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N70-42181

NASA CR-72717

ULTRASONIC MEASUREMENT OF CORE MATERIAL TEMPERATURE

by

L. C. Lynnworth and D. R. Patch

PANAMETRICS
A Subsidiary of Esterline Corporation

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-10284
Miles O. Dustin, Project Manager

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National Aeronautics and Space Administration
Scientific and Technical Information Facility
P. O. Box 33
College Park, Md. 20740

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FINAL REPORT

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March 1970

CONTRACT NAS 3-10284

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ABSTRACT

Sheaths designed to protect a thin wire ultrasonic thermometer from the 5300°R graphite/hydrogen environment expected in nuclear rocket engines were tested in the authors' laboratory, and at the Los Alamos Scientific Laboratory (LASL). Eight Ta-sheathed lines were installed in mock fuel elements at LASL and cycled in a graphite/helium environment up to 5400°R. The sheaths survived. Attenuation was overcome, permitting signals to be observed clearly, and measured automatically. The measured transit times exhibited drift, which is not yet explained.

PROGRAM SUMMARY

Temperature measurements are required in the core of a nuclear rocket engine for diagnostic and, ultimately, for control purposes. Until now, these measurements have been obtained mainly by using thermocouples. W/W-Re thermocouples have operated successfully in graphite/hydrogen/nuclear environments up to ~4700°R, and occasionally to 5100°R. But to improve thermocouple reliability, they are usually positioned where temperatures do not exceed 4000°R. Estimates of the maximum temperatures in the core are then obtained by extrapolation. (Temperature conversions inside back cover.)

To obtain direct measures of maximum anticipated core temperatures, without recourse to extrapolation, an alternative to thermocouples is required. An approach based on the temperature dependence of sound velocity in a thin wire has been pursued to develop an alternative sensing technique. The initial temperature goal was 5000°R (Contract NAS 3-7981). The present contract (NAS 3-10284) increased the temperature goal to 5300°R.

Previous laboratory work on the thin wire ultrasonic temperature measuring system, reported by us in Ref. 1-4, showed that Re appeared useful as a temperature sensor for a graphite/hydrogen/nuclear environment up to ~5000°R (accuracy ± 50°R) and 8.7×10^{19} nvt (thermal) and 2.6×10^{19} nvt (fast).

System tests were conducted in a non-nuclear graphite/hydrogen environment at Westinghouse Astronuclear Laboratories in June, 1967. These tests showed that, in some cases, reasonably clear signals could be obtained up to ~4500°R. But above that temperature, the Re sensor and the heated portion of the ~1 m long W lead-in wire attenuated the relatively short pulses (2 to 5μsec) used in these tests.

At that time, such short pulses were used in order to clearly resolve echoes in sensors that were required to be as short as 25 to 75 mm. Subsequently, the sensor length restriction was relaxed to allow up to 125 mm lengths. This opened up the possibility of using longer pulse widths, corresponding to lower frequencies and lower attenuation. To the extent that attenuation could be overcome by resorting to lower frequencies, and to the extent that a sheath protection system could prove successful, operation above the Re/W/C eutectic, ~4740°R, above the Re/C eutectic, 4966°R, and even to 5300°R appeared reasonable (Re melts at 6216°R [3180°C]).

Accordingly, the primary goals of the present three-phase contract were to determine the temperature above which a sheath was needed to protect Re in a graphite/hydrogen environment. This included:

I. determining the maximum usable frequencies (minimum pulse widths) for Re sensor lengths up to 125 mm, and of 0.25 to 2.5 mm dia, for temperatures up to 5300°R;

II. designing, constructing and testing sheaths intended to protect Re from a graphite/hydrogen environment up to 5300°R;

III. testing the complete thin-wire ultrasonic temperature measuring system in a mock fuel element at the Los Alamos Scientific Laboratory (LASL); and

IV. determining the capability of a bare rhenium sensor to measure temperature in a graphite reactor core.

In Phase I, sound velocity was measured in Re up to its melting point, to establish the feasibility of using Re to at least 5300°R, and to generate an approximate calibration curve beyond the limits of equipment available in the previous contract. Attenuation, α, was then measured in a preliminary manner in Re and W to 5300°R using a 10 µsec pulse width. It was observed that α increased rapidly in the 3000-4000°R range, reached a maximum of ∼0.05 db/cm for Re and ∼0.02 db/cm for W at ∼4500°R and then decreased somewhat as 5300°R was approached. Attenuation was later measured for pulse widths as long as 40 µsec, the longer pulses generally giving the expected lower attenuation. (For a 125 mm Re sensor, for non-interfering echoes, the maximum allowable "video" pulse width, measured between zero crossings of the pulse's main lobe, is ∼25 µsec at room temperature, and ∼35 µsec at 5300°R.) Besides these velocity and attenuation measurements in vacuum, velocity was measured in self-heated Re wires surrounded by graphite felt to 4800°R. Above ∼3500°R, enough carbon diffused into the Re in one hour to perturb the calibration, in fact making sound velocity in the resulting material relatively insensitive to temperature. This showed that a sheath was needed not only to permit operation beyond the Re/C eutectic, but even at lower temperatures, to avoid calibration drift. Also in Phase I, temperatures were measured at two adjacent sensor regions on one wire using, sequentially, two different modes of vibration - extensional and torsional waves. Details are reported in Ref. 2.

The major conclusion reached in Phase I was that, for the expected temperature profiles, a sheath would be required. Also, use of video pulse widths of at least 10 µsec could overcome attenuation in a W lead-in and in a Re sensor, for sensor temperature up to 5300°R.

The object of Phase II was to develop a sheath to protect Re from the effects of graphite up to 5300°R.

Regarding candidate sheath materials, the philosophy both in this program and its predecessor has been to rely on thermocouple sheath designs as much as possible. Thus, while several oxide and composite or alloy sheath materials have been suggested, attention has been focussed on Ta and W (W-2% ThO_2) tubes as sheaths. Depending on future temperature requirements, W-Re sheaths should also be considered.

The most notable difference between the ultrasonic sheaths and those used to protect thermocouples lies in the absence of electrical insulators for the ultrasonic case.

A second difference, explored in only a few ultrasonic test lines, is the use of a short sheath in the region of the sensor only. The idea here is that the effect of carbon and/or hydrogen on a lead-in does not affect the <u>sensor</u> calibration. At the present time, W sheaths are extremely fragile and expensive except in lengths comparable to the ultrasonic sensor. In lengths as short as ~0.3 m, W tubes 2 to 3 mm dia x 0.2 to 0.3 mm wall thickness should give good protection despite graphite and hydrogen, including cycling from liquid hydrogen to 5300 °R temperatures. (W-Re tubes, while less brittle, are eutectic-limited in graphite environments to ~4740 °R.)

The main drawback to short W sheaths lies in the stringent mechanical and acoustical requirements at the joint near the 5300 °R sensor. This problem, however, does not appear insurmountable, and in time may be obviated by the availability of 0.5 to 1 m long W tubes of less brittle character. Some data indicate that W is inferior to Ta as a carbon barrier. But this point does not appear too important in view of the NbC coating in the instrumentation hole which would greatly reduce carbon attack on the W sheath (Ref. 5).

Besides W, only Ta was given serious consideration as a sheath. Ta is less fragile, less costly, available in lengths to 2 m, and is an excellent barrier to carbon, independent of any assist from a NbC coating. The main drawback of Ta is that it hydrides below ~2700 °R (~1200 °C). If, however, in the presence of carbon, Ta is quickly heated ($<$ 2 minutes) above the hydriding range, it will carbide, so that on subsequent cycles, not Ta, but tantalum carbide compounds, face the hydrogen, and may be expected to be relatively resistant to hydriding, compared to pure Ta.

To summarize Phase II, short W and long Ta sheaths were designed, constructed and tested. A wrought W-2% ThO_2 sheath protected a Re sensor from graphite for one hour at 4900°R. A Ta sheath protected one particular Re sensor from graphite for two hours at 4800°R, at 5100°R for over one hour, and then at 5300°R for a few minutes. Details are reported in Ref. 2.

The object of Phase III was to test at LASL in a mock fuel element, sheathed lines designed to operate in a graphite/hydrogen/nuclear environment to 5300°R.

A series of two tests were run at LASL. In the first series, conducted in January, 1969, various short and long sheaths, and single and double sheaths, were tested at high temperatures, including a ~one-hour run in a graphite/hydrogen atmosphere at ~5000°R. These tests demonstrated survival of Ta sheaths, and showed that pulses longer than 10 μsec could overcome attenuation at 5000°R. Unfortunately, despite numerous preliminary tests in our laboratory to ~5300°R, some unexpected difficulties arose in these initial tests at LASL, which confused signal interpretation. These difficulties were later understood, based on observations during the second series of LASL tests in June 1969.

In the second series of LASL tests, eight 0.25 mm wall thickness Ta-sheathed lines were installed in NbC-coated instrumentation holes in mock fuel elements and cycled in a graphite/helium environment. These lines yielded 141 points between 3800 and 5400°R, of which 68% fell within 100°R of a straight line (least squares fit) calibration of ultrasonic transit time in Re sensors vs corrected optical pyrometer temperature. The data exhibited drift and hysteresis, not previously observed, which still need to be explained.

In summary fashion, attention is also drawn to preliminary tests on W wires as small as 0.025 mm (0.001 inch) dia. These room temperature tests were conducted to demonstrate an approach to avoiding gamma heating errors at high temperatures by aiming for a high surface/volume ratio for the sensor.

Tests were also done on composite SS/MoTa sheaths, the SS/Mo portions being positioned where temperature would be expected to remain below ~2200°R, i.e., in the hydriding range for Ta. Further, tests on Ta tubing verified that exposure to hydrogen in the hydriding range caused severe embrittlement and tube failure. But heating Ta tubing beyond 2200°R in three minutes or less avoided hydriding. Apparently, in such a brief exposure, not enough hydrogen diffuses into the Ta to be catastrophic.

The significant conclusions reached in Phase III are as follows:

1. Calibration drift, observed at LASL but not in previous laboratory tests at Panametrics, is not yet understood. Until this drift can be explained and/or eliminated, the Re sensor/Ta sheath system cannot be used in graphite/hydrogen environments to 5300°R.
2. Using Ta sheaths, W lead-in lines and Re sensors developed in this program, clear signals could be obtained beyond the contractual goal of 5300°R. Oscillograms were obtained up to 5330°R, and an automatic reading of transit time in the Re sensor was obtained at 5400°R. This was accomplished using a 10 μsec pulse width to overcome attenuation in the ∼1 m long W lead-in and the ∼100 mm long Re sensor.
3. A Ta sheath survived a ∼1-hour soak at ∼5000°R in a graphite/hydrogen atmosphere at LASL.
4. Another Ta sheath survived numerous cycles from ∼500 to nearly 5000°R in a graphite/helium environment at LASL, including over two hours accumulated exposure above 4000°R, and a rapid drop from ∼5000 down to ∼500°R (due to power interruption) prior to successful, brief use to 5330°R.
5. Uncertainties relative to the January LASL tests, concerning diffusion bonding of the Re sensor to the sheath, horizontal vs vertical installations, interference between two closely spaced transducer coils and effect of seal fitting, were clarified by a series of controlled experiments in June at LASL. These problem areas were avoided using the simpler line in the latter tests (June 1969).
6. Multiple echoes in the Re sensor were observed up to ∼4500°R. This permits temperature sensitivity to be magnified by the number of reverberations. Thus, sensitivity of 10°R was achieved.
7. The optimum design for an ultrasonic line depends on the maximum temperature, the temperature profile, and cyclic behavior. Designs for 4000, 4500, 5000, and 5300°R may differ substantially.
8. Even if all June 1969 LASL data points between 3800 and 5400°R are considered, 68% of these points fall within 100°R of a straight-line calibration fit, in the least squares sense.

Recommendations for future work are as follows. To the extent that interest continues for a reliable temperature sensor for a graphite/hydrogen/nuclear environment, it is recommended that experiments be performed to explain the drift observed in the LASL tests. The effects of contaminants, the possible use of materials of higher purity, such as higher purity Re, and other new candidate sensor materials (e.g., NbC, pyrolytic graphite, vitreous carbon, possibly W), use of a getter, use of the same material for sensor, spiral isolator and sheath, etc., remain to be considered in greater detail. Transducers and lines could be tested in a simulated nuclear rocket engine environment at Jackass Flats alongside thermocouples. Now that attenuation has been overcome, the possibility of maintaining adequate temperature sensitivity in a short sensor, by means of multiple echo, pulse superposition and/or resonance techniques should be explored. Some of these areas for future acoustical development are not restricted to the nuclear rocket engine environment, and should be considered in the light of current applications.

INTRODUCTION

Statement of the Problem

One of the most important measurements required in nuclear rocket engine technology is the measurement of core temperature. This measurement has proven to be extremely difficult because of the high temperature involved, because of compatibility problems with some of the materials involved (graphite and hydrogen) and because of the intense transient and sustained neutron and gamma fluxes. Additional difficulties stem from the possibility of temperature overshoot, high pressure, flow, accessibility and geometrical restrictions, shock and vibration levels expected in some locations, etc.

Until now, these measurements have been obtained mainly by using thermocouples. W/W-Re thermocouples have operated successfully in graphite/hydrogen/nuclear environments up to ~4700 °R and occasionally to 5100 °R (Ref. 6-9). But to improve thermocouple reliability, they are usually positioned where temperatures do not exceed 4000 °R. Estimates of the maximum temperatures in the core, ~5000 to 5300 °R, are then obtained by extrapolation. (Temperature conversion chart inside back cover.)

With respect to high temperature measurements in a nuclear environment, some of the limitations on thermocouples, such as transmutation effects, were appreciated in the early sixties (Ref. 10). Continued thermocouple testing has, on the one hand, led to improvements in materials, construction and performance. On the other hand, continued meticulous thermocouple testing has brought to light some of the more subtle difficulties, some of which include: insulator stability; thermal stressing in grounded-junction construction; thermoelectric outputs from insulators, coupled with different diameter thermocouple legs; measured emf dependent upon He or H_2 atmosphere; tungsten transport (Ref. 6-9, 11).

To directly measure maximum core temperatures, ~5300 °R, without recourse to extrapolation, an alternative to thermocouples is required. An approach based on the temperature dependence of sound velocity in a thin wire has been pursued to develop an alternative sensing technique. (Continued development of the ultrasonic thin line technique, both in U. S. laboratories and abroad, has led to improved performance on the one hand, and, on the other hand, a better understanding of the ultrasonic technique's limitations.

Phase III Objective

The main objective of Phase III was to test the complete thin-wire ultrasonic temperature measuring system in a mock fuel element at the Los Alamos Scientific Laboratory (LASL), in a graphite/hydrogen atmosphere, at high pressure, up to the maximum temperature capability of the furnace, with 5300°R for one hour as a goal.

SHEATH DESIGN CONSIDERATIONS

In the present program, sheaths were required (a) to permit the Re sensor to be used to 5300°R, well above the Re/C eutectic of 4966°R, and (b) to prevent decalibration of the Re sensor due to carbon diffusion into it. (The possibility of using bare, unsheathed sensors in a NbC-coated hole, wherein the graphite available, especially below 5000°R, is smaller than in an uncoated hole, was attempted only once, in field tests at LASL in June 1969. Signals were obtained up to 4732°R in this instance.)

In a few ultrasonic test lines, we tested a short sheath in the region of the sensor only. The idea here is that the effect of carbon and/or hydrogen on a lead-in does not affect the sensor calibration. At the present time, W sheaths are extremely fragile and expensive except in lengths comparable to the ultrasonic sensor. In lengths as short as ~0.3 m, W tubes 2 to 3 mm dia x 0.2 to 0.3 mm wall thickness should give good protection against graphite and hydrogen, including cycling from liquid hydrogen to 5300°R temperatures. (W-Re tubes, while less brittle, are eutectic-limited in graphite environments to ~4740°R.)

The main drawback to short W sheaths lies in the stringent mechanical and acoustical requirements at the joint near the 5300°R sensor. This problem, however, does not appear insurmountable, and in time may be obviated by the availability of 0.5 to 1 m long W tubes of less brittle character. Some data indicate that W is inferior to Ta as a carbon barrier (Ref. 5). But this point does not appear too important in view of the NbC coating in the instrumentation hole which would greatly reduce carbon attack on the W sheath.

Besides W, only Ta was given serious consideration as a sheath. Ta is less fragile, less costly, available in lengths to 2 m, and is an excellent barrier to carbon, independent of any assist from a NbC coating. The main drawback of Ta is that it hydrides in the ~1200 to ~2200°R range. If, however, in the presence of carbon, Ta is quickly heated (< 3 minutes) above the hydriding range, it will carbide, so that on subsequent cycles, not Ta, but tantalum carbide compounds, face the hydrogen, and may be expected to be relatively resistant to hydriding, compared to pure Ta.

To summarize Phase II, short W and long Ta sheaths were designed, constructed and tested. A wrought W-2% ThO_2 sheath protected a Re sensor from graphite for one hour at 4900°R. A Ta sheath protected one particular Re sensor from graphite for two hours at 4800°R, at 5100°R for over one hour, and then at 5300°R for a few minutes. Details are reported in Ref. 2.

Figures 1a-f show the sheath systems that were considered for use in the January 1969 Los Alamos tests (henceforth denoted "preliminary LASL tests"). In order to check the acoustic isolation between the long sheath systems and the ultrasonic lines, tests were performed using inexpensive stainless steel (SS) tubing and an acoustic line consisting of a Ta or W lead-in and a Re sensor. It appeared that, at least at room temperature, no spiral spacer wire was needed to isolate acoustically the line from the sheath. This was attributed to the lightness of the ultrasonic line and to the small area of the line which is in contact with the sheath. Under these conditions, very little of the sound energy would be coupled out of the line.

In independent tests with horizontal lines at high temperatures, where a SS lead-in was run through a 20 ft SS tube at ~1700°R, S. S. Fam found that, whether using a spiral spacer wire or not, attenuation was small, and did not interfere with reading echoes in a Re sensor inside a (vertical) Ta tube at ~5500°R (Ref. 3).

In order to further check the acoustic isolation between the sheath systems and the ultrasonic lines, room temperature tests were performed using Mo and Ta tubing and a W acoustic line. Figure 2 presents oscillograms which appeared to show that the sheath system planned could be used with the ultrasonic line with no serious effect on the ultrasonic signal. At that time, repetition of hour-long high temperature tests was not thought necessary, as such tests had already been run in Phase II.

