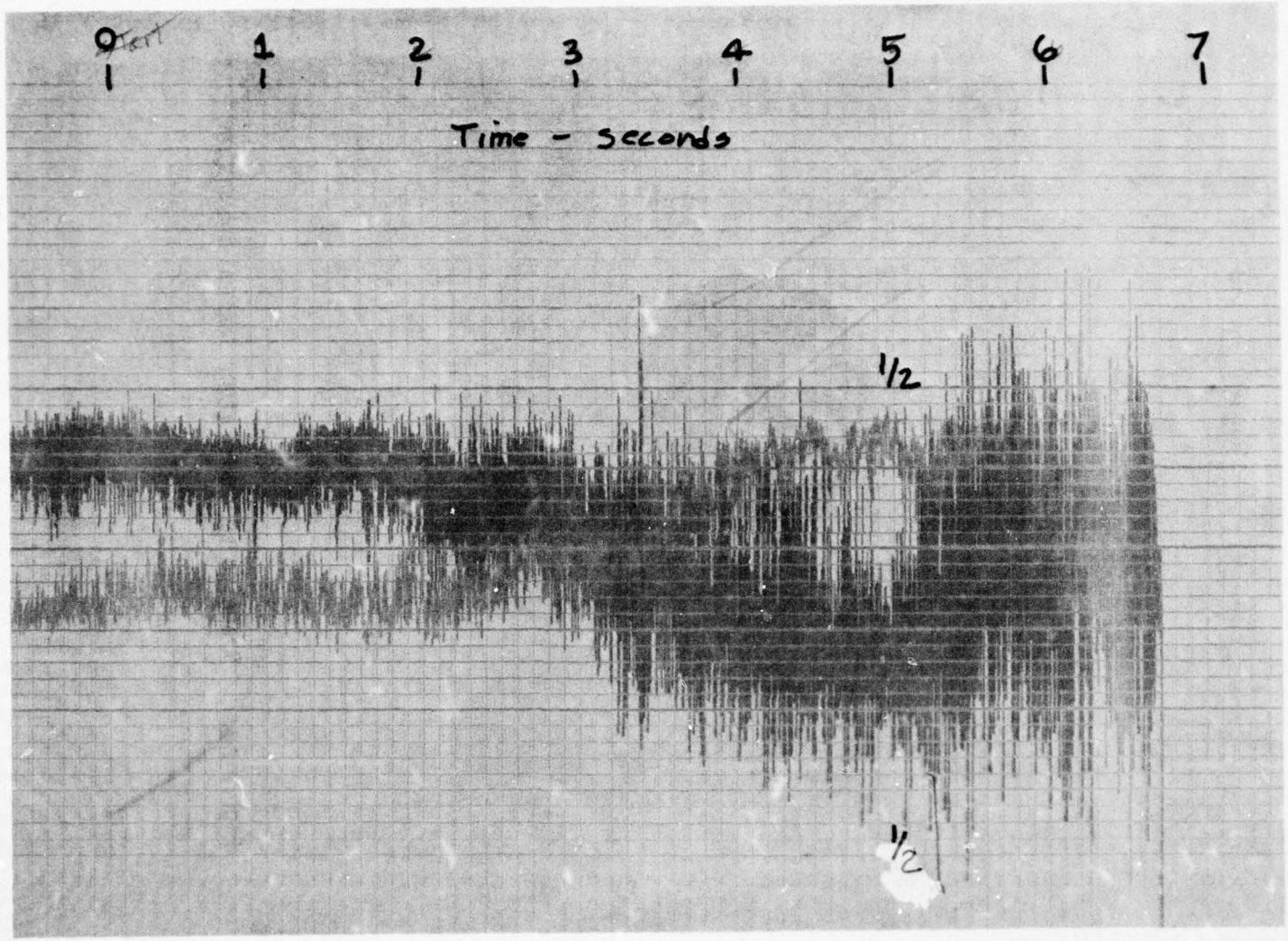


Figure 30. Fringe motion traces for thermal expansion of graphite disk.

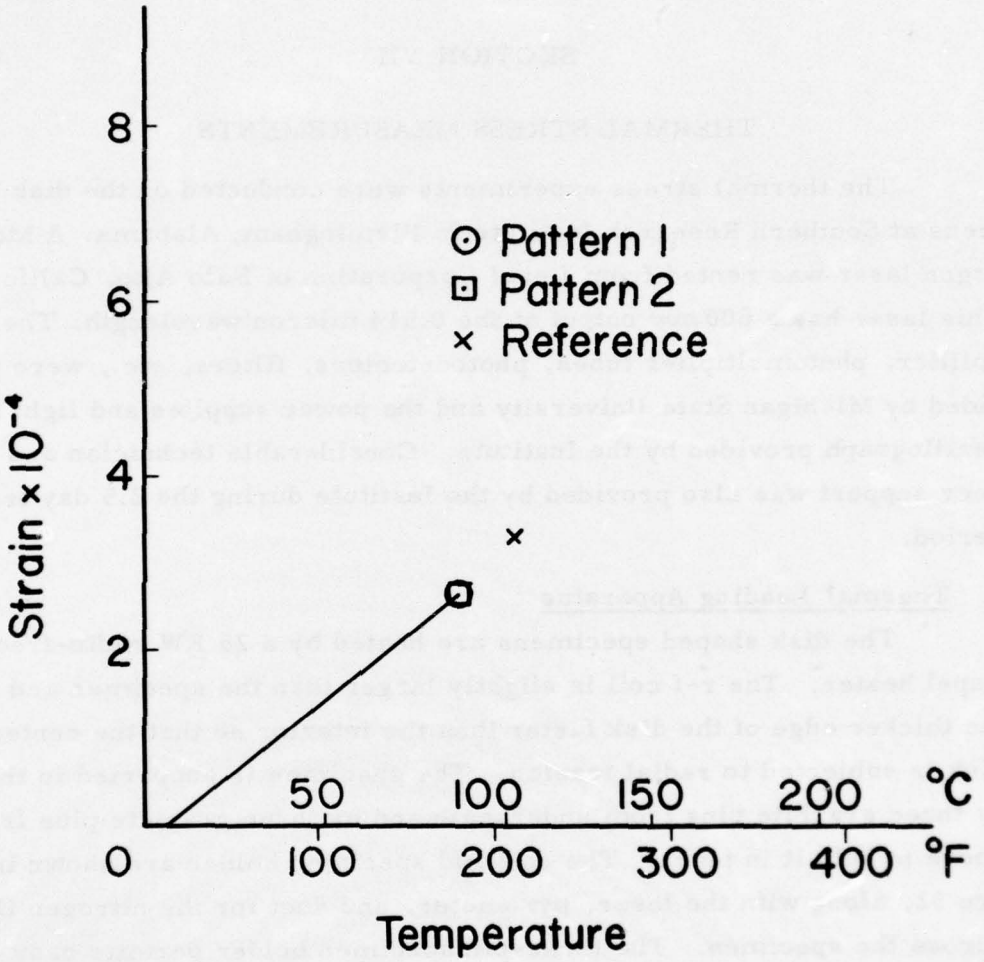


Figure 31. Thermal expansion of graphite disk.

After the specimen had cooled, the tabs were still attached, but were easily dislodged. This phenomenon of the adhesive being driven out of the graphite at high temperatures precludes the use of this adhesive to bond tabs at very high temperatures.

SECTION VII

THERMAL STRESS MEASUREMENTS

The thermal stress experiments were conducted on the disk specimens at Southern Research Institute in Birmingham, Alabama. A Model 85 argon laser was rented from Lexel Corporation of Palo Alto, California. This laser has a 600 mw output at the 0.514 micron wavelength. The beam splitter, photomultiplier tubes, photodetectors, filters, etc., were provided by Michigan State University and the power supplies and light beam oscillograph provided by the Institute. Considerable technician and engineer support was also provided by the Institute during the 2.5 day test period.

1. Thermal Loading Apparatus

The disk shaped specimens are heated by a 25 KW radio-frequency Lepel heater. The r-f coil is slightly larger than the specimen and heats the thicker edge of the disk faster than the interior so that the center of the disk is subjected to radial tension. The specimen is supported in the coil by three graphite pins from underneath and by three graphite pins from above to hold it in place. The coil and specimen holder are shown in Figure 32, along with the laser, pyrometer, and duct for the nitrogen flow across the specimen. The three-pin specimen holder permits easy entrance of the laser beam and exit of the fringe pattern.

A larger view of the experimental arrangement is given in Figure 33. The photomultiplier tubes are mounted approximately six feet (1.8 m) from the specimen. The overhead lights were turned off during setup and run of the experiment, but a completely dark room is not required. A typical heating cycle for a thermal stress test is shown in Figure 34; this temperature is recorded by an optical pyrometer focused on the inner rim of the disk specimen. The temperature actually goes to much higher values than the 2500°F--on the order of 3500°F before the graphite specimens break.

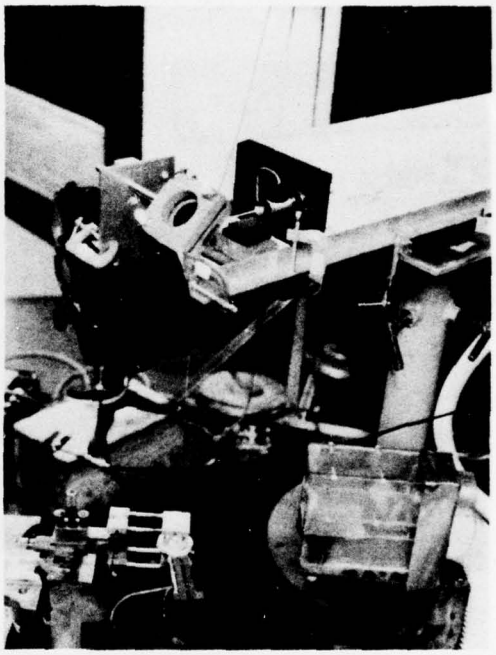


Figure 32. Photograph of the setup at Southern Research Institute.

Figure 33. Photograph of the setup at Southern Research Institute.