Fabrication of Preliminary Sheaths. Since the sensor echoes in Fig. 2 appeared reasonably clear, the problem of joining tubes of different refractory materials was reviewed next. Based on this review, it was decided to electron beam weld some of the joints and to braze the rest. The decision was based on the following considerations:

1) If the joint was likely to see a temperature greater than the melting point of the possible brazing materials, then electron beam welding should be used.

2) Usually a welded joint will be embrittled. If it is necessary to avoid this embrittlement, the joint should be brazed, operating temperatures permitting.

3) Depending upon the specific materials and geometries being used, electron beam welding or brazing might be the appropriate way to join tubing. For example, Ta can be joined to SS by a nickel-copper braze; Ta/Ta should be welded.

The joints between Ta/Mo or Ta/SS were to be located in the LASL furnace at port #1, where the temperature is approximately 2500°R. The joints had to withstand the temperature and materials environment and the differential pressure expected in that furnace.

Ta Hydriding. It has been reported by other investigators that the integrity of Ta is destroyed in the presence of hydrogen at room temperature, i.e., the Ta apparently hydrides and, as a result, breaks up into pieces. To verify this, we placed Ta tubing in a hydrogen atmosphere at room temperature for twenty minutes. The hydrogen pressure was 200 psi. The tubing was examined after this exposure and no noticeable effects were observed by the unaided eye. It was then planned to perform the same experiment at higher temperatures.

In order to examine high temperature hydriding of Ta, a high pressure gas container was built. It was planned to use this container to determine the lifetime of Ta in 150 psi of hydrogen, and at 2000°R, the temperature at which the rate of absorption of hydrogen is a maximum.

Tantalum wire 100 mm in length and 1/2 mm in diameter was self-heated in ten atmospheres of hydrogen. Simultaneously, the Ta wire was monitored ultrasonically. The temperature of the Ta sensor was determined by measuring the time necessary for an extensional wave to traverse it. The Ta wire lasted for approximately three minutes before splintering. It is to be understood that even brief exposures, however, may initiate failure of Ta sheaths.

Additional tests were performed under the same conditions by self-heating a Ta tube measuring ~1.5 mm OD x 1 mm ID. The temperature of the tube was determined by placing an ultrasonic temperature sensor inside the tube. The Ta tube maintained its integrity for approximately three minutes at a temperature of 2000°R in a hydrogen atmosphere.

These experiments showed that if the LASL furnace were heated very rapidly, i.e., heated above 2000°R in less than ~1 min, then the problems associated with hydriding should be minimized. Since the furnace was reportedly capable of being heated at a rate of ~30°R/sec, no problem with hydriding was anticipated (Ta hydrides primarily in the 1200-2200°R range).

Barrier coatings to postpone hydriding until the Ta got above $\sim 2200^{\circ}$R were also considered. Plasma-sprayed Mo was one possibility. The permeability of H_2 in Mo was calculated using the following equation:

$$P\left[\frac{\text{cc (STP) mm}}{\text{sec atm}^{1/2}\,\text{cm}^{2}}\right] = 0.166\ \exp\left[\frac{-19300\ \text{cal/mole}}{RT\,({}^{\circ}K)}\right]$$

where R is the universal gas constant, mm the thickness of the sheath, cm^2 the amount of exposed area, and $atm^{1/2}$ is the square root of the driving pressure. [Chandler, W. and Walter, R., "Hydrogen Effects in Refractory Metals," Symposium on Metallurgy and Technology of Refractory Metal Alloys, Washington, D. C. (25-26 April 1968).] The amount of hydrogen that will diffuse through a 600 mm (2 ft) length of ~ 2.5 mm OD Mo with a wall thickness of 0.25 mm at a temperature of 3240°R for a period of one hour and a driving pressure of 40 atmospheres is given by this equation as approximately 12 cc. Since the above equation is the best fit to data of five different authors, the result is only approximate.

Another possible coating material, suggested by C. R. Tallman of LASL, was Re, chemically vapor deposited.

These approaches would yield some protection during the first heat-up, until the coating melts. After the coating melted, tantalum carbide would form, to provide integrity for subsequent heating cycles.

Pre-carbiding a Ta tube is another possibility, if fragility problems associated with handling can be overcome--not an unrealistic possibility, based on our tests at LASL.

Preliminary Field Tests at LASL

The primary purpose of the field test was to measure temperature for one hour in a graphite/hydrogen environment with a goal of 5300°R. The secondary purpose was to determine if the sheath systems would protect the Re sensors from graphite, and if the sheath systems would survive a graphite/hydrogen environment for one hour.

The sheath systems that were to be used in the preliminary LASL tests were tested and found to be leak tight. The ultrasonic lines were tested in the sheaths at room temperature, and briefly, at slightly elevated temperatures.

Figure 3 shows a block diagram of the experimental apparatus used during the week of January 20, 1969. With this arrangement it was possible to selectively monitor any one of up to five sensors. The electronics was switched manually to selected ultrasonic lines.

Above ∼2000 R, as determined by an optical pyrometer, the ultrasonic signals generally could not be identified due to noise problems, i. e., the signal-to-noise ratio was less than two. The reasons for the noise can probably be attributed to contact between the ultrasonic line and sheath as a result of bowing or spot welding. Compression fittings with sealants other than neoprene may also have contributed to the noise.

Six sheaths were tested in three separate runs. The most important positive results of these preliminary tests were that the Ta sheaths survived the graphite environment for one hour at ∼5000°R and apparently protected the Re sensors from the graphite. Also, after preheating in He, a Ta sheath survived for one hour at ∼4800°R in a H_2 environment. Use of long coils apparently overcame attenuation, which was the major acoustical problem in our tests at Westinghouse Astronuclear Laboratories (WANL) of June 1967 (Ref. 1)

In these preliminary LASL tests, although signals could generally not be identified, it is probably significant that, on at least one occasion, the line apparently freed itself from the sheath long enough for signals to be clearly discerned.

Information now available indicates that the principal and most significant difference between our previous tests, in which clean signals were obtained up to high temperatures, and the preliminary LASL tests, in which signals were confused, even at moderate temperatures, appeared to be simply the orientation of the sheath and line (vertical vs horizontal).

In this program, prior to preliminary LASL testing, high-temperature testing of wires in sheaths had been inadvertently restricted to vertical orientations. For example, the WANL furnace required vertical installation. Should tests be required at Jackass Flats, this too would be a vertical installation. Pre-WANL tests in our graphite SRI furnace naturally involved vertical access. In the present contract, Phase II, wires were tested in 500 mm long Ta tubes vertically oriented (Ref. 2, Fig. 4). In all of these vertical tests, there had been no indication of metallurgical bonding of line to sheath. Further, our recent tests in vertical sheaths were run without benefit of spiral spacer wires, and yet the two sensor echoes were clearly resolved at 5100°R for an hour. Independent of this program, our experiences, and those of other investigators, indicated that vertical installations may be less subject to metallurgical bonding than horizontal ones, but the interactions between lines of sheath depend not only on orientation, but also on materials, clearances, duty cycles, position (centering) of the line in the sheath, the environment, etc.

Whether the LASL furnace, being horizontal, adequately simulated gravity or other acceleration-induced loading conditions to be expected at Jackass Flats or in free space, was not in question. It was clearly necessary to simulate at Panametrics, as closely as possible, the LASL furnace conditions prior to June field tests. Conduct of these latter field tests was agreed to be contingent upon successful operation of a complete sheath system at Panametrics, under conditions essentially simulating the LASL furnace.

FURTHER LABORATORY TESTS

Heading Horizontally--LASL Furnace Simulation. To more closely simulate conditions of the LASL furnace, it was decided to run an ultrasonic line inside a self-heated sheath in a horizontal position, Fig. 4.

The horizontal system, run in nongraphite vacuum environment, was cycled 10 times to at least 5000°R, with a lead-in line temperature of 4500°R. The sensor was also cycled in the same line 15 times to about 4000°R. During this cycling procedure, the lines were brought to high temperature for no more than one minute at a time. (Exposure was purposely kept brief to avoid overheating the fused silica tube--see Fig. 4.) There was no evidence of the Re sensor joining to the Ta sheath, which was a suspected problem at LASL. However, for longer exposures at high temperature, acoustical and/or metallurgical isolation was considered necessary.

During this cycling procedure it was observed that at room temperature the ultrasonic reflections at the beginning and end of the sensor were well defined and well separated. At high temperature, although attenuated, the signals remained readily measurable.

When long lines are at temperatures in the range of 5000°R, it becomes necessary to use a low frequency, to overcome attenuation. However, in the LASL test, maximum allowable sensor length was 125 mm (5 in.). This means the reflections at the beginning and end of the sensor can interfere if the broad pulse widths afforded by the low frequency (75 mm) coil are not properly separated. This can be a problem whether one uses one coil or two.

As a compromise, it was decided to experiment with a single, shorter coil, to separate signals at the expense of increased attenuation. Consequently, one 50 mm coil, pulse width 15 μsec, was chosen for the horizontal heating experiment. At 5000°R the attenuation was not excessive. The signals were measurable at high temperature while being clear and well separated at low temperature. The single 50 mm coil lent itself very well to being affixed to the end of the sheath.

Since it turned out to be relatively simple to self-heat the sheath (Fig. 4), it was decided to design and construct a more rugged self-heating system capable of sustained operation at 5300°R.

Self-Heated Furnace Capabilities. The furnace was designed to handle approximately 300 amperes. This could easily self-heat sheaths up to at least 6 mm dia x 1/4 mm wall to temperatures of 5300°R.

The furnace could self-heat bare wires as well as sheaths. The item being heated could be any length up to 1 m. Environments could be vacuum or pressure, inert gas, or hydrogen. Operating position, horizontal or vertical, could also be changed while the experiment was in progress.

For some experiments a sight port would enable observation of the sensor by looking axially down the center of the tubular heater or sheath.

Physically, the furnace measured ~1.6 m long x 75 mm dia. It was mounted to rotate about a horizontal axis. The furnace was water-cooled on the outside and had multiple Ta reflecting shields on the inside. The furnace also employed fused silica tubes for insulators.

A furnace with these dsign characteristics was constructed (Fig. 5).* Minor problems relating to electrode connections and outgassing were quickly eliminated. Subsequently, the furnace was used to heat acoustic lines to a temperature of 5300°R for one hour in a graphite environment.

In the first two experiments conducted in the newly constructed furnace to 5300°R, the sensor end reflection attenuated sharply and distorted in shape after ~ten minutes, in <u>both</u> experiments (Fig. 6). The probable cause was a fusing of the sensor to the Ta heating tube (sheath).

To correct this problem, isolation was necessary. A 0.05 mm (0.002 inch) dia W wire was spiraled on the sensor. In the next two experiments, with a spiral wire on the sensor and in a graphite environment, the signals, though attenuated, were clean and lasted an hour at 5300°R with essentially no deterioration in signal over the one hour span (Fig. 7).

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*Description of Parts (see Fig. 5) - Item #1, Tantalum baffles (0.05 mm) 6 layers; #2, Inner fused silica tube - provides added heat insulation; #3, Tubular electrode containing alumina tube which acoustic line(s) are fed through; #4, 4a, Electrode adapters - interchangeable with adapters of a different size to match tantalum heating tube OD; #5, 5a, Aligning sleeves - hold electrodes in alignment inside of stainless steel tube (8); #6, Expansion electrode - allows for expansion and contraction of tantalum heating tube without buckling or high resistance contact; #7, Sight port - mounted on the end of electrode (6) enables observation of sensor by looking axially down the center of tubular heater; #8, Stainless steel tube - allows electrode, heater, baffle and inner fused silica tube assembly to enter outer shell (10) as a unit and in alignment; #9, Outer fused silica tube - fits inside outer shell (10) and provides added insulation; #10, Outer shell - shown with cooling coils (~30 m) and mounting bracket; #11, Acoustic lead-in line; #12, Rhenium sensor.

These results and hardware were discussed and witnessed on June 16, 1969 by M. O. Dustin, NASA technical monitor. A line that had previously been run to 5300°R was tested on that date to about 4500°R to demonstrate that the complete temperature sensing system was ready for final tests at LASL the following week.

Final Test Preparations. Within the two week period prior to the LASL test of June 23-27, 1969, several transducers and lines were completed. They were then installed in a total of nine sheaths. Six of these sheaths were provided by LASL; three remained from the preliminary LASL test of January 1969.

The simpler sheaths consisted of Ta tubing ~2.5 mm OD x 0.25 mm wall x ~1 m long, brazed to SS lead-out tubes. The hot end was plugged with Ta; the cool end was closed with a coaxial connector. A single transducer coil, without any magnet, was contained and mounted near the cool end, shielded electrically by the sheath.

Small Diameter Sensors. Phase I of this program was concerned with Re sensor diameters in the 0.25 to 2.5 mm range. In the first part of Phase III, some additional but only preliminary consideration was given to much thinner sensors - down to 0.025 mm (0.001 inch) dia.

One of the important problems in measuring temperature in nuclear reactors is the error caused by gamma heating. In order to overcome this problem sensors down to ~0.025 mm diameter might be used. The reason for using such small sensors can be easily understood. The heat energy radiated by a sensor is proportional to the surface area while the energy absorbed due to gamma heating is approximately proportional to the volume. For a sensor of fixed length, the ratio of surface area to volume is equal to $2/r$, where r is the radius of the sensor. Thus, by using sensors with very small diameters, the errors due to gamma heating will be reduced. (Using a computer program at LASL to estimate temperature errors due to gamma heating, C. R. Tallman verified the above conclusion. In general, the smaller the overall temperature measuring package diameter, the smaller the gamma heating errors.)

Experimentally, echoes were obtained from a 63 mm long x 0.05 mm dia W sensor. The sensor was constructed by etching a 0.75 mm dia W wire.

Subsequently, echoes were obtained from a 50 mm long x 0.025 mm dia W sensor The sensor was constructed by etching a 0.75 mm dia W wire. Later, electropolishing was found effective in reducing Re wire diameters from 0.25 mm down to 0.13 mm. The possibility of fabricating polycrystalline W and Re wires down to 5μ (0.0002 inch) dia is under investigation at Battelle-Geneva (process due to Dr. B. Lux).

FINAL FIELD TESTS AT LOS ALAMOS SCIENTIFIC LABORATORY

Again, the object of Phase III was to test at LASL in a mock fuel element, sheathed lines designed to operate in a graphite, hydrogen/nuclear environment to 5300°R, for one hour.

In the second series of LASL tests, June 1969, eight 0.25 mm wall thickness Ta-sheathed lines were installed in NbC-coated instrumentation holes in mock fuel elements and cycled in a graphite/helium environment (Fig. 8). These lines yielded 141 points between 3800 and 5400°R, of which 68% fell within 100°R of a straight line (least squares fit) calibration of ultrasonic transit time in Re sensors vs corrected optical pyrometer temperature. The data exhibited significant drift, not previously observed, which still need to be explained.

Lines. As is evident from Fig. 8a, a typical Ta-sheathed ultrasonic line used in the final LASL field tests is handled like a sheathed thermocouple line. It is slender enough so that, as was later demonstrated, several can be installed in adjacent holes in a mock fuel element. A single 50 mm long transducer coil is contained within the SS sheath portion at the cool end (Fig. 8b). No biasing magnets are used. A small coaxial connector, threaded into and thereby grounded to a sheath, connects to a 6 m cable to convey echo signals (as on oscilloscope in Fig. 8a) from test area to the instrumentation room next door. Schematically, the test is basically as depicted in Fig. 3, except only one T/R coil is used on pulse-echo mode.

Results

Individual plots of measured round trip transit time in the 100 mm Re sensors, vs corrected optical pyrometer temperature, are contained in Figs. 9-16. Results on a bare line inserted from the "opposite" end a short distance are given in Fig. 17. Figure 18 gives the exposure history of the sheath denoted "#6." This sheath was run nearly ten hours above 3000°R, including ~5-1/2 hours above 4000°R. Figure 19 is a photomicrograph showing carbon penetration into this sheath, at a sight port location point aggravated by the fact that the NbC coating in the hole was removed, in drilling for optical access. This photomicrograph is due to Dr. C. P. Kempter of LASL, who also gave us permission to reproduce Fig. 20 prior to publication (Ref. 12). For the Ta-sheathed lines, analysis of the 141 points between 3800 and 5400°R, yielded a best straight-line approximation, in the least-squares sense, of the form:

$$T_{\substack{\text{corrected}\\ \text{opt pyro}}} = 111\, t_{\mu\,\text{sec}} - 2729^{\circ}\text{R}\ .$$

It was observed that 68% of the data points fall within ± 100°R of this line. This spread is mostly due to drift, not yet explained, plus some recrystallation, and some spread due to sensors not all being of identical lengths.

Transit times were measured automatically, using a Panatherm® 5010, to the nearest 0.1 μsec. While not necessary for the transit time measurements, numerous oscillograms were recorded to provide a more complete record of noise buildup, attenuation, and for illustrative purposes. Representative oscillograms are included in Fig. 21. Particular oscillograms are referred to below in support of certain conclusions concerning both the preliminary LASL tests of January, and the final LASL tests in June.

The reference temperature for sensors was obtained by sighting an L & N optical pyrometer through a ~6 mm dia hole in the graphite heater, then through a ~3 mm dia x 3 to 6 mm deep hole in the graphite mock fuel element, onto the Ta sheath at a point corresponding to the center of the Re sensor. Blackbody conditions were assumed, and window corrections based on previous calibrations were applied. In one instance the flatness of the temperature profile was verified by moving a Mo-sheathed W/W-Re thermocouple back and forth near the port in question. This cross check indicated that, when port #4 appeared to be 3520°R (pyrometer), the thermocouple-indicated temperature within about ± 25 mm of that port was within 30°R of 3460°R.

Discussion

Sheaths run in the final field tests at LASL are identified in Table I.

TABLE I. SHEATH IDENTIFICATION FOR FINAL FIELD TESTS

Date of Test	Sheath No.	Remarks (A = "annealed"; N = "not annealed")	
24 June 1969	4,2	Sensor at port #4	
25	2	Bare line also run at port #4	
		W lead-in	Re sensor (at port #3)
26	3	N; spiral	N; spiral
	6	A; spiral	A; spiral
	7	N; spiral	N; spiral
27	1	N; double spiral	N; double spiral
	6	A; spiral	A; spiral
	7a	N; spiral	N; spiral
	8	N; spiral	N; spiral

Sheath #4 was tested in the first two runs on 24 June 1969. Figure 21-#4-1 is a room temperature check of this sheath installed in a mock fuel element. A Tektronix 565 dual beam scope (ser. no. 001045) displayed the Panatherm® 5010 "Receiver Monitor" (upper trace, 5 V/cm, 100 μsec/cm) and the "Selector Monitor" (lower trace, 2 V/cm, 100 μsec/cm) outputs. The Receiver Monitor output is the amplified echo pattern. The Selector Monitor output includes a pair of pedestals corresponding to selected echoes between which time is automatically measured. (In this case, the time is 46.2 μsec.) Further details are in Ref. 3. For all oscillograms, Fig. 21, the sweep was maintained constant, 100 μsec/cm.