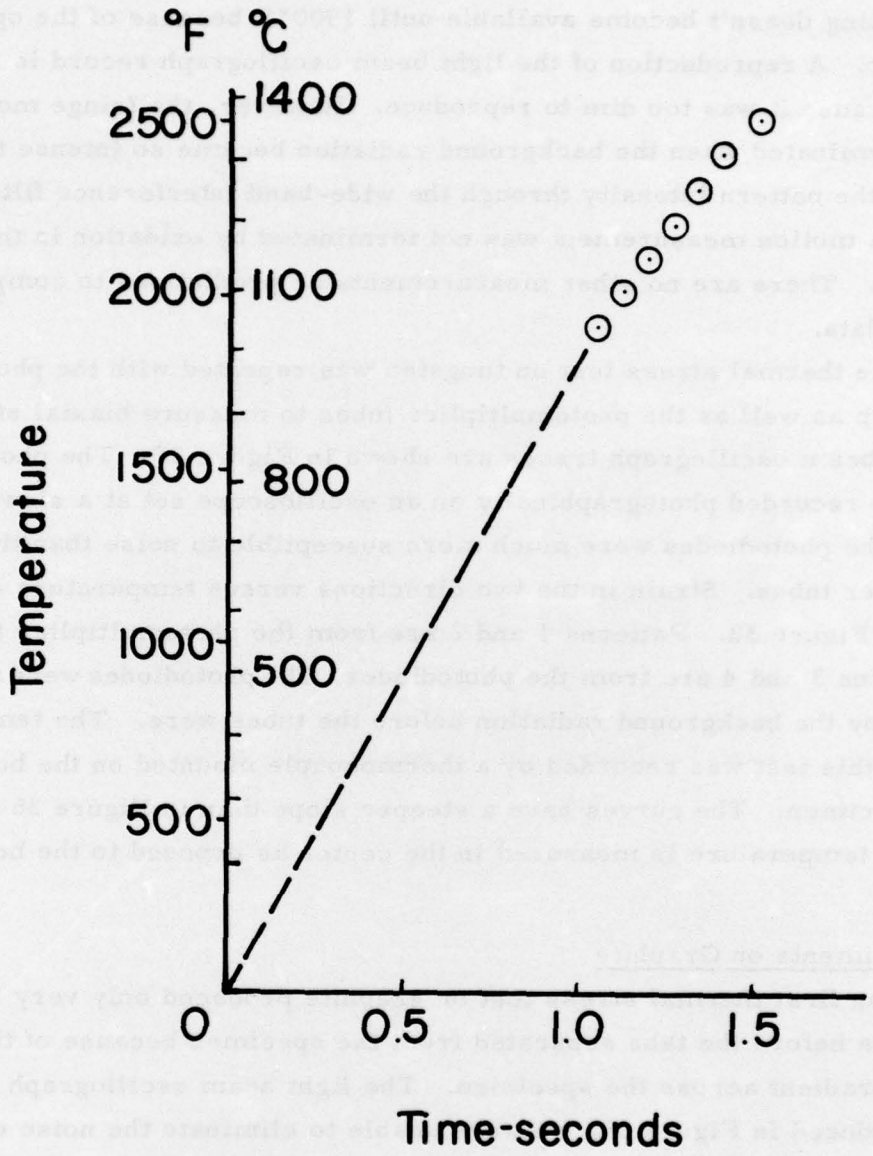


Figure 34. Temperature-time record for a thermal stress test. Recorded by an optical pyrometer.

2. Experiments on Tungsten

The strain versus time of the first thermal stress test on tungsten is presented in Figure 35. When combined with the temperature-time data, the strain temperature plot of Figure 36 is obtained. Note that the temperature reading doesn't become available until 1900°F because of the optical pyrometer. A reproduction of the light beam oscillograph record is not included because it was too dim to reproduce. However, the fringe motion record terminated when the background radiation became so intense that it swamped the pattern intensity through the wide-band interference filters. The fringe motion measurement was not terminated by oxidation in this rapid test. There are no other measurements or predictions to compare with this data.

The thermal stress test on tungsten was repeated with the photodiodes set up as well as the photomultiplier tubes to measure biaxial strain. The light beam oscillograph traces are shown in Figure 37. The photodiodes were recorded photographically on an oscilloscope set at a slow sweep speed. The photodiodes were much more susceptible to noise than the photomultiplier tubes. Strain in the two directions versus temperature are plotted in Figure 38. Patterns 1 and 2 are from the photomultiplier tubes, and patterns 3 and 4 are from the photodiodes. The photodiodes were driven off scale by the background radiation before the tubes were. The temperature for this test was recorded by a thermocouple mounted on the bottom of the specimen. The curves have a steeper slope than in Figure 36 because the temperature is measured in the center as opposed to the hotter edge.

3. Experiments on Graphite

The first thermal stress test on graphite produced only very limited strain data before the tabs separated from the specimen because of the rapid thermal gradient across the specimen. The light beam oscillograph records are reproduced in Figure 39; we were unable to eliminate the noise on one channel in the time available. One complete fringe shift is observed on each channel before the tabs pop loose at about 0.5 seconds. As the specimen began to glow, the signals came back on the chart. This test produced three results pertinent to the technique development. First, the tab attachment technique does not work. Second, the background radiation from the edge of the specimen is a problem (with these filters) even though the temperature