The second oscillogram, Fig. 21-#4-2, was taken at a corrected optical pyrometer temperature of 4766°R. There is about 5 db attenuation in the W lead-in, and another 5 db in the Re sensor. Transit time in the sensor increased to 69.3 μsec, clearly readable. Even a multiple echo is visible, with polarity reversed since the sensor's impedance is less than that of the larger diameter lead-in.

Figure 21-#4-3, obtained nine minutes later at about the same temperature, shows a spurious echo before the Re sensor echoes, confusing interpretation. Cooling down to 3890°R (Fig. 21-#4-4) appears to have removed the early echo, but sensor echoes are still not clear. Further cooling to 2440°R (Fig. 21-#4-5) cleared up the echoes, the time between selected echoes being 52.6 μsec. This is about a 1% decrease, apparently due to annealing (Fig. 21-#4-6).

On reheating to 3465°R (Fig. 21-#4-7) (Run 2) echoes were still clear (56.4 μsec), but four minutes later (Fig. 21-#4-8) the spurious, early noise pulse reappeared, and attenuation increased. Power was turned off. Half an hour later the line was removed from the furnace, and then from the sheath. It was observed that the spiral spacer had slipped about 25 mm. This slippage would appear to be related to the spurious early pulse.

Sheath #2 was tested in the next two runs. Oscillograms at room temperature and at 4524°R are shown for Run 3 (Fig. 21-#2-1 and 2, respectively). Although the 4524°R echoes are readable (65.4 μsec), an early noise pulse is developing. Because of time restrictions, the run was terminated. After cooling overnight, the room temperature transit time decreased to 45.1 μsec, again about 1% less than its value of 45.6 μsec prior to Run 3.

Sheath #2 was cycled on 25 June in Run 4 to 4732°R, including three hours above 4000°R, during which signals remained readable up to 4546°R.

On cooling to room temperature, the transit time was 45.8 μsec, an unexpected increase over the previous values of 45.6 and 45.1 μsec.

During Run 4, a bare line was inserted from the right side of the furnace as seen in Fig. 8a. Readings after heat-up were generally within about ± one microsecond of those for the sheathed line (Table II). Based on our Phase I work on carbon diffusion into Re, it would appear that the NbC coating substantially retarded carbon transport into the sensor, or else the bare and sheathed Re sensors would have shown systematic discrepancies of ~4 μsec (Ref. 2, NASA CR-72395, Figs. 14-16). In passing, it is of interest to note the possibility that up to 4000°R, or maybe even up to 4500°R, a bare line might be feasible, to the extent that NbC would, in a sense, play the role of a sheath between graphite and the line. This possibility, not yet demonstrated as reliable or accurate, is potentially attractive both from a cost standpoint as well as from a thermal response standpoint. Further, if one considered optimizing the line for use to only 4000°R, use of multiple echoes could provide sensitivity of 10°R in a 100 mm Re sensor (automatic reading of a 4700°R multiple echo is illustrated in Fig. 21, Run 6, Sheath #6).

Examining the data from the first four runs, two troublesome points are apparent:

1. (1) Room and elevated temperature calibrations show drift; and
2. (2) Spurious noise pulses develop in front of the Re sensor after extended periods at elevated temperature.

Accordingly, to understand these points better, and to possibly eliminate the sources of difficulty, the next test, Run 5, (see Tables I and III for conditions) was planned so as to vary several conditions <u>inside</u> three otherwise (nominally) identical Ta sheaths. Because of time and available material (maximum Ta tubing length ~1 m) limitations, it was decided to make up a few new Ta-sheathed lines and test them with sensors at port #3 instead of port #4. This avoided the usual Mo or SS extension sheath at the cool end, and also meant that tests would be restricted to a He atmosphere. If hydrogen were used, that portion of the Ta near the cool end, between ~1200 to 2200°R, would hydride within about three minutes. This limitation on gas environment was viewed as a reasonable sacrifice, since Ta tubes had already survived hydrogen exposure at high temperature during the preliminary LASL tests of January 1969.

The contents of Sheaths 3, 6 and 7 are identified in Table I. The data are in Table III, and may be visualized with reference to the corresponding plots and oscillograms.

TABLE II. COMPARISON OF BARE AND SHEATHED RE SENSORS IN RUN 4, 25 JUNE 1969

		ROUND TRIP TRANSIT TIME, μsec		
Clock Time, Hours	Corrected Opt Pyro Temp., °R	Sheath #2 (annealed)	Bare Line (not annealed)	Difference (Bare-Sheath)
1335	Rm Temp	45.2	47.1	1.9
1350	-	55.2	56.9	1.7
1353	3360	55.5	56.7	1.2
1355	3423	55.6	56.6	1.0
1359	3440	55.6	56.3	0.7
1411	3467	56.3	56.7	0.4
1423	4105	61.5	61.8	0.3
1432	4137	61.5	61.9	0.4
1444	4447	64.6	65.2	0.6
1448	4484	64.5	65.1	0.6
1501	4266	62.1	63.1	1.0
1514	3986	59.6	59.7	0.1
1518	-	56.1	55.6	-0.5
1520	3562	56.0	-	-
1530	2893	52.8	52.7	-0.1
1533	2870	52.6	52.6	0.0
1542	4105	60.7	61.1	0.4
1549	4380	63.3	64.7	1.4
1559	4556	64.2	65.3	1.4
1609	4732	64.4	65.5	1.1
1617	4546	62.3	-	-
-	4008	59.5	-	-
-	4028	57.9	-	-
26 June, 0820	Rm Temp	45.8	-	-

TABLE III. ULTRASONIC DATA FOR LASL TESTS ON 26 JUNE 1969

Clock Time 26 June 1969	Port #3	Sheath #3 µs	Sheath #6 µs	Sheath #7 µs
1130	Rm Temp	46.4	46.4	46.9
1153	3068 @ #4	-	64.3	66.7
1206	-	-	62.4	66.0
1211	3096 @ #4	-	64.5	66.7
1214	-	Recover @ ~53	-	-
1335	Rm Temp	46.1	46.2	46.5
1358	4498	out	65.0	65.4
1400	4509	-	64.8	66.1
1402	4509	-	64.7	65.7
	Going Down	64.8	-	-
1409	4286	63.4	63.2	64.4
1417	4208	63.9	61.9	64.8
1428	3946	60.0	58.5	60.4
1438	3475	55.9	56.0	56.4
1446	3884	59.4	58.9	59.2
1458	4251	63.2	61.5	63.1
1510	4494	65.1	64.6	64.9
1528	4172	62.0	61.1	61.7
1540	3904	59.0	58.6	58.5
1555	3438	55.5	55.9	56.3
1612	4504	63.3	64.3	-
1616	4515	-	63.9	-
1625	4744	66.0	67.0	66.0
1635	4876	-	66.2	-
1647	3904	59.3	59.6	59.7
27 June 1969				
0850	Rm Temp	45.9	46.3	46.5

TABLE IV. ULTRASONIC DATA FOR LASL TESTS ON 27 JUNE 1969

Clock Time, Hours 27 June 1969	Port #3 Pyro °R	Sheath #1 µs	Sheath #6 µs	Sheath #7a µs	Sheath #8 µs
1026	Rm Temp	45.9	46.4	46.7	46.8
1236	3351	57.0	55.5	56.8	56.5
1240	3366	56.0	55.4	56.5	56.5
1250	3840	60.9	58.3	61.3	59.8
1300	4108	63.7	60.6	63.0	62.4
1310	4467	66.7	63.5	65.8	65.5
1320	4591	67.1	64.8	66.2	66.7
1330	4707	67.3	65.5	68.0	65.6
1345	4717	66.1	66.2	69.2	66.0
1400	-	66.0	66.1	68.9	66.0-67.0
1410	4623	65.7	65.5	67.4	66.5
1425	4494	65.0	63.8	65.1	65.1
1436	4184	62.3	60.6	62.4	62.3
1446	3941	60.0	59.7	59.8	60.0
1455	3532	56.0	56.6	56.5	56.5

		Pulse	Width	Pulse	Width	Pulse	Width	Pulse	Width
		Min	Opt	Min	Opt	Min	Opt	Min	Opt
1518	2917	53.5	53.7	54.2	54.3	53.9	54.2	54.1	54.2
1528	4082	60.5	61.2	60.7	60.8	61.2	62.0	60.3	62.0
1538	4487	64.9	65.3	64.0	64.7	65.2	66.2	65.2	66.1
1547	4080	61.0	61.2	60.6	61.0	61.4	62.2	60.4	62.0
1558	4484	65.0	65.1	63.1	64.4	64.5	65.9	65.0	65.8
1617	4712	65.8	66.5	64.3	64.9	66.9	67.5	65.8	65.3
1625	4936	67.0	67.7	66.3	66.3	-	64.8	69.2	69.8
1635	5042	67.3	68.1	66.1	65.9	-	65.7	67.8	68.4
1640	5235	67.7	68.4	-	64.3	-	68.2	-	66.9
1651	5332	69.2	69.5	70.2	70.9	-	-	-	70.1
1655	5405	-	69.5	-	-	-	-	-	

Regarding the first troublesome point above, we note from Table III that room temperature data were essentially reproducible (46.4, 46.3 μsec) for Sheath #6, in which an annealed W-Re line was tested. Also encouraging, at elevated temperature, although drift was still evident, Sheath #6 showed less drift than either #3 or #7. This suggests that after sufficient annealing, much of the drift may be taken care of. (Recent, independent tests on Re reproducibility in AEC work, Contract AT(30-1)-3906, reported in NYO-3906-8, lends some credence to this view.) Cleaning the Ta sheaths with much care, however, did not provide any apparent benefit.*

Regarding the second troublesome point, we note from Table III as well as from the oscillograms that Sheath #3, whose W lead-in was not spirally isolated, quickly lost signals on heating. All three lines used isolation for the Re sensor, but only #6 and #7 had the lead-in isolated too.

As the spurious noise pulse is so evident in many of these oscillograms, the question arises, how come it was not always observed in earlier tests? The following explanation is offered. In the WANL test of June 1967, the installation was vertical. Likewise, tests at Panametrics prior to the preliminary LASL tests of January 1969 were also vertical. Assuming the noise is due to diffusion bonding of lead-in to sheath, vertical installations do not provide enough pressure or intimate contact to produce an echo-yielding bond. What about horizontal tests as in Figs. 4 and 5? Here we tested only one sheath at a time. Therefore the line was continuously agitated ultrasonically, preventing a bond. In the absence of substantial pressure contact, then, we apparently maintained an "ultrasonic unwelding" situation. This is exemplified by several multiple-exposure oscillograms, for example, Fig. 21-Run 5-3, 4, 5. In Fig. 21-Run 5-5, for example, at ~4250°R, the upper trace shows the diffusion noise echo. The middle trace is an erase, wherein ultrasonic pulse width is increased to "shake loose" the joint. The lower trace shows the Re signals which can be read despite the reappearing but smaller noise pulse preceding them. This behavior was reproducible and became apparent in switching from one line to another. Lines which were not interrogated tended to fuse to their sheath. But they could be freed with a strong erase pulse, that is, a pulse which tends to erase the noise preceding Re sensor echoes. (In subsequent and independent tests, Fam (Ref. 3) found that lines apparently "stuck" in their sheaths could be freed by using a high repetition rate (~1 kHz) even at normal pulse width.)

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*Although cleaning the Ta sheath did not eliminate drift, reference to Figs. 19a and b suggests that some carbon was absorbed in the Re sensor, even though carbon did not penetrate the Ta sheath more than ~1/3 of the wall thickness. The source of the carbon found in the Re sensor is not known at present. Whatever its source, however, its presence could account in part for the observed drift.

Diffusion Bonding. Reinforcing this point, observe in Fig. 21-Run 5-3, at ~3950°R, how for Sheath #3 a strong pulse applied after the upper trace was recorded, essentially eliminated the noise pulse. Again, in the next oscillogram (Fig. 21-Run 5-4) for the same sheath at ~4000°R, 24 minutes later, the four traces record the following sequence: first, W-Ta diffusion joint forming in lead-in; second, echoes observed on returning to this line, after it had been left idle for ≲ one minute (note growth of noise pulse at expense of sensor echoes); third, erase pulse is applied, to unbond lead-in; fourth, diffusion echo is small enough not to prevent sensor echo identification. This suggests a measuring sequence where an "erase" pulse would be applied to free or break any possible bonds, followed by the usual "read" interrogation pulse.

Most of the ultrasonic data was obtained using a minimum pulse width, ~10 μsec. In some cases, however, it was observed that the signal-to-noise ratio could be improved by slightly increasing pulse width, as illustrated for Sheath #6 at 4876°R (Fig. 21-Run 5-7). Changing the pulse width from "minimum" to "optimum" changed the measured transit time by as much as 1 μsec in some cases (Table IV).[*]

At ~3900°R, (Fig. 21-Run 5-6) both first and second round trips or multiple echoes in a Re sensor for Sheath #6 were selected. The first negative pair, denoted $\overline{AB}$, read 58.8 μsec; the multiple, $\overline{A}B$, read 117.8 μsec, almost exactly double.

Since these tests, Run 5, indicated that Re "improved with age," i.e., soaking appeared to reduce subsequent calibration drift, it was decided to run Sheath #6 again. Also, since one spiral spacer over both Re and the adjacent part of the W lead-in helped, it was thought that a double spiral over these portions might help still more. Accordingly, Sheath #1 was prepared with its line isolated by a double spiral of 0.05 mm dia W wire.

Sheaths #1, 6, 7a and 8 were tested in Run 6 on 27 June 1969 (Table IV). Sheaths #1, 7a and 8 were of equal lengths, and shorter than #6. For each temperature, the oscillogram trace sequence is 1, 6, 7a and 8 (except at 4936°R, where the trace for #7a is not available).

All four sheaths employed spiral isolation over Re and part of the W. Sheath #1 had a *double* spiral. Significantly, this sheath provided the *clearest* echo signals at 5332°R, and further, provided the *only* readable signals (69.5 μsec) at 5405°R. However, even these echoes were attenuated (perhaps due to bonding) before an oscillogram could be obtained. Because of time limitations Run 6 had to be terminated here.

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[*] The transit time observed in this test depended somewhat on electrical parameters such as pulse shape, amplitude and signal/noise ratio. It may be that the interaction of sensor, isolator and sheath affected these electrical parameters enough to contribute to the apparent "drift" in calibration.

All sheaths were removed from the mock fuel element. A portion of #6 (Ta sheath and Re sensor) were submitted to Dr. C. P. Kempter of LASL for metallurgical analysis. Figure 19 is a photomicrograph showing that in the sheath region of greatest carbon attack, at a sight port location, TaC penetration was only 0. 16 mm (0. 0068 inches). Preliminary x-ray analysis of the Re sensor using soft x-rays indicated a surface coating of W and W-Re alloys. Since the 0. 05 mm dia W spiral spacer wires maintained their integrity, it seems that not much W could have been transported to the Re surface. Nevertheless, W transport, a source of drift in thermocouples at these temperatures, presently must be considered as a possible source of ultrasonic calibration drift. Perhaps the interactions of three different metals (W lead-in and spiral; Ta sheath; Re sensor) at high temperatures unavoidably result in drift during one-hour runs. This suggests using one material for lead-in, sensor and sheath, if possible. Along this track, W is most promising from the standpoint of attenuation and hydriding. To date, however, W has exhibited extreme hysteresis in our cyclic tests to 5300°R. Ta suffers from attenuation and, potentially, hydriding. Re is eutectic-limited to 4966°R.

Since the instrumentation hole itself is lined with NbC to minimize erosion, it is natural to inquire into the possible use of NbC as a sensor. Data presently available, based on ultrasonic resonance experiments conducted at LASL, are encouraging. Figure 20 shows the reproducibility observed in several cycles to ~5000°R for $NbC_{0.969}$ (Ref. 12). Dr. Kempter suggests that TaC is also promising, but cautions that these compounds lose carbon preferentially at high temperatures unless a suitable inert gas pressure is maintained.

Returning to the Re data, it is to be appreciated that some of the drift is due to annealing. Sensors used in these tests were not previously annealed, to avoid the risk of fracture to the recrystallized Re. However, as the carbided sheaths and recrystallized W and Re lines survived removal from the furnace, and shipment back to Panametrics, it is now felt that pre-annealing is not an unreasonable procedure.

The data show that the line which was doubly isolated briefly survived to a temperature of 5400°R. This appears to be the highest temperature at which a sensor has survived and still had readable ultrasonic signals in the particular simulated environment in question. Furthermore, a number of lines, including one bare line, survived over one hour above 4000°R.

Conclusions and recommendations, based on the above observations, are presented next.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1) Attenuation at high temperature was overcome. Using Ta sheaths, W lead-in lines and Re sensors developed in this program, clear signals were obtained in the final field tests at LASL beyond the contractual goal of 5300°R. Oscillograms were obtained up to 5332°R, and an automatic reading of transit time in the Re sensor was obtained at 5405°R. This was accomplished using a 10 μsec pulse width to overcome attenuation in the ~1 m long W lead-in and the ~100 mm long Re sensor.

2) A Ta sheath survived a ~1-hour soak at ~5000°R in a graphite/hydrogen atmosphere at LASL, in preliminary tests, January 1969.

3) Another Ta sheath survived numerous cycles from ~500 to nearly 5000°R in a graphite/helium environment at LASL, including over two hours accumulated exposure above 4000°R, and a rapid drop from ~5000°R (due to power interruption) prior to brief use to 5332°R.

4) Calibration drift was observed and still needs to be explained and/or eliminated before the present system can be used reliably up to ~5300°R in the graphite/hydrogen/nuclear environment. A small part of this drift includes annealing. The effects of contaminants, the possible use of materials of higher purity, use of a getter, use of the same material for sensor, spiral isolator and sheath, etc., remain to be considered in greater detail.

5) Uncertainties relative to the January 1969 LASL tests, concerning diffusion bonding of the Re sensor to the sheath, horizontal vs vertical installations, interference between two closely spaced transducer coils and effect of seal fitting, were clarified by a series of controlled experiments in June 1969 at LASL. These problem areas were substantially avoided using simpler, isolated lines in the latter tests, including single T/R pulse-echo coils mounted within the sheath.

6) Multiple echoes in the Re sensor were observed beyond 4700°R. This permits temperature sensitivity to be magnified by the number of reverberations. Thus, sensitivity of 10°R was achieved in a 100 mm Re sensor.