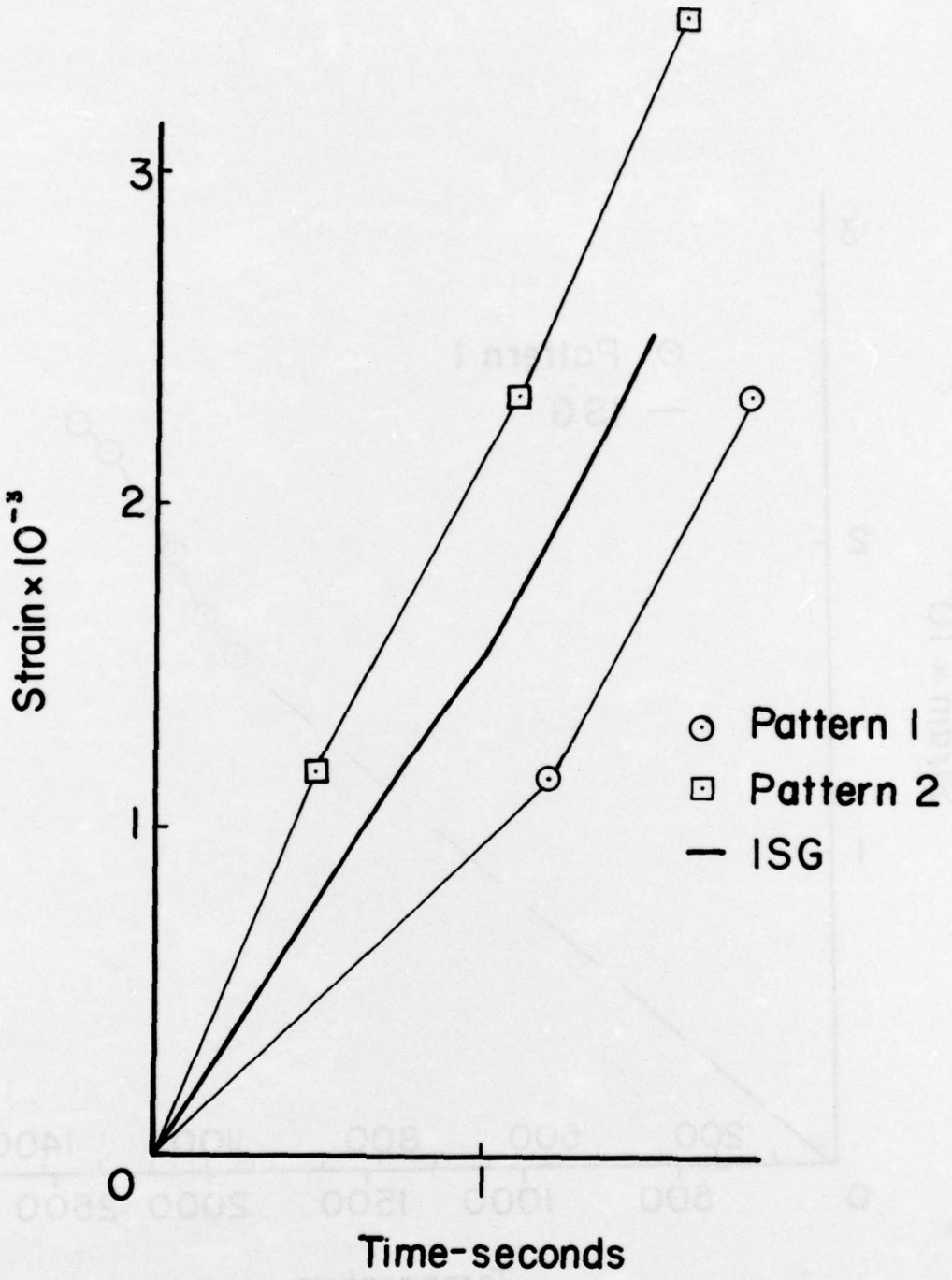


Figure 35. Strain versus time for a thermal stress test on tungsten.