7) The optimum design for an ultrasonic line depends on the maximum temperature, the temperature profile, and cyclic behavior. Designs for 4000, 4500, 5000, and 5300°R may differ substantially.

Recommendations

As this program ends, the main unanswered question involves the calibration drift.

To the extent that interest continues for reliable 5300°R temperature sensors for a graphite/hydrogen/nuclear environment, it is recommended that this drift be explained and/or eliminated, based on further controlled experiments and analysis. Additionally, transducers and lines should be tested in a simulated nuclear rocket engine environment at Jackass Flats alongside thermocouples.

Further, higher purity Re, and other new candidate sensor materials (e. g., NbC, TaC, pyrolytic graphite, vitreous carbon, possibly W) should be investigated.

Now that attenuation has been overcome, the possibility of maintaining adequate temperature sensitivity in a short sensor, by means of multiple echo, pulse superposition and/or resonance techniques, should be explored.

In Phase I, measurement of two adjacent temperatures on one line was demonstrated. Since that time, under another contract, AT(30-1)-3906, measurements of four adjacent zones have been demonstrated, with potential applications to LMFBR or FFTF fuel pin profiling. These ultrasonic profiling concepts should be extended to other applications, where the number of lead lines is limited. Even ten or more regions can now be measured with only a single lead line (Ref. 13).

The use of pulse pairs (long and short, or torsional and extensional modes, etc.) to avoid diffusion bonding of lines within sheaths could be explored.

Optimizing a line for use at the maximum temperature where sheaths are not required should be fruitful in reducing costs. As a corollary, the maximum time/temperature exposures for lines protected only near the sensor by short sheaths should be determined, also with a view toward reducing costs, consistent with reliability and accuracy.

Applications of thin sensors, 0.25 down to 0.025 mm (0.010 down to 0.001 inch) diameters, should be considered.

Some of these areas for future materials and acoustical developments are not restricted to the nuclear rocket engine environment, and should be considered in the light of current or anticipated applications.

ACKNOWLEDGMENT

The authors gratefully acknowledge the cooperation of the Los Alamos Scientific Laboratory for making their excellent test facilities and personnel available for the preliminary and final field tests conducted in January and June 1969. Particular thanks are due Dr. Joseph L. Perry, Jr. (N-7), Dr. John C. Rowley (N-4), and Dr. C. P. Kempter (N-1). C. R. Tallman, B. Goodier, O. R. Norris, and R. Renfro assisted with the tests. Without their help the tests could not have been accomplished. C. R. Saunders provided much of the instrumentation. Dr. M. S. McDonough, now with Sylvania, Waltham, Massachusetts, participated in the preliminary tests at LASL, and the Ta hydriding experimented. He has contributed substantially to the Phase I and II efforts in this program. The continuing encouragement and technical guidance of M. O. Dustin and H. Heppler of NASA-Lewis Research Center is sincerely appreciated.

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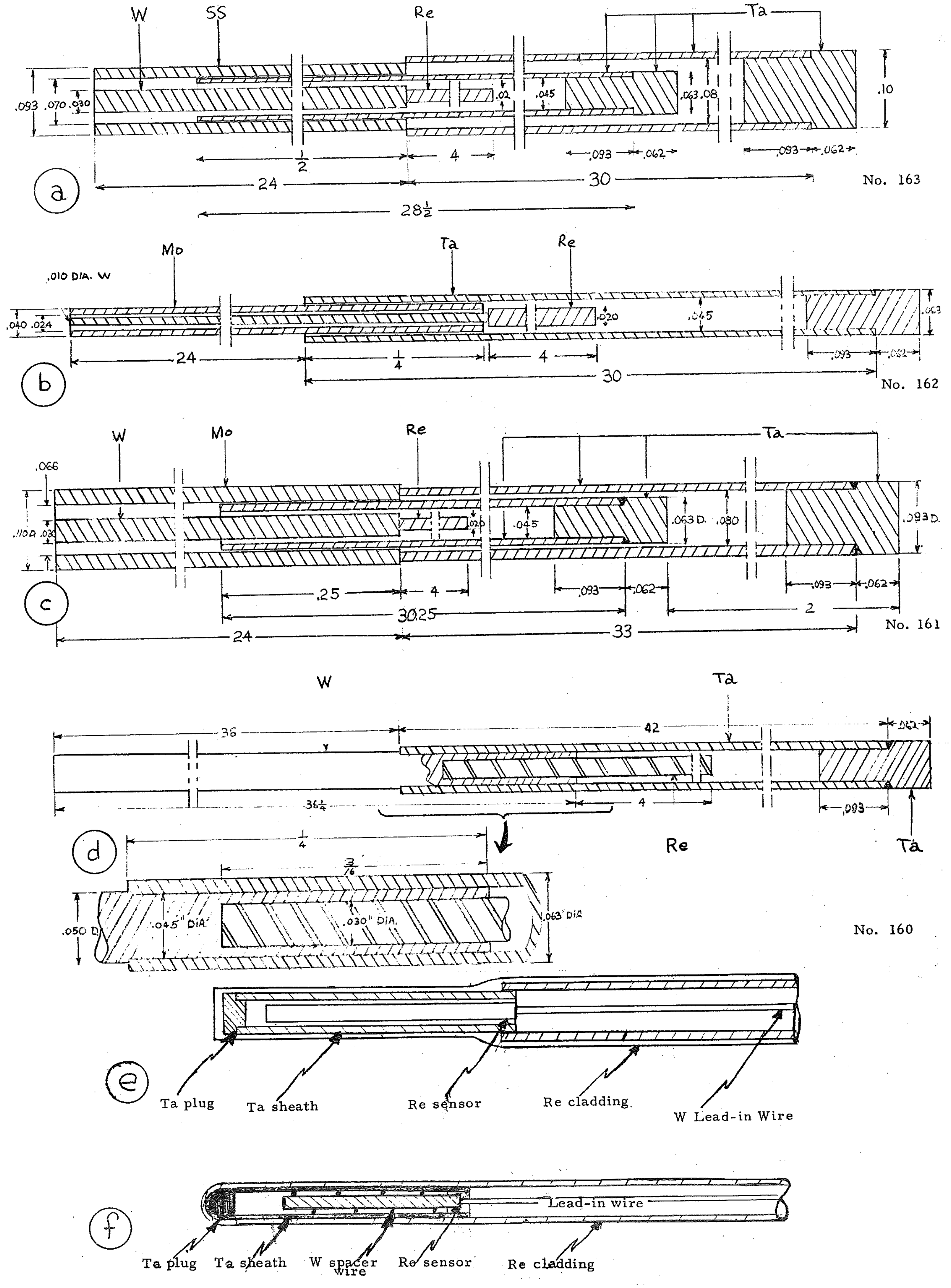


Fig. 1. Design of sheath systems intended to operate up to 5300°R for one hour in a carbohydrogen environment.

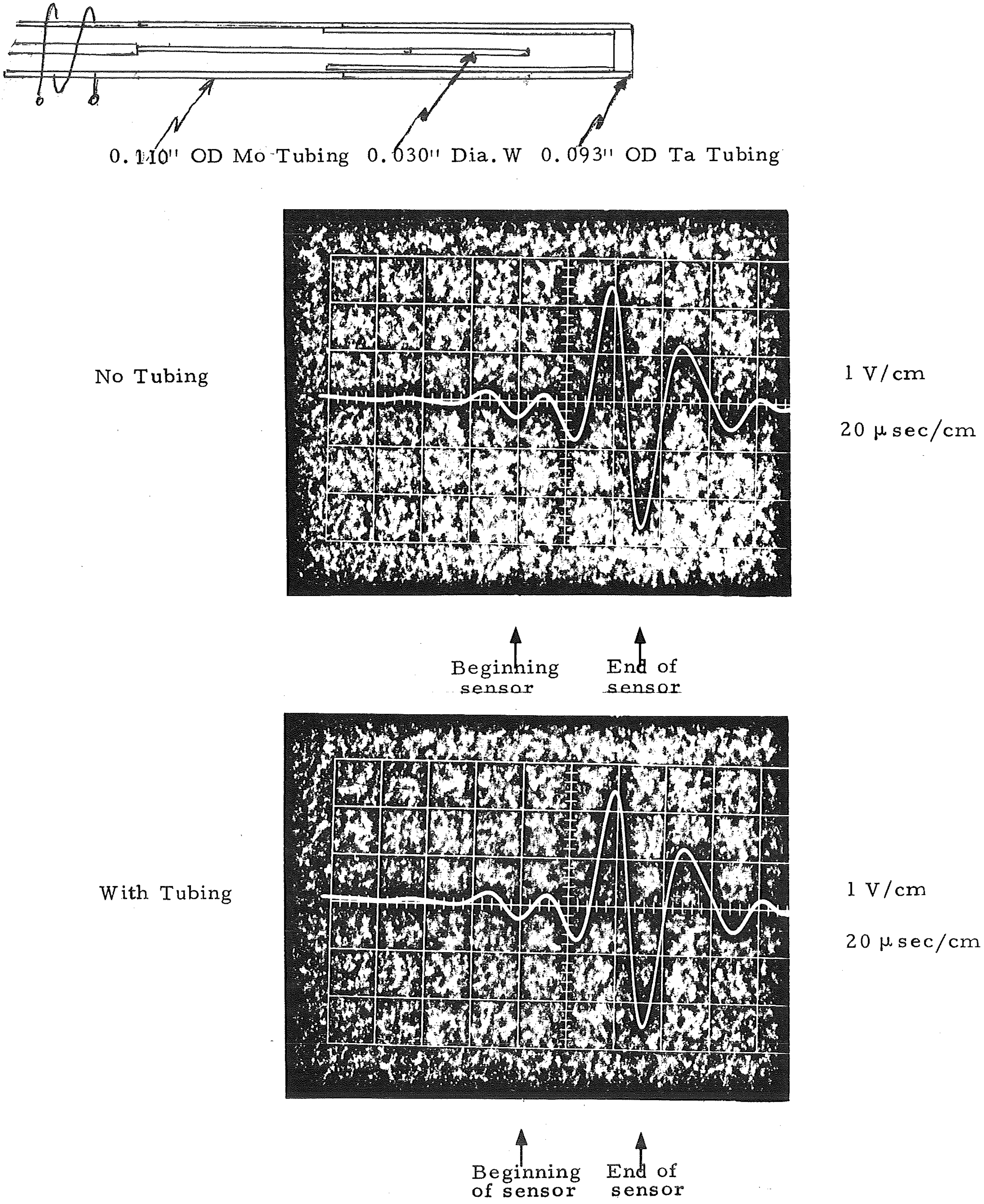


Fig. 2. Sensor echoes before and after installation in sheath

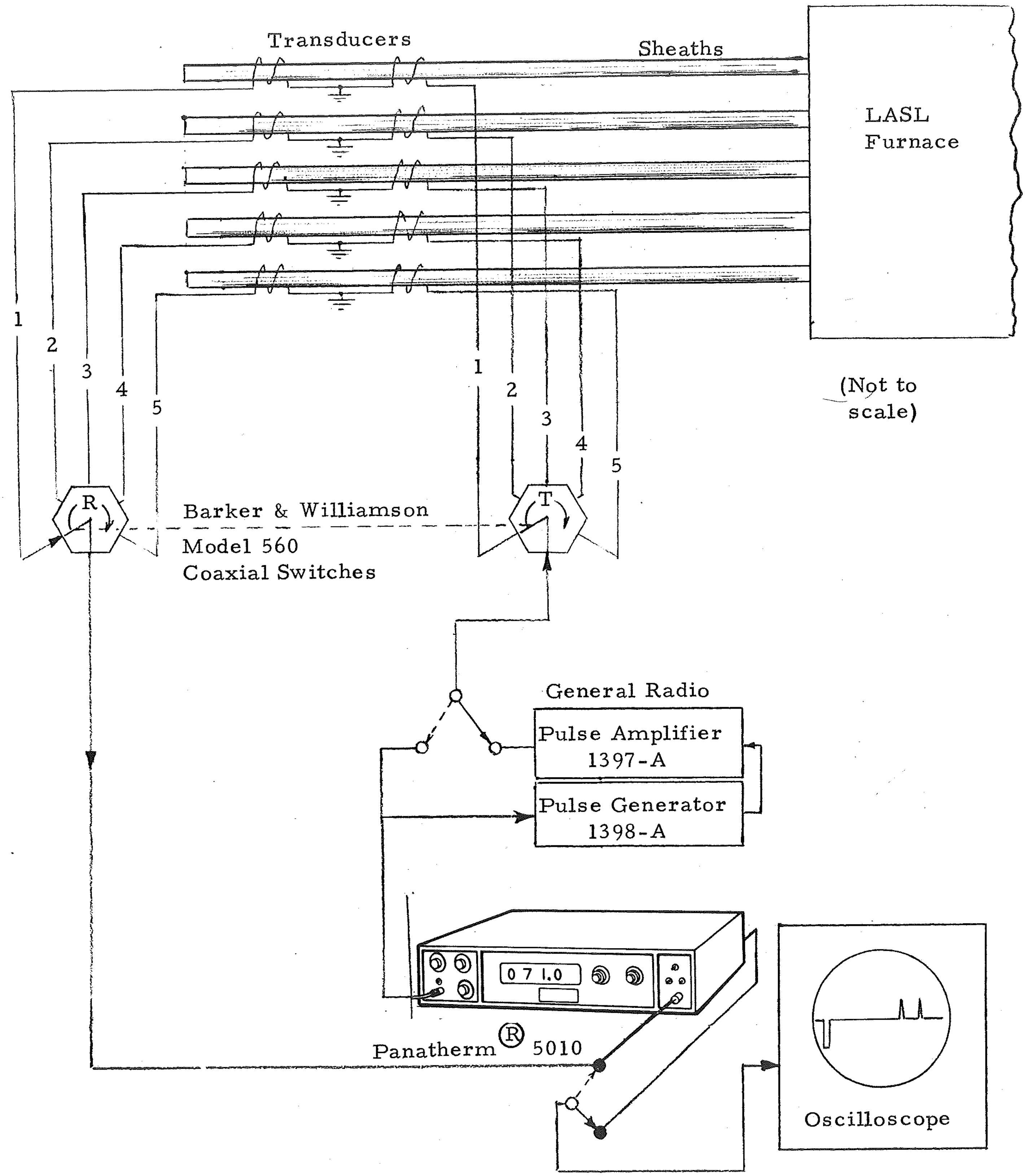


Fig. 3. Block diagram of preliminary LASL field test, January 1969

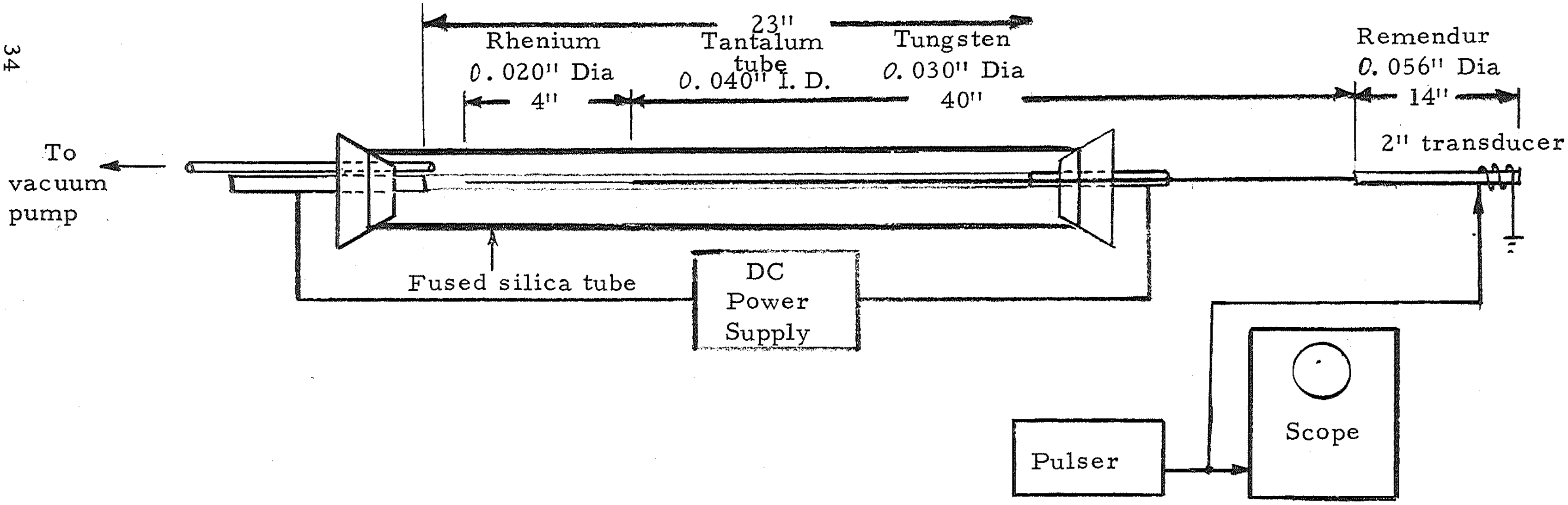


Fig. 4. Schematic of self-heated sheath furnace (horizontal position)

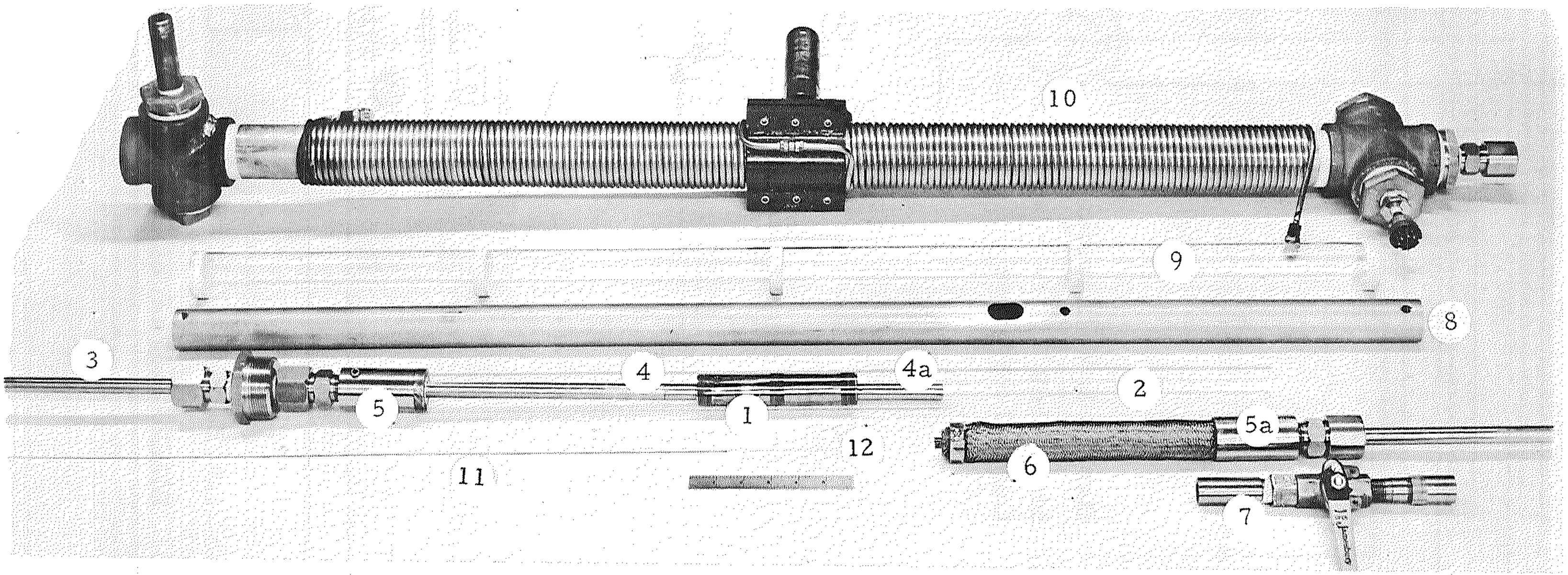
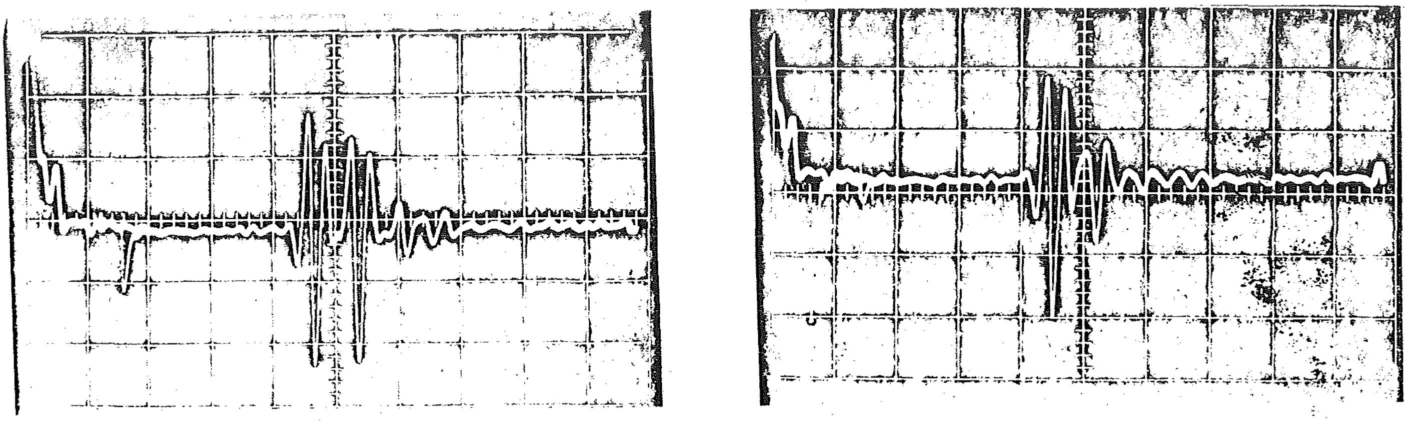


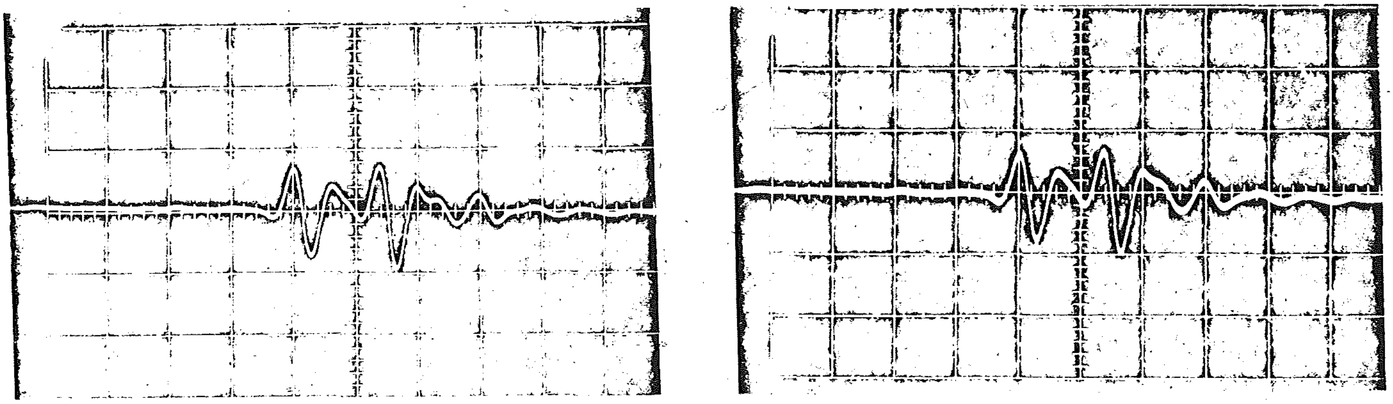
Fig. 5. Self-heated sheath furnace, exploded view



0.2V/cm 100 μs/cm
5300°R - 8 min

0.2V/cm 100 μs/cm
5300°R - 11 min

Fig. 6. Oscillograms from sensor inside horizontal 5300°R sheath, without spiral isolator. Note sudden attenuation and distortion of end echo between 8 and 11 min at 5300°R.



0.2V/cm 50 μs/cm
5300°R - 0 hr

0.2V/cm 50 μs/cm
5300°R - 1 hr

Fig. 7. Oscillograms from sensor inside horizontal 5300°R sheath, with spiral isolator. Spiral wire on sensor prevents fusing of the sensor to sheath. After one hour at 5300°R signals remain virtually unchanged.

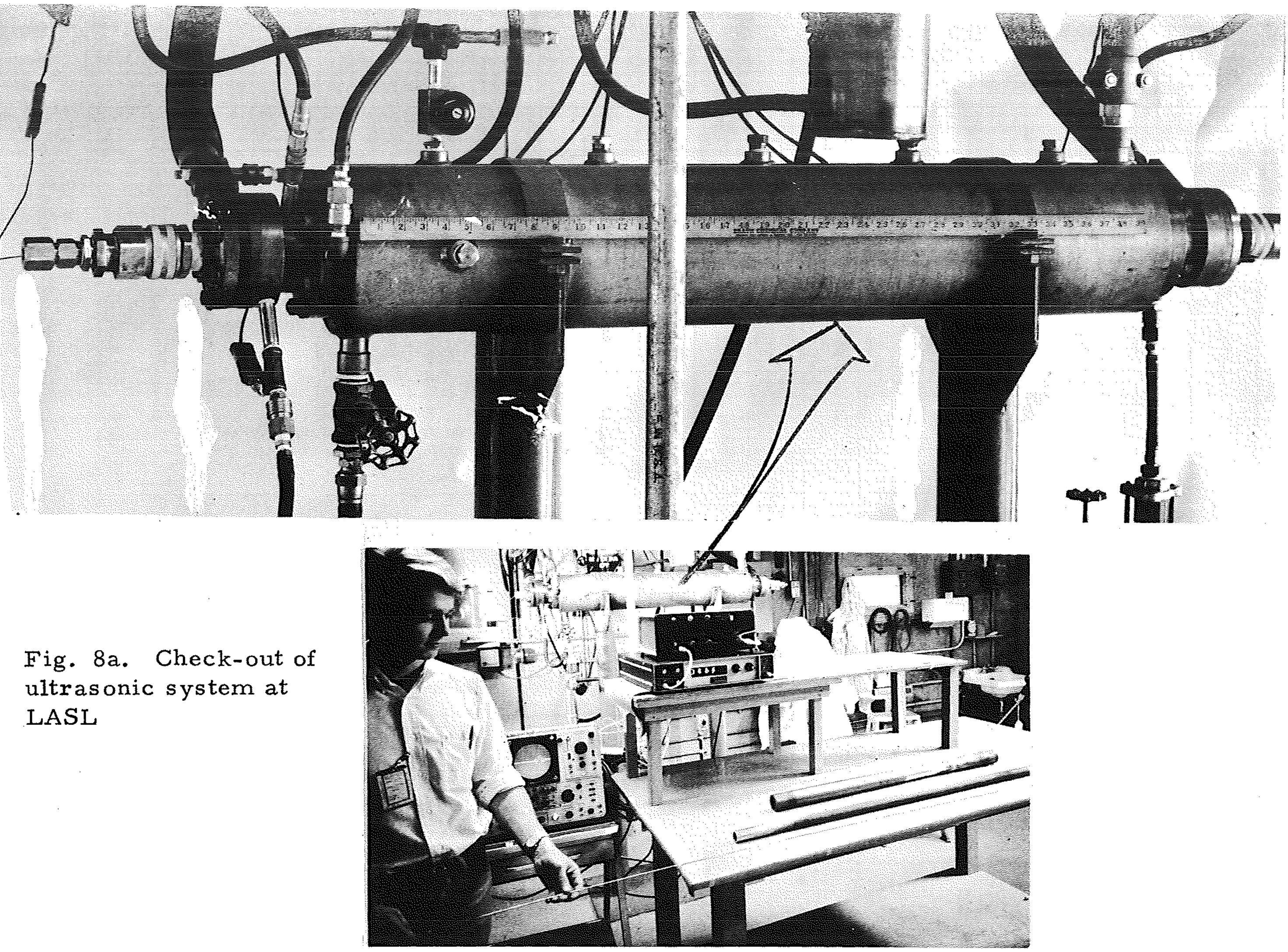


Fig. 8a. Check-out of ultrasonic system at LASL

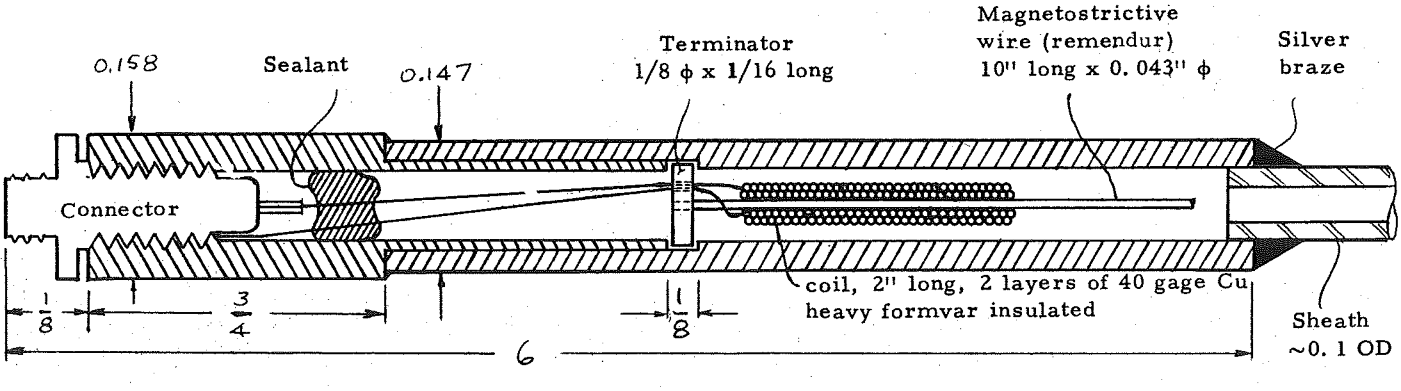


Fig. 8b. Transducer assembly used in LASL tests, June 1969. (Not to scale).

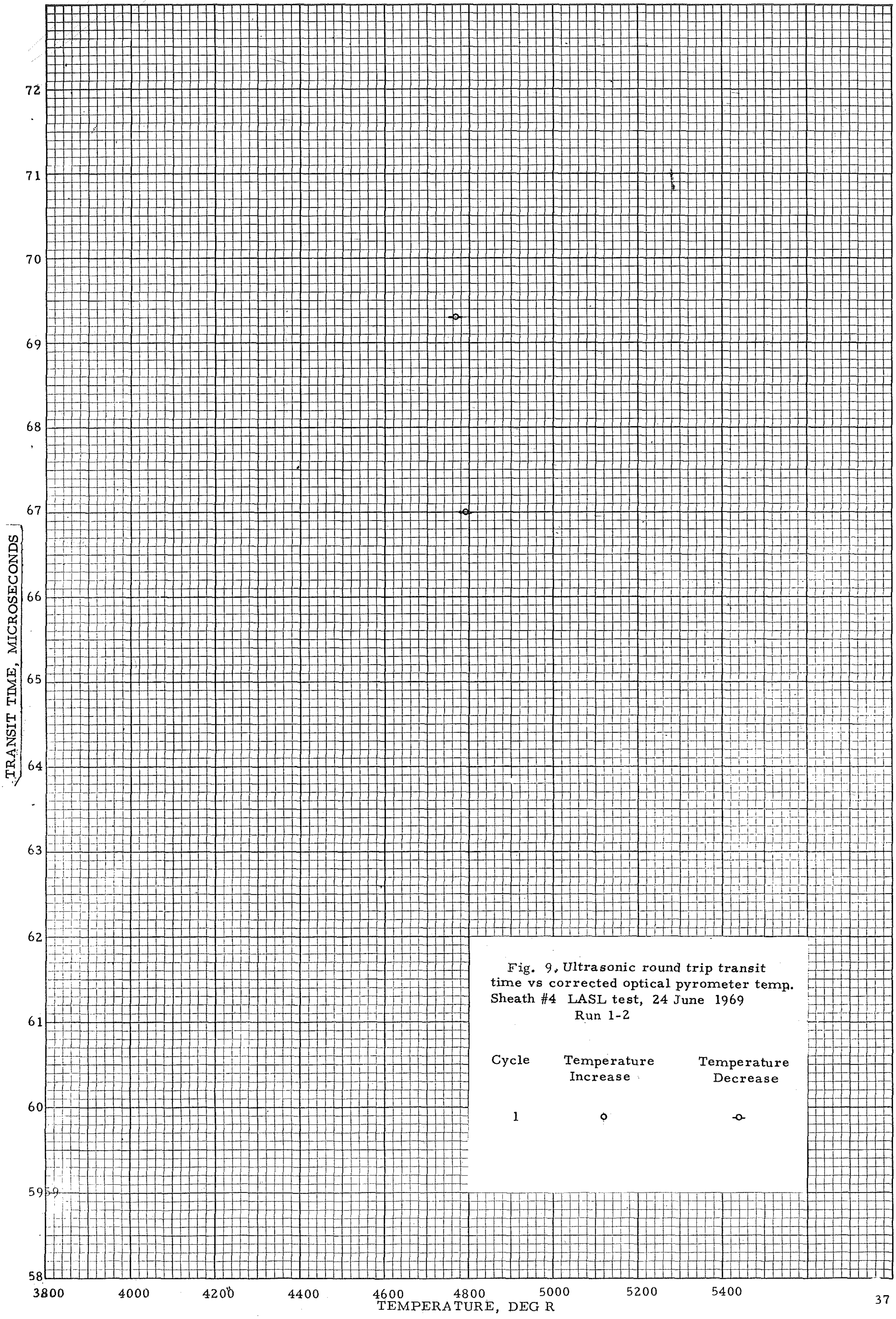


Fig. 9, Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #4 LASL test, 24 June 1969 Run 1-2

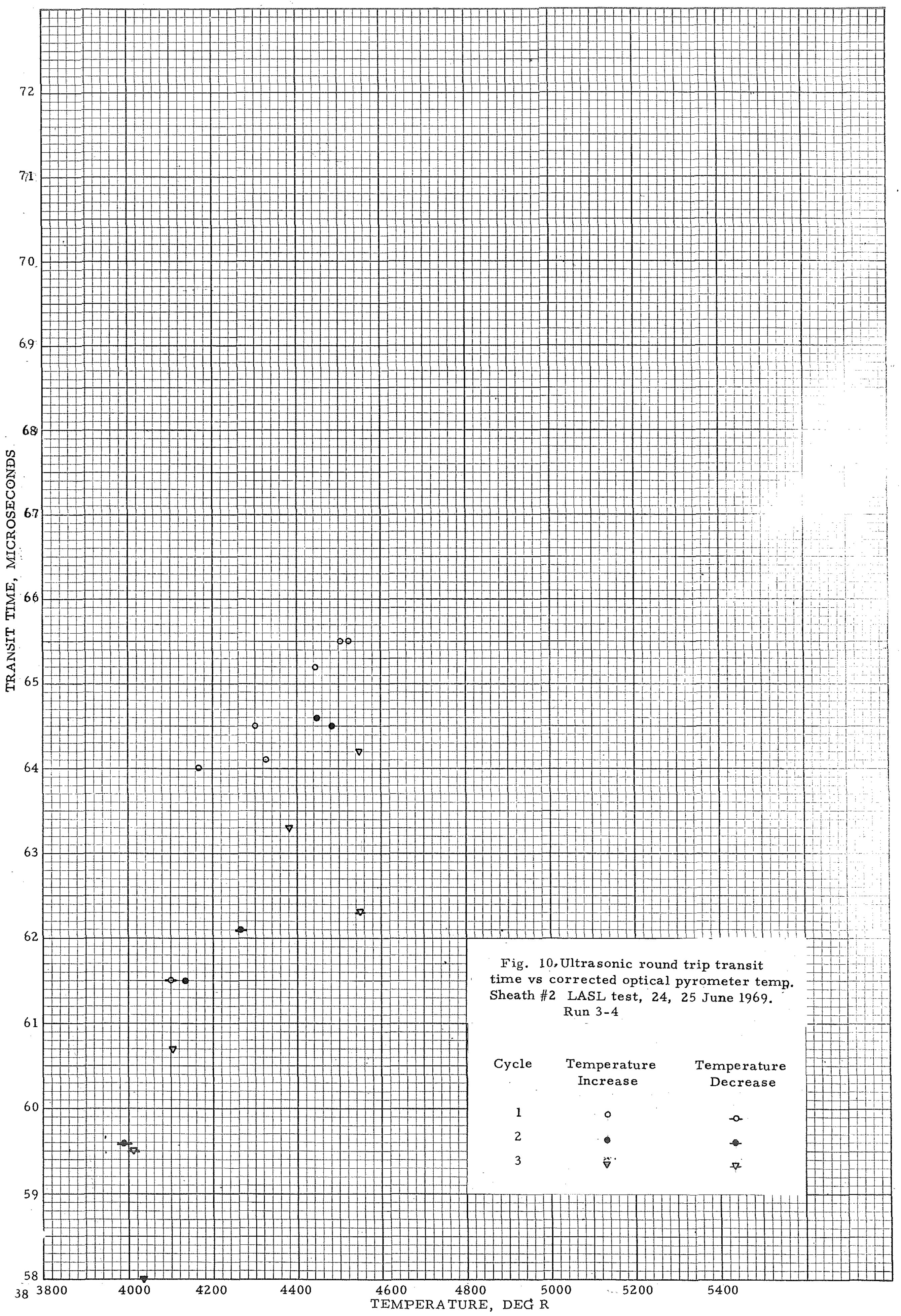


Fig. 10. Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #2 LASL test, 24, 25 June 1969. Run 3-4