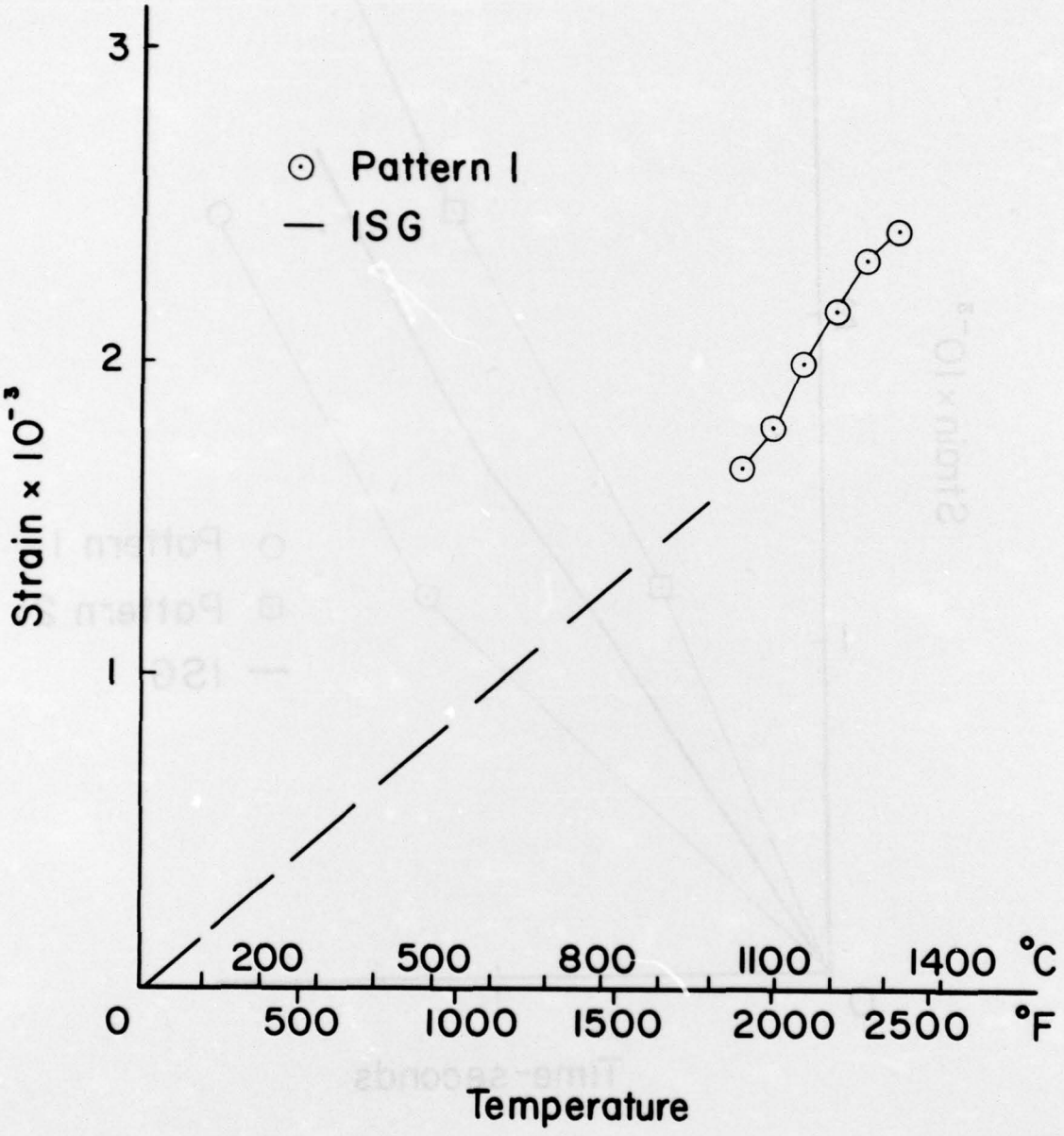


Figure 36. Strain versus temperature for a thermal stress test on tungsten.

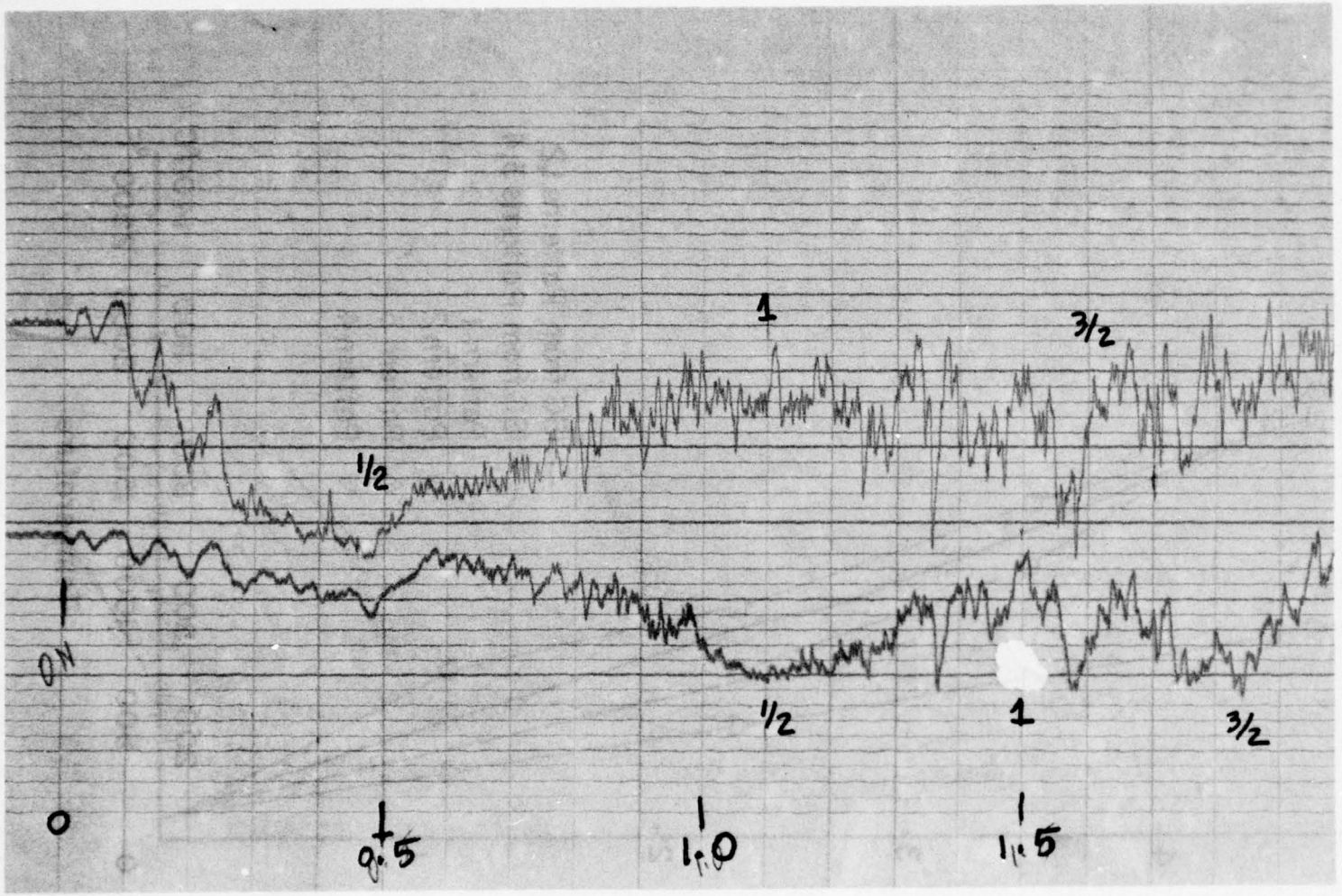


Figure 37. Fringe motion traces for a thermal stress test on tungsten.

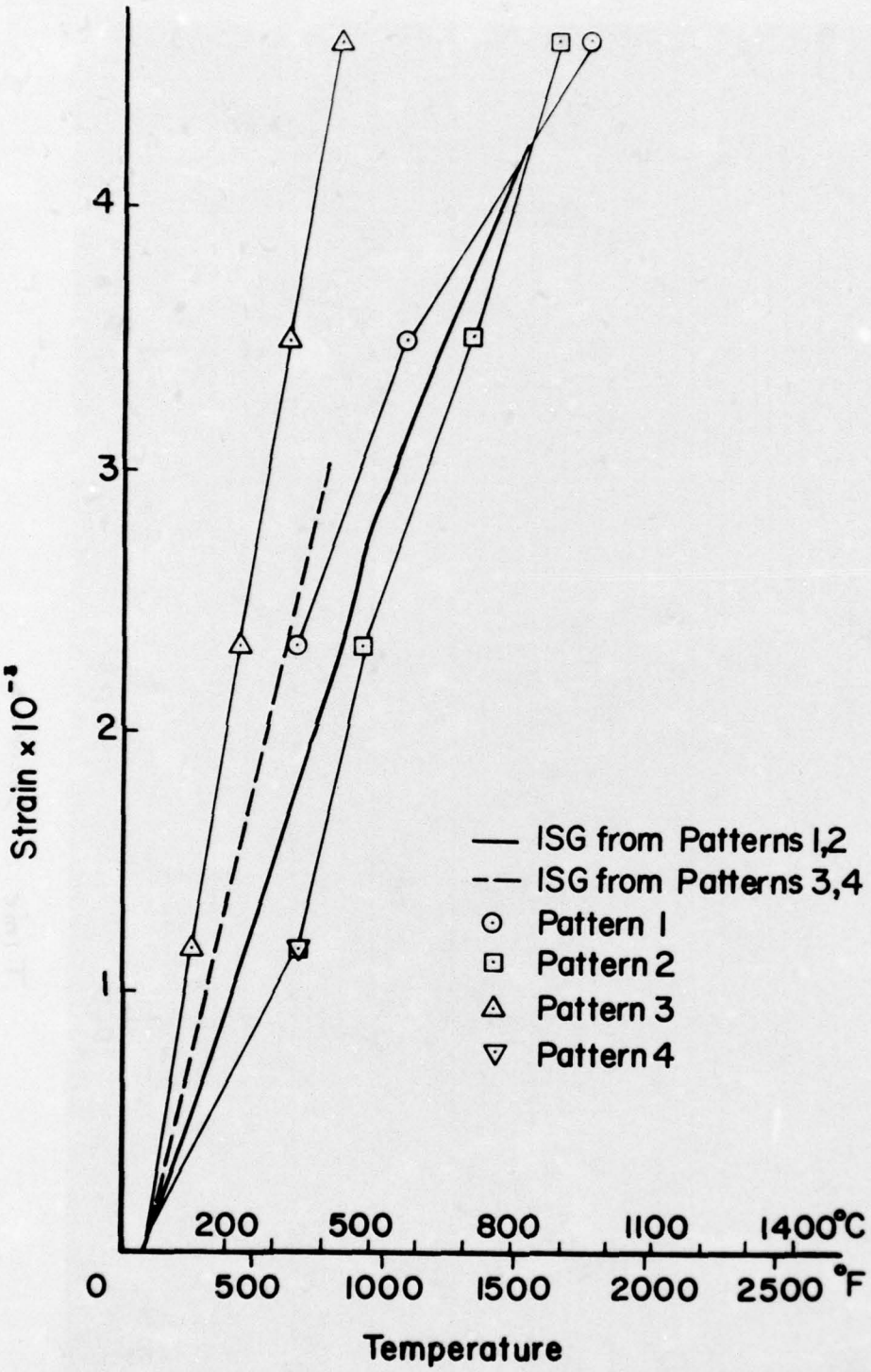


Figure 38. Biaxial strain for a thermal stress test on tungsten.