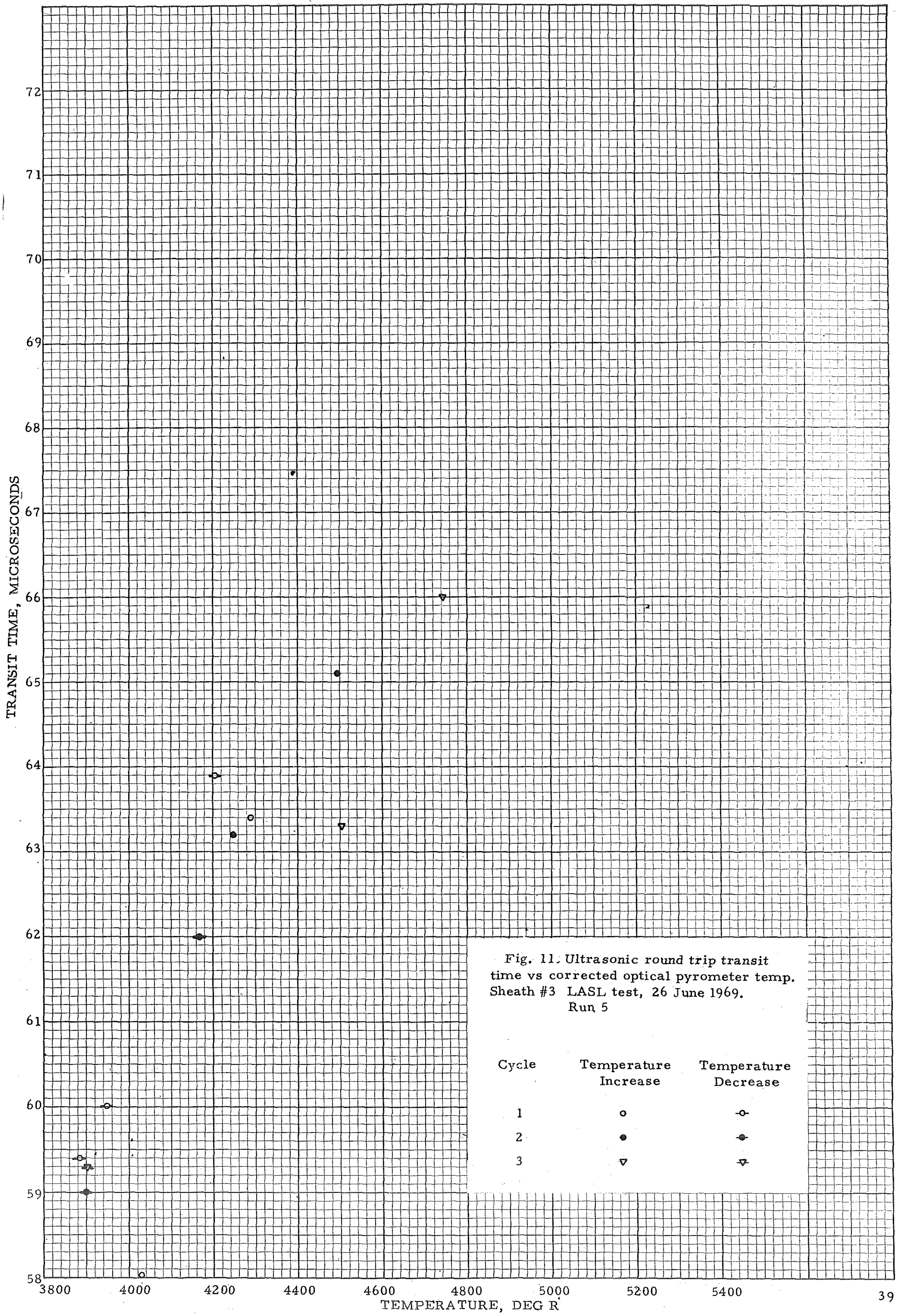


Fig. 11. Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #3 LASL test, 26 June 1969. Run 5

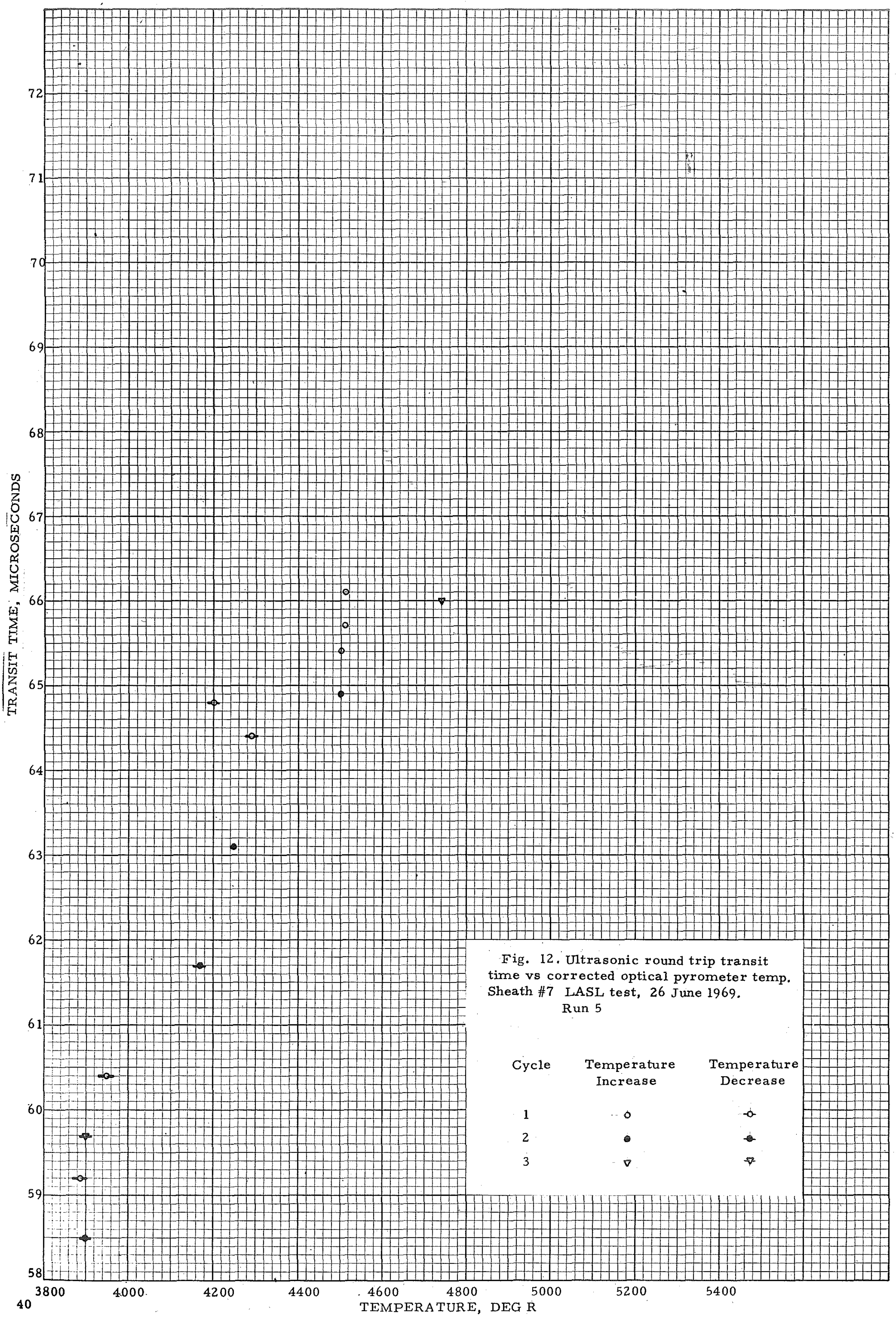


Fig. 12. Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #7 LASL test, 26 June 1969.
Run 5

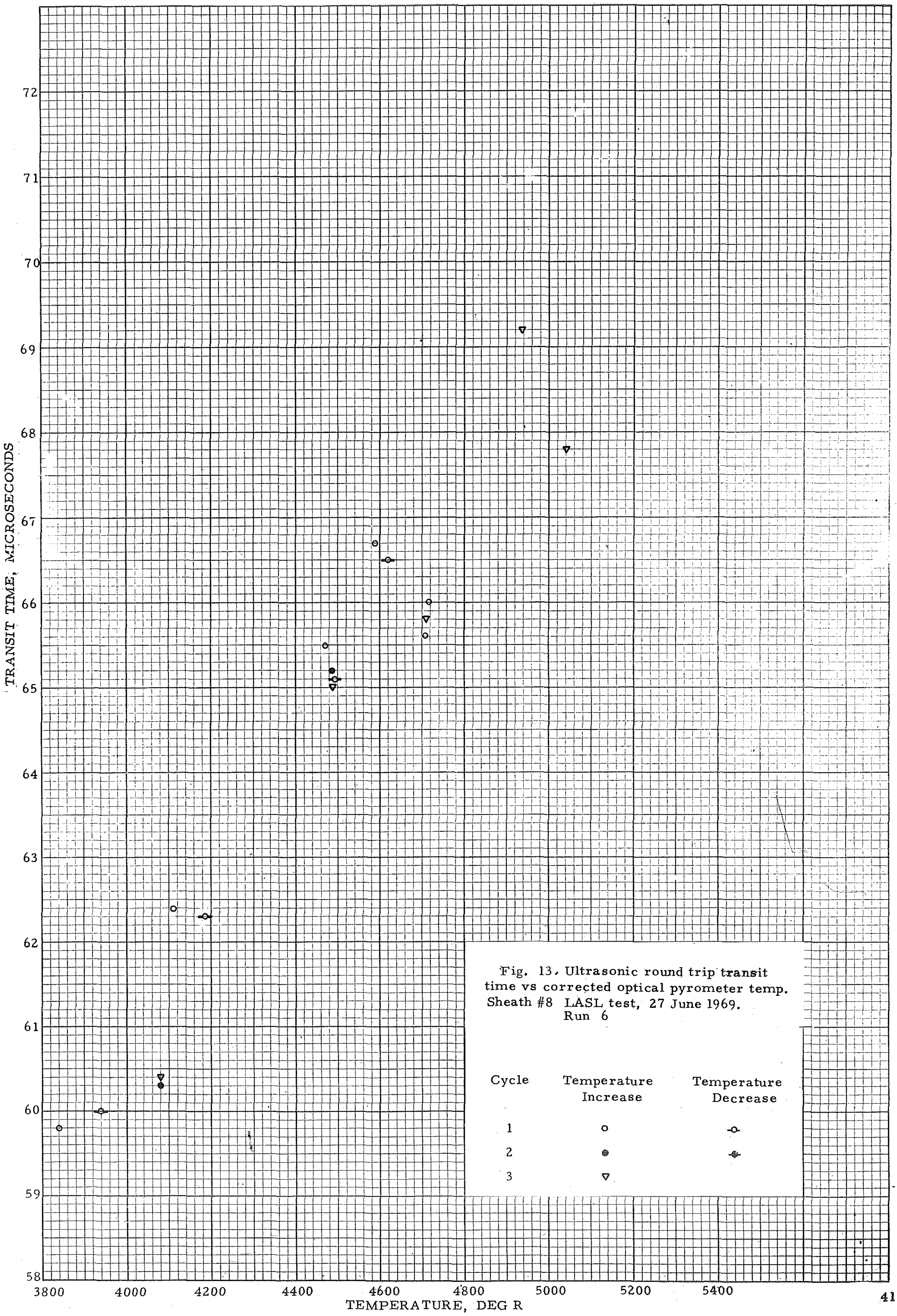


Fig. 13. Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #8 LASL test, 27 June 1969. Run 6

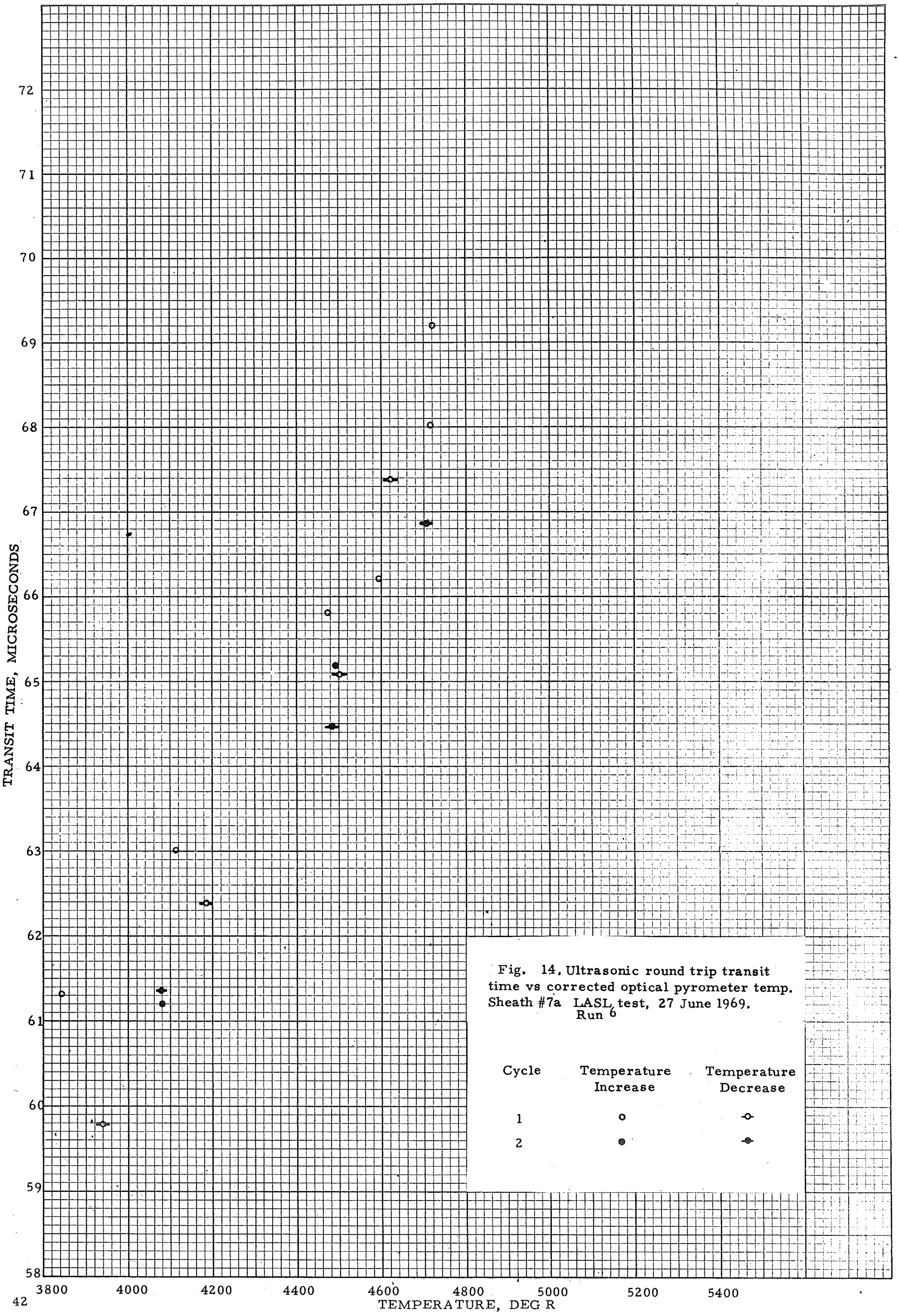


Fig. 14. Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #7a LASL test, 27 June 1969. Run 6

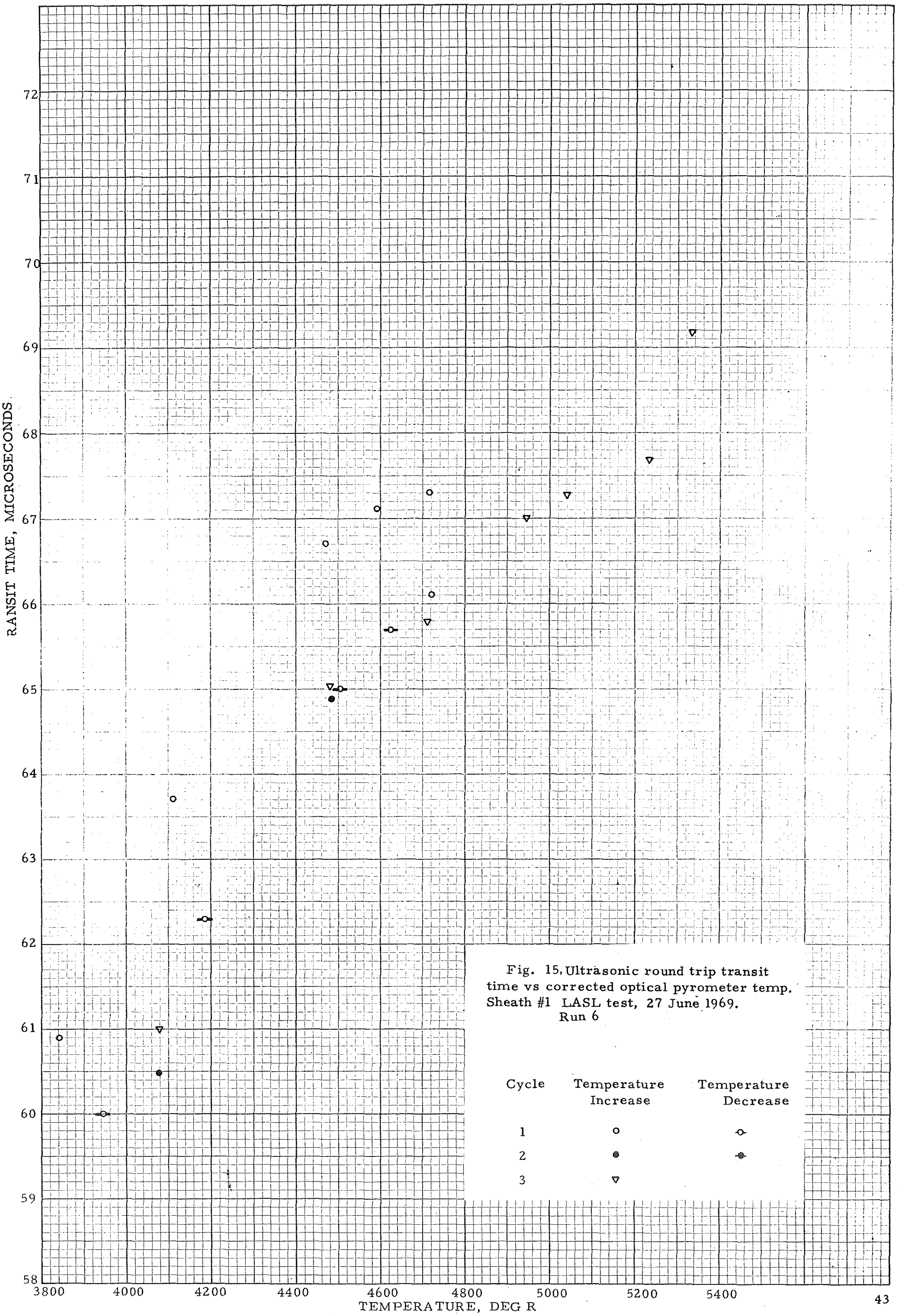


Fig. 15, Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #1 LASL test, 27 June 1969. Run 6