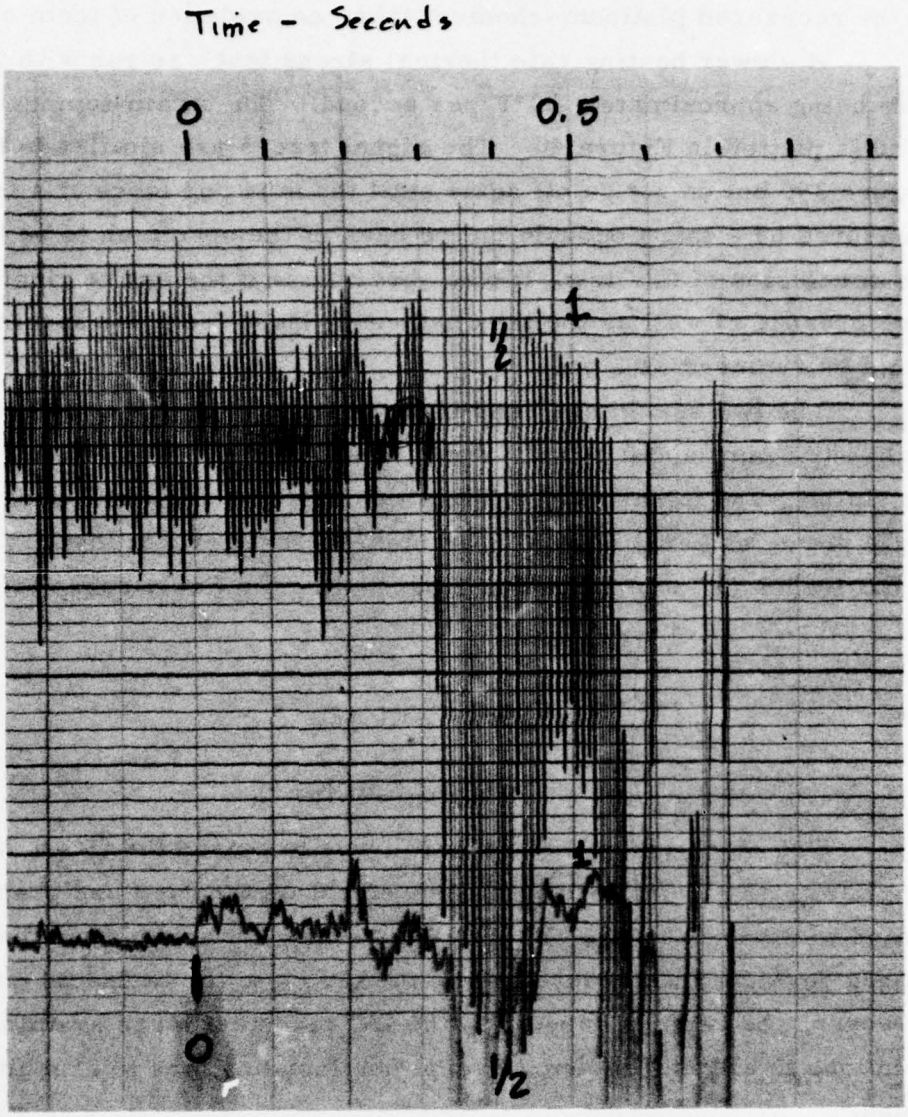


Figure 39. Fringe motion traces for a thermal stress test on graphite.

at the measurement point is less than 2500°F. Thirdly, from examination of the recovered platinum-rhodium tabs, no oxidation of them occurs.

A slower heating rate thermal stress test was run with the heating rate being approximately 60°F per second. The strain-temperature record is plotted in Figure 40. The signal traces are similar to those in Figure 39, but do not go off scale until the tabs pop loose at a temperature measured by a thermocouple on the back of the specimen to be 800°F. At the conclusion of this test, it was observed that the entire circle of adhesive precoat as well as the tabs had come loose from the specimen and could be removed as a unit.

The two specimens used in these tests had gage length of 0.098 inches (2.5 mm) and 0.100 (2.5 mm), respectively. The fringes were easily visible, and it was easy to align the slits in a darkened room with the more powerful Lexel laser. Specimens had been prepared with gage lengths of 0.2 inches (5.1 mm), but the fringes were too finely spaced to use.

SECTION VIII

CONCLUSIONS

The objective of this research was to extend the ISG to high temperature (2500°F) biaxial strain measurement on tungsten and graphite subjected to thermal shock. That objective was not achieved in the sense that a fully developed procedure can be passed on to the technical community. However, the various aspects of the ISG technique were examined, and solutions to all the problems except one (bonding tabs to graphite) were found.

The first and most positive conclusion is that Pt/10% Rh is a suitable material for tabs to which reflective indentations can be applied. One would have expected a material of this type with a low oxidation rate to be suitable. However, the myriad details of making the technique work--such as cutting into small tabs, polishing, handling, indentations--had to be proved in the laboratory. Once this is established, the technique has great potential for other kinds of high temperature strain measurement, provided a suitable method of tab attachment can be found.

The ceramic adhesive used in this research is not suitable in the

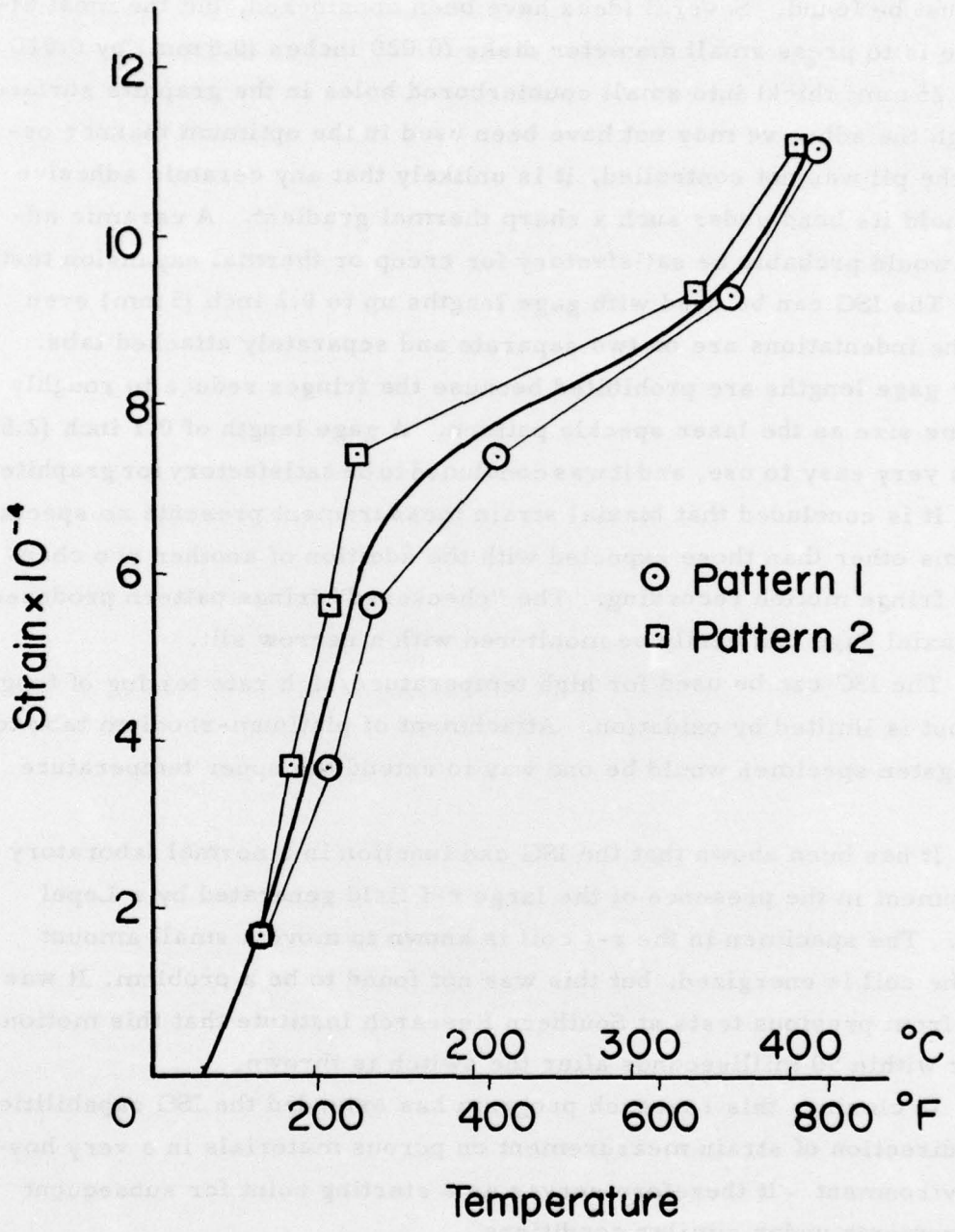


Figure 40. Strain versus temperature for a "slow" thermal stress test on graphite.

high thermal gradient experienced by a disk-shaped specimen of graphite. If the technique is to be used on graphite, another means of attaching the tabs must be found. Several ideas have been considered, but the most attractive is to press small diameter disks (0.020 inches (0.5 mm) by 0.010 inch (0.25 mm) thick) into small counterbored holes in the graphite surface. Although the adhesive may not have been used in the optimum manner because the pH was not controlled, it is unlikely that any ceramic adhesive would hold its bond under such a sharp thermal gradient. A ceramic adhesive would probably be satisfactory for creep or thermal expansion tests.

The ISG can be used with gage lengths up to 0.2 inch (5 mm) even when the indentations are on two separate and separately attached tabs. Larger gage lengths are prohibited because the fringes reduce to roughly the same size as the laser speckle pattern. A gage length of 0.1 inch (2.5 mm) is very easy to use, and it was concluded to be satisfactory for graphite.

It is concluded that biaxial strain measurement presents no special problems other than those expected with the addition of another two channels of fringe motion recording. The "checkered" fringe pattern produced by a biaxial gage can easily be monitored with a narrow slit.

The ISG can be used for high temperature/high rate testing of tungsten, but is limited by oxidation. Attachment of platinum-rhodium tabs to the tungsten specimen would be one way to extend the upper temperature limit.

It has been shown that the ISG can function in a normal laboratory environment in the presence of the large r-f field generated by a Lepel heater. The specimen in the r-f coil is known to move a small amount when the coil is energized, but this was not found to be a problem. It was known from previous tests at Southern Research Institute that this motion is over within 50 milliseconds after the switch is thrown.

In closing, this research program has extended the ISG capabilities in the direction of strain measurement on porous materials in a very hostile environment. It therefore serves as a starting point for subsequent measurements under similar conditions.

SECTION IX

RECOMMENDATIONS

The recommendations are of two kinds: those for using the ISG at high temperatures and high rate and those for future research.

1. ISG Equipment and Techniques

   a. An argon laser should be used. Its wavelength is short compared to the background radiation, and they are available at high power levels (200 mw or greater) which produce easily visible patterns.

   b. Narrow band-pass interference filters should be used. The less expensive interference filters used on this project are quite satisfactory to 2500°F. However, other portions of the specimen or furnace may be at higher temperatures than the gage region. This glow enters the photomultiplier tubes.

   c. Photomultiplier tubes are preferred. They have a large aperture and high sensitivity which means they can be covered with a long, thin slit to give optimum fringe definition.

   d. The gage length should be restricted to 0.1 inch (2.5 mm) or less.

   e. Recording fringe motion with time is the only choice for high heating-rate tests, but fringe tracking (in which one fringe is followed by mechanical or electrical means) should be used for slow tests.

   f. Electrical noise should of course be minimized, but the mechanical noise generated by vibration of the detectors should not be overlooked.

   g. Techniques of surface preparation of the specimen or tabs, application of indentations, alignment of laser, etc., are well-established for room temperature tests and should be followed.

2. Further Research

More research is needed only in the area of tab attachment. The most efficient way to proceed would be to examine this problem alone, i.e., subject the specimens to thermal shock without making fringe motion measurements. In this way, a larger number of tests can be run. Anyhow, the generation and measurement of fringes is not the problem.

It would be prudent to investigate ceramic adhesives under carefully controlled adhesive preparation conditions. Purely mechanical means of

attaching the tabs should also be examined; perhaps a combination of adhesive and mechanical bond would be optimal.

The tab attachment problem is impeding the use of this technique at this time.

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