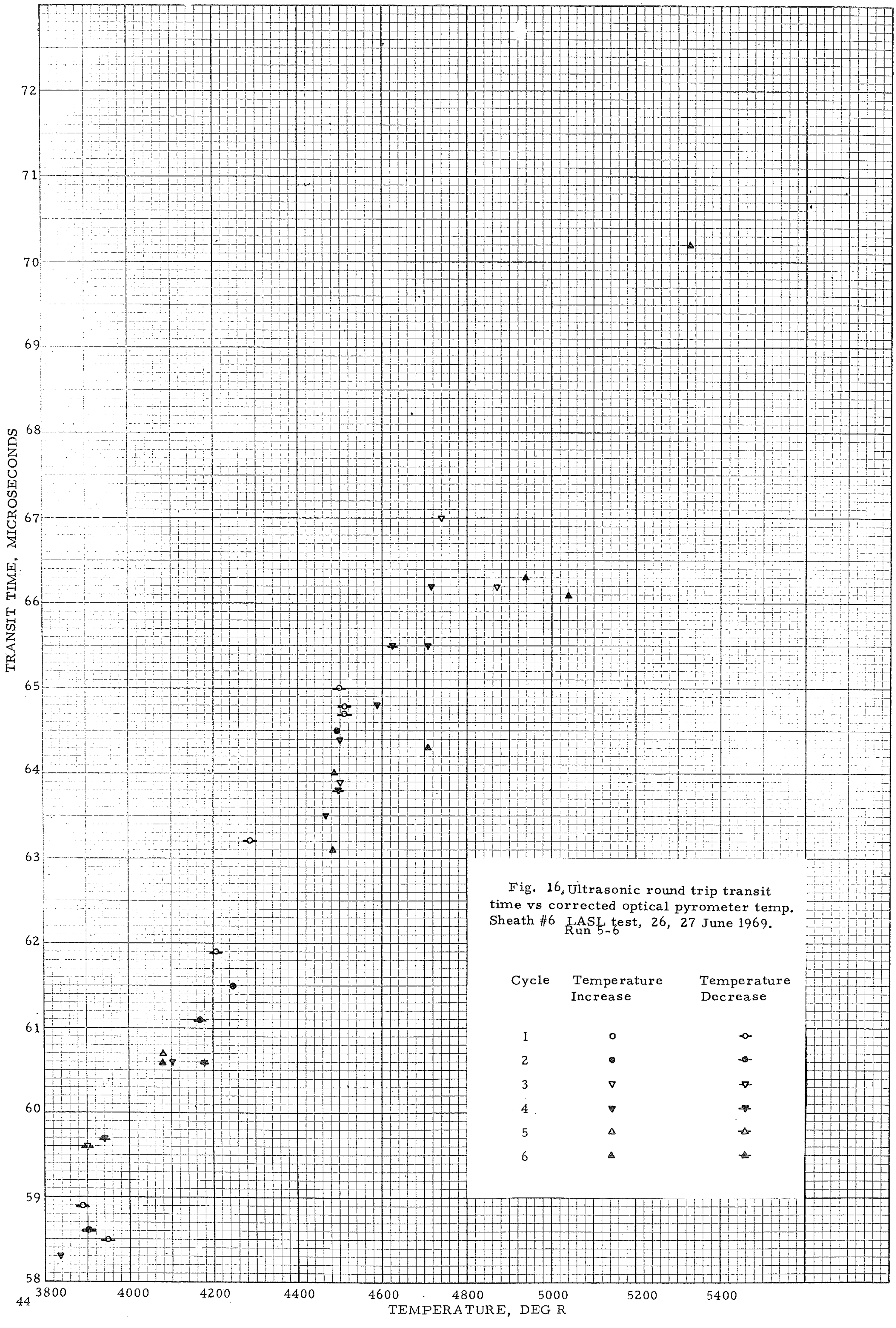


Fig. 16, Ultrasonic round trip transit time vs corrected optical pyrometer temp. Sheath #6 LASL test, 26, 27 June 1969. Run 5-6

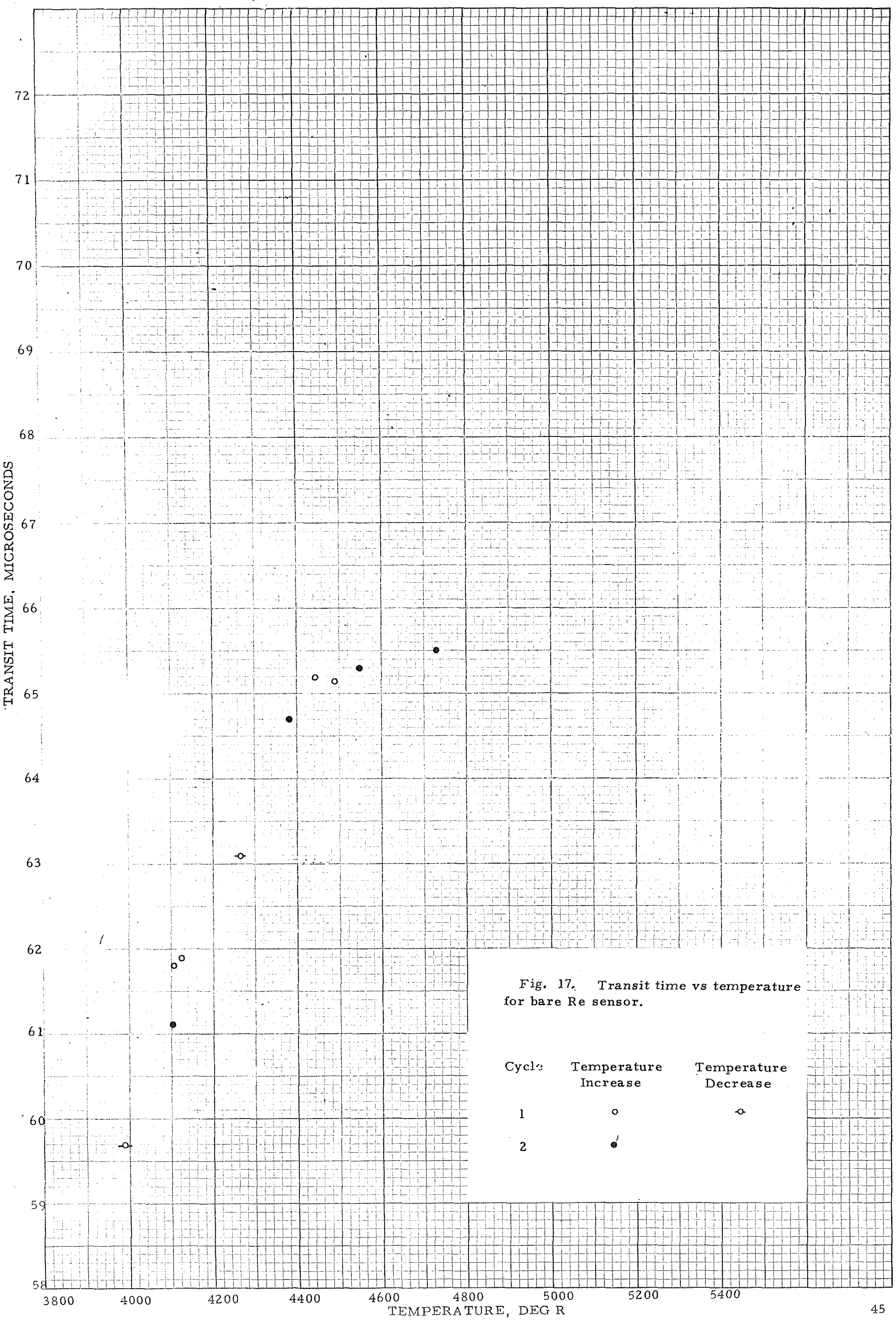


Fig. 17. *Transit time vs temperature* for bare Re sensor.

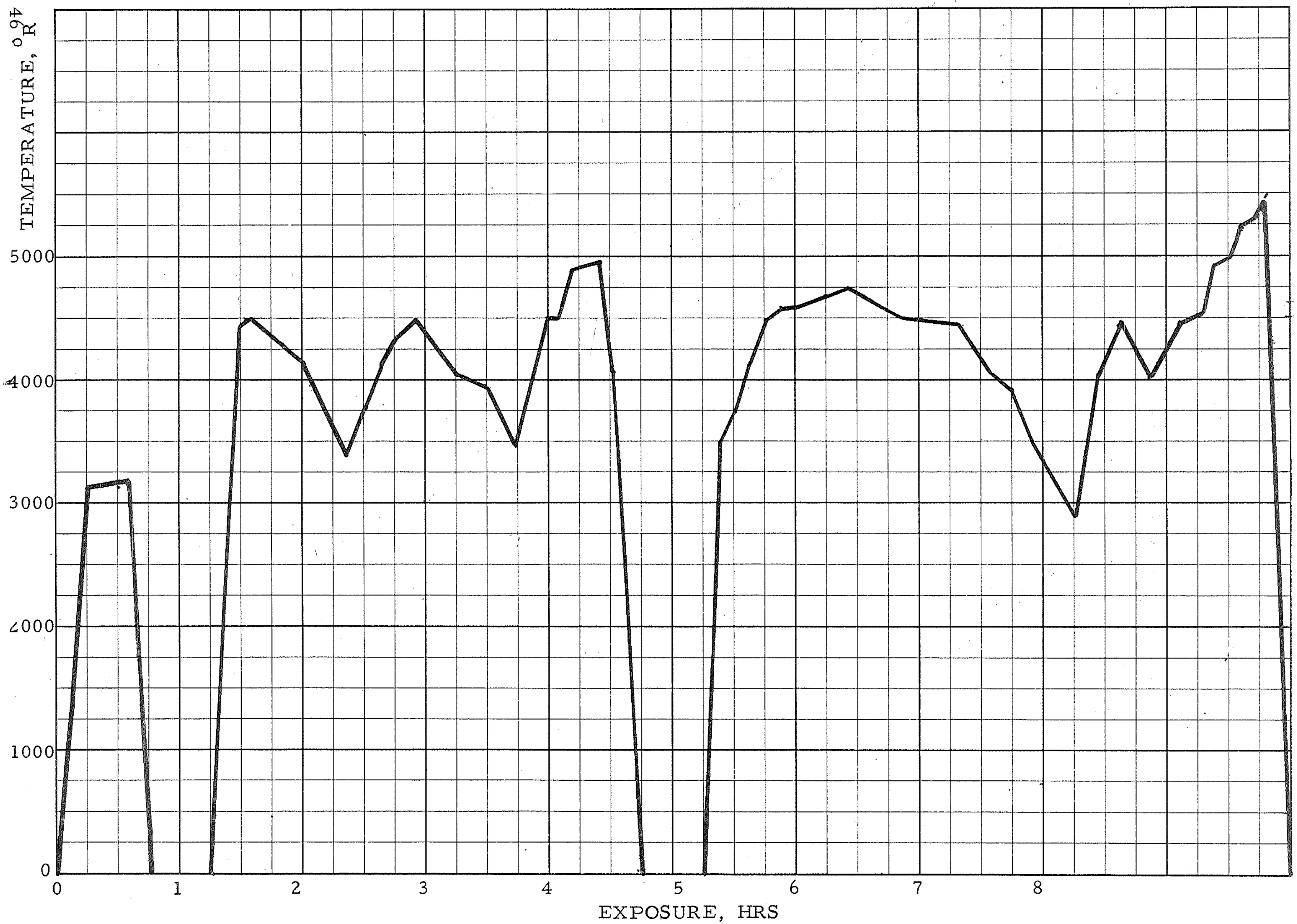
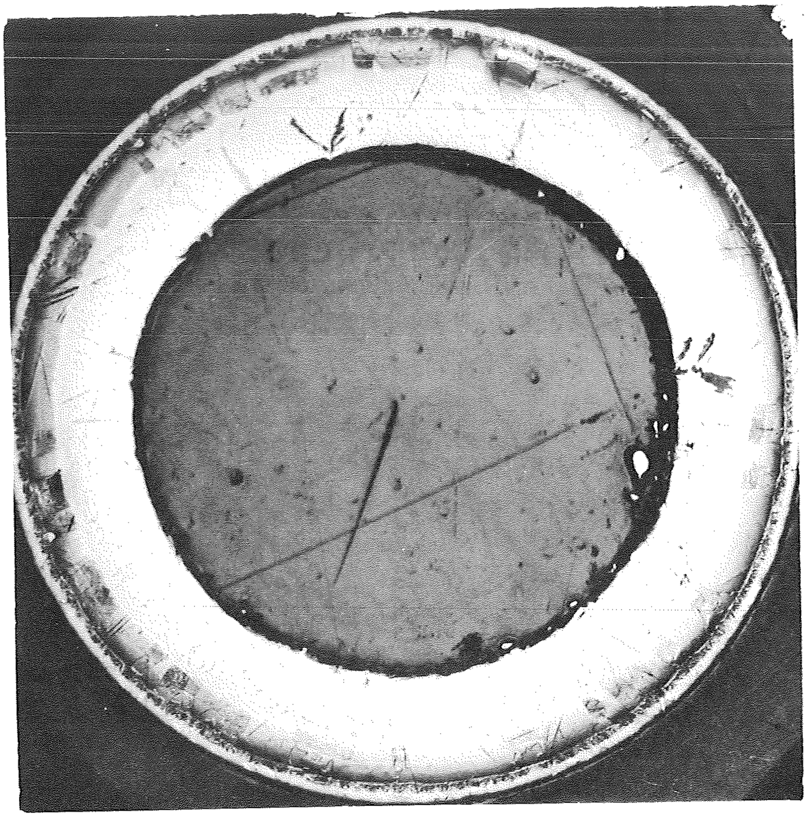
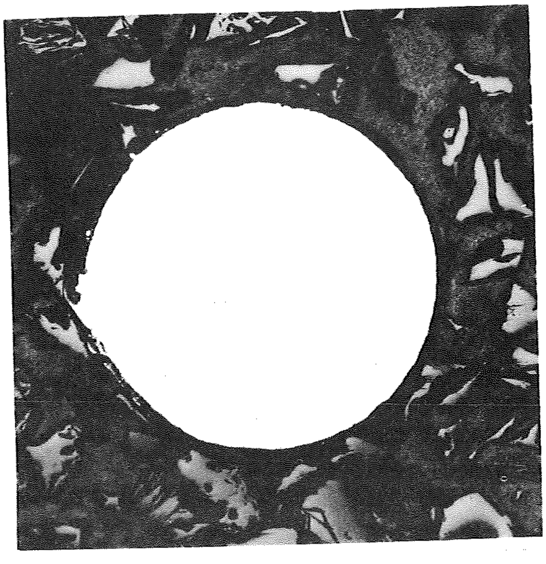


Fig. 18. Exposure history of Sheath #6, in LASL tests of June 1969



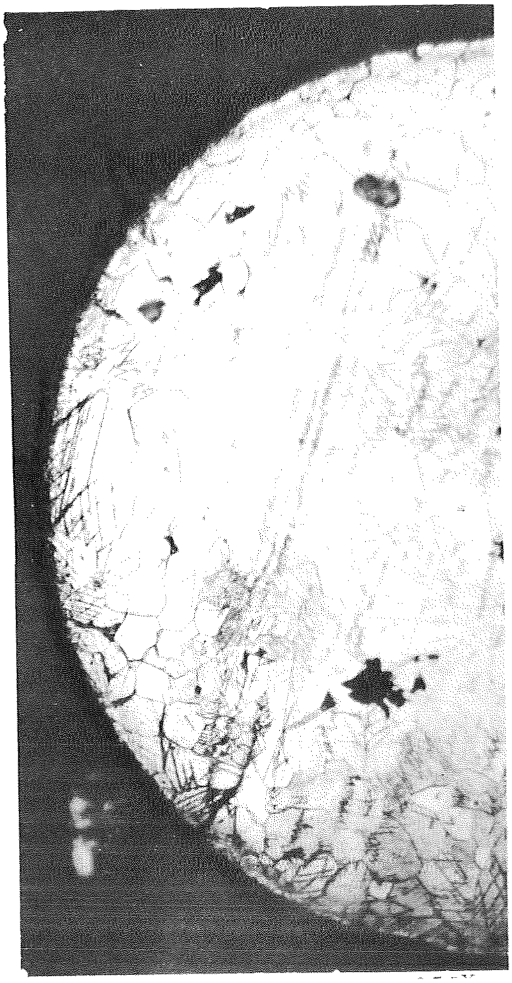
585-1-1 ~ 50x

Fig. 19a. Photomicrograph showing carbon penetration into tantalum Sheath #6, to a depth of 0.17 mm (0.0068 in.), or about one-third of the way through the sheath. Courtesy LASL/CRT 8-5-69.



618-1-1 ~100x

Re Wire X69-230



618-1-3 250x

Fig. 19b. Photomicrographs of rhenium wire sensor tested in Sheath #6 at LASL in June 1969. Above, before etching. Right, after etching. According to LASL's interpretation the enhanced grain boundary delineation near the surface is probably due to carbon absorption. Photomicrographs courtesy of LASL/BGG 9-19-69.

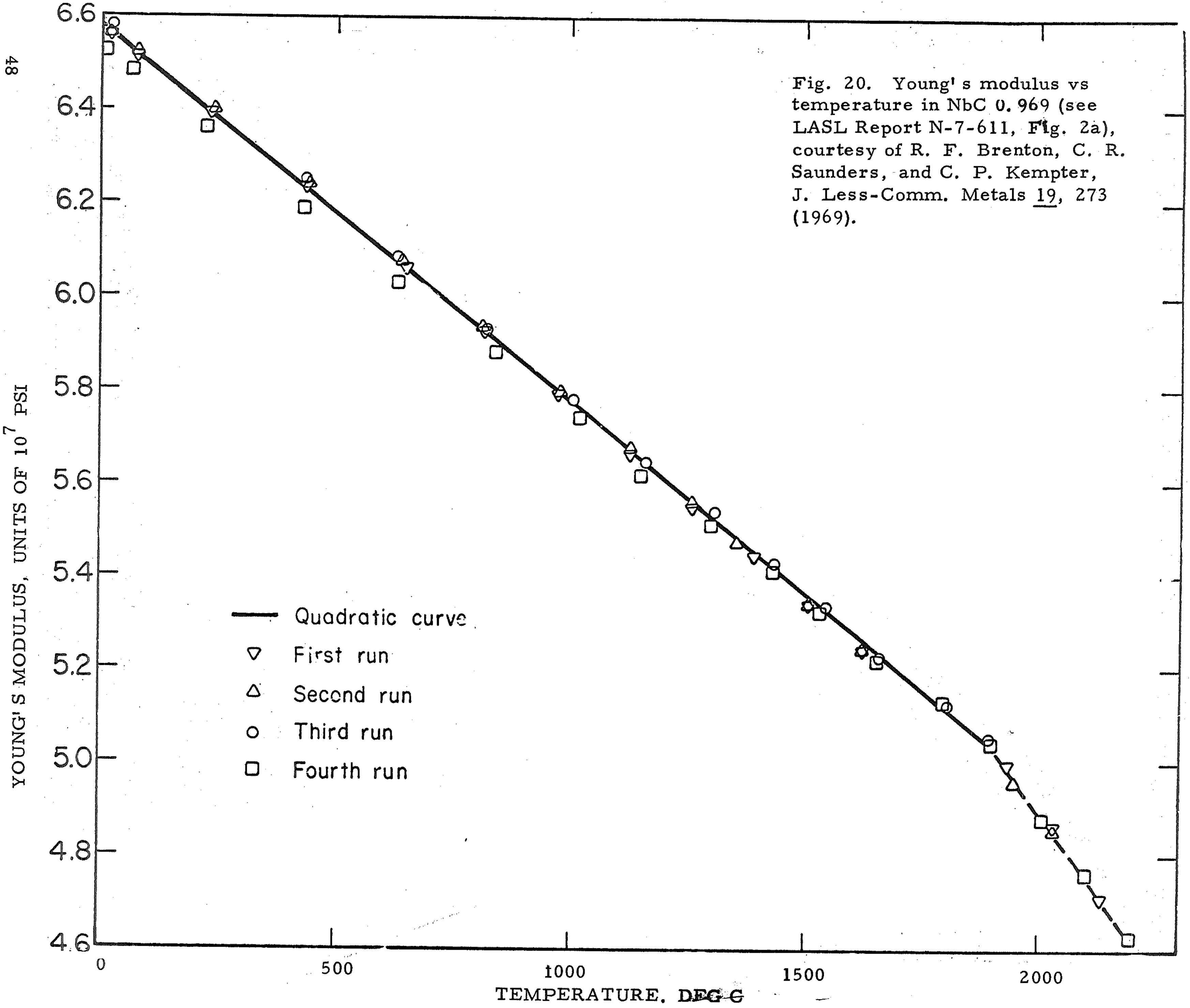
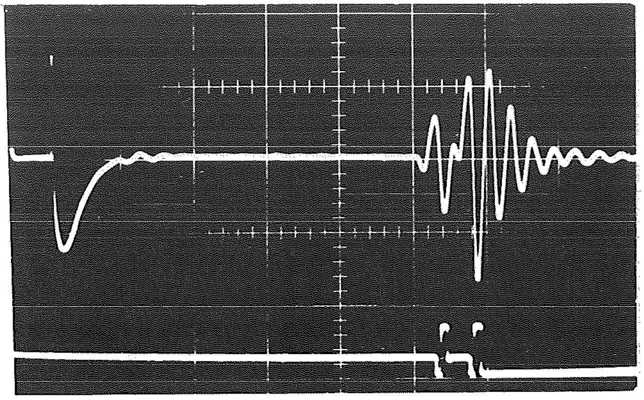
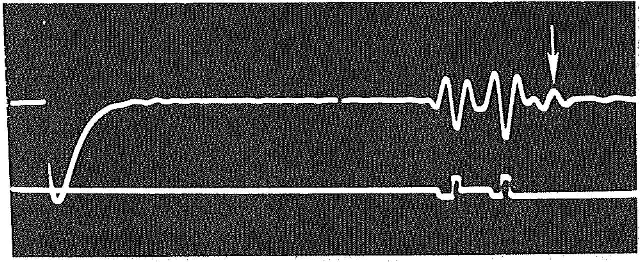


Fig. 20. Young's modulus vs temperature in NbC 0.969 (see LASL Report N-7-611, Fig. 2a), courtesy of R. F. Brenton, C. R. Saunders, and C. P. Kempter, J. Less-Comm. Metals 19, 273 (1969).

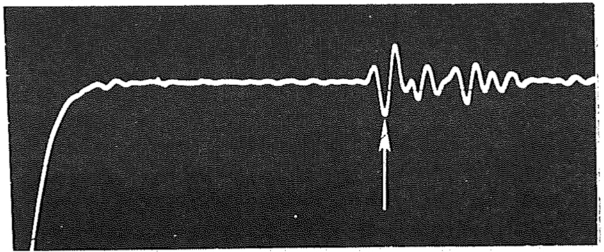
#4-1 Sheath #4 Room Temp



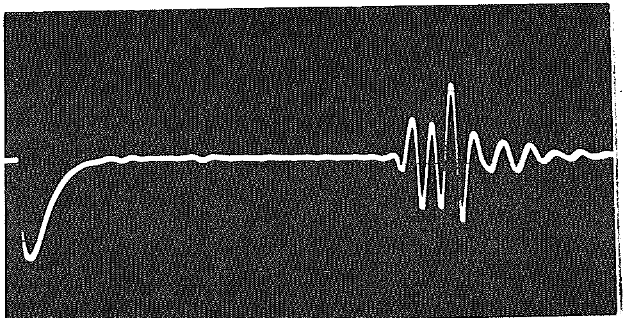
#4-2 Multiple Sensor Echo at 4766°R



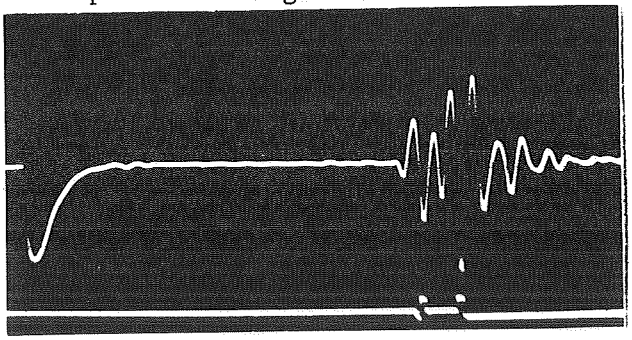
-3 Spurious echo after 9 min at ~4750°R



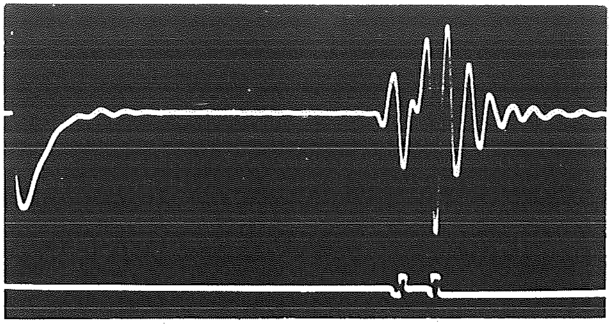
-4 Spurious echo after 9 min at ~3890°R



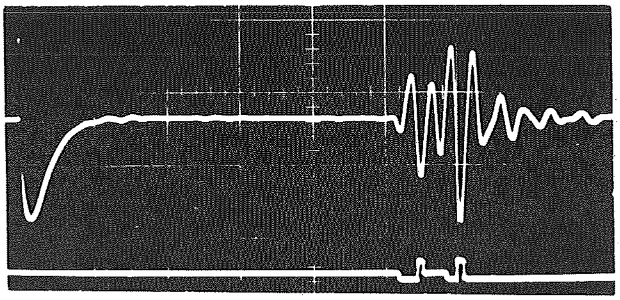
-5 Spurious echo gone at 2440°R



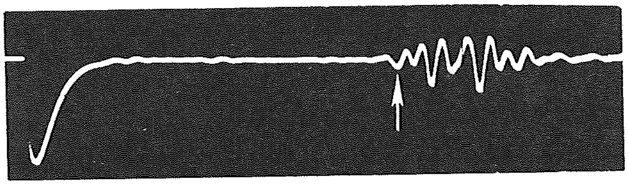
-6 Clear echoes prior to reheating



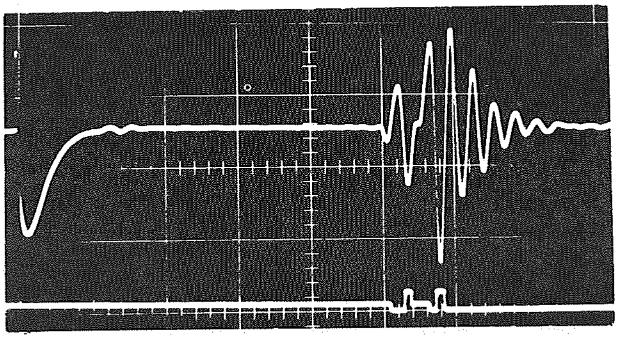
- 7 Clear echoes at 3465°R



-8 Spurious echo and atten. at ~3500°R



2-1 Sheath #2 Room Temp



#2-2 Sheath #2 4524°R

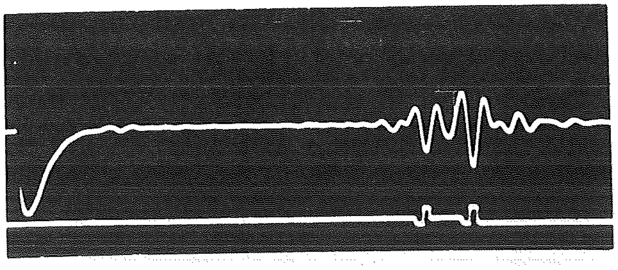


Fig. 21-#4-1 to 8; #2-1, #2-2. Oscillograms for sheaths tested at LASL on 24 June 1969. Sweep, 100 μsec/cm.

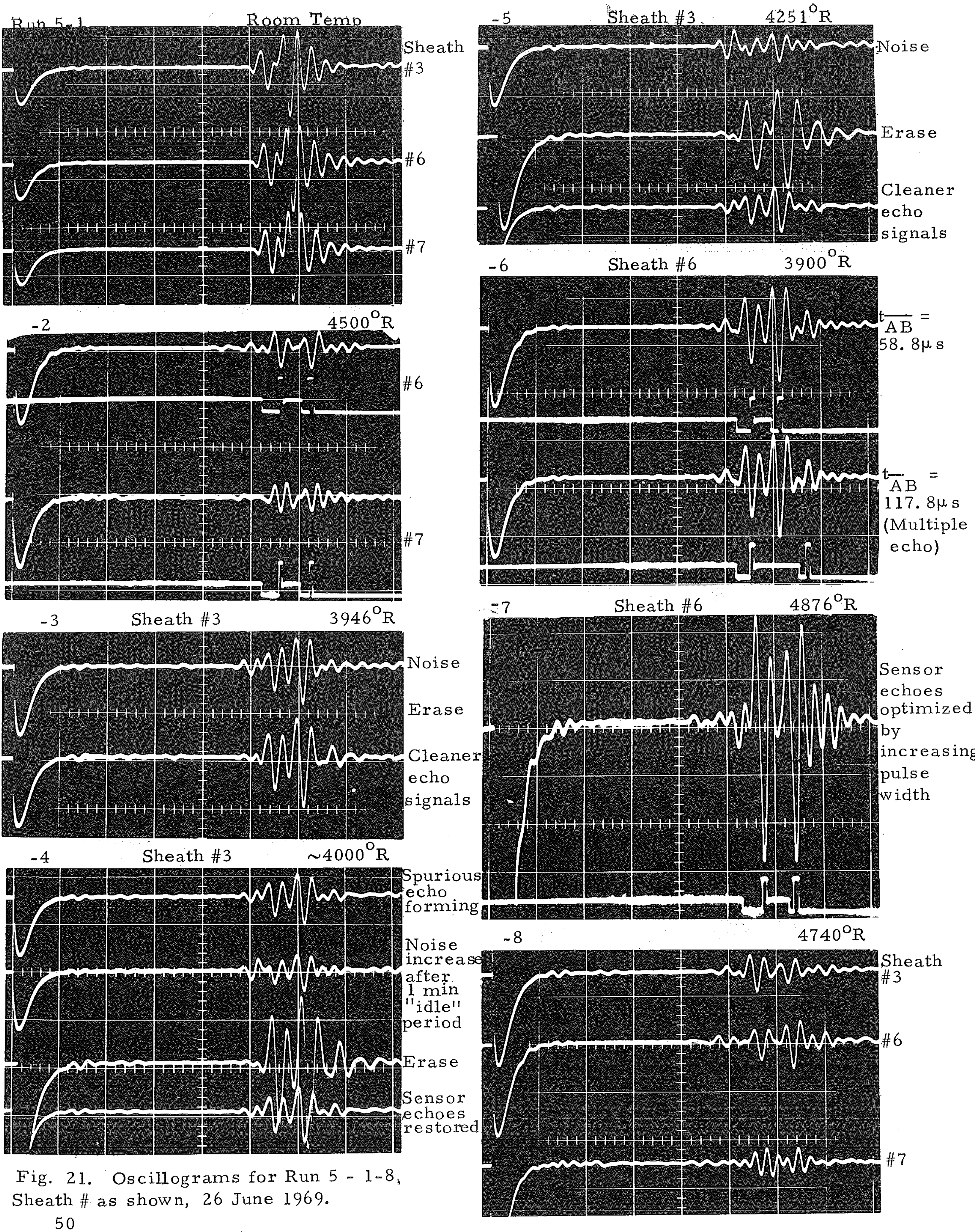


Fig. 21. Oscillograms for Run 5 - 1-8, Sheath # as shown, 26 June 1969.

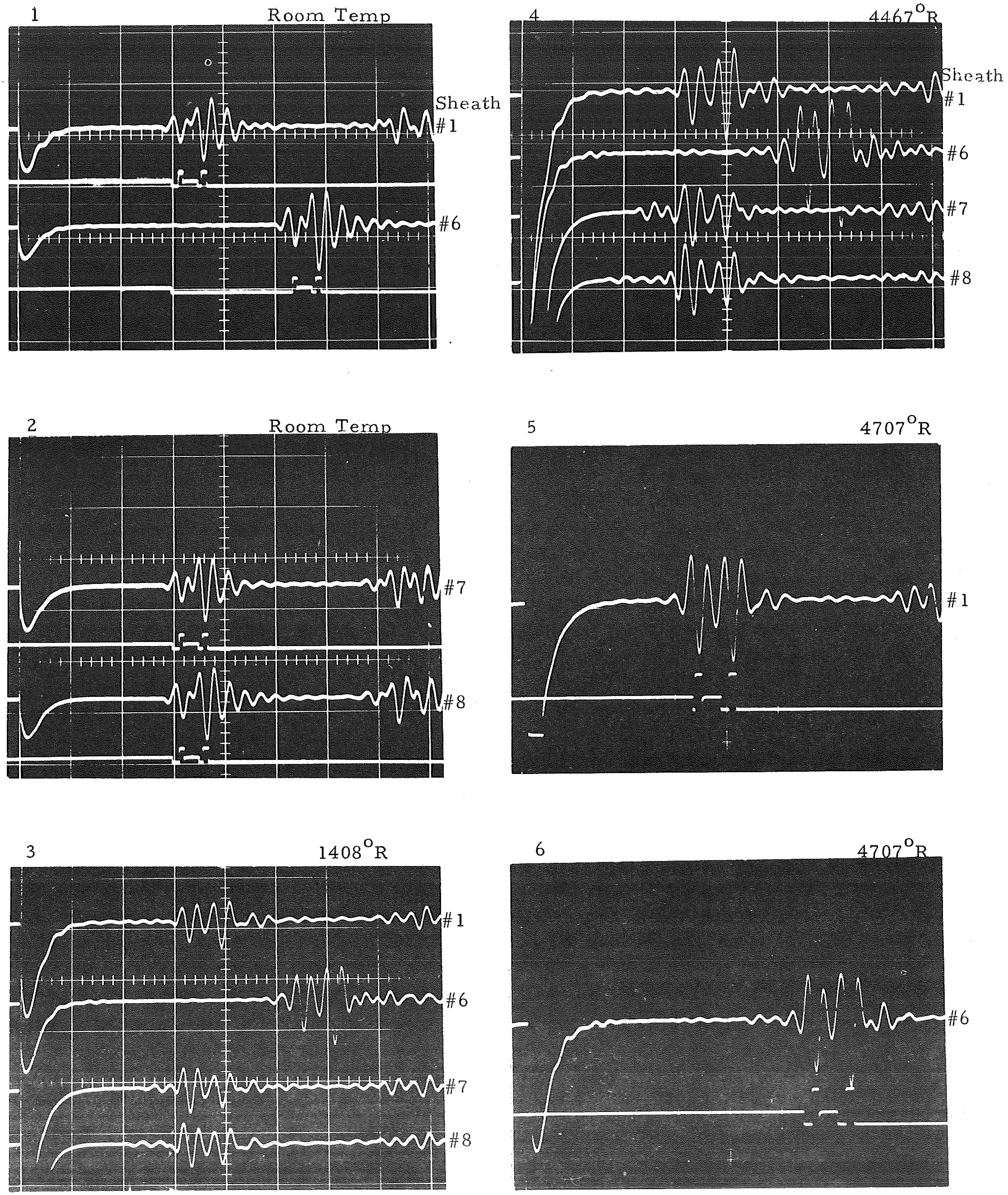


Fig. 21. Oscillograms 1 to 6 for Run 6, Sheath # as shown, 27 June 1969.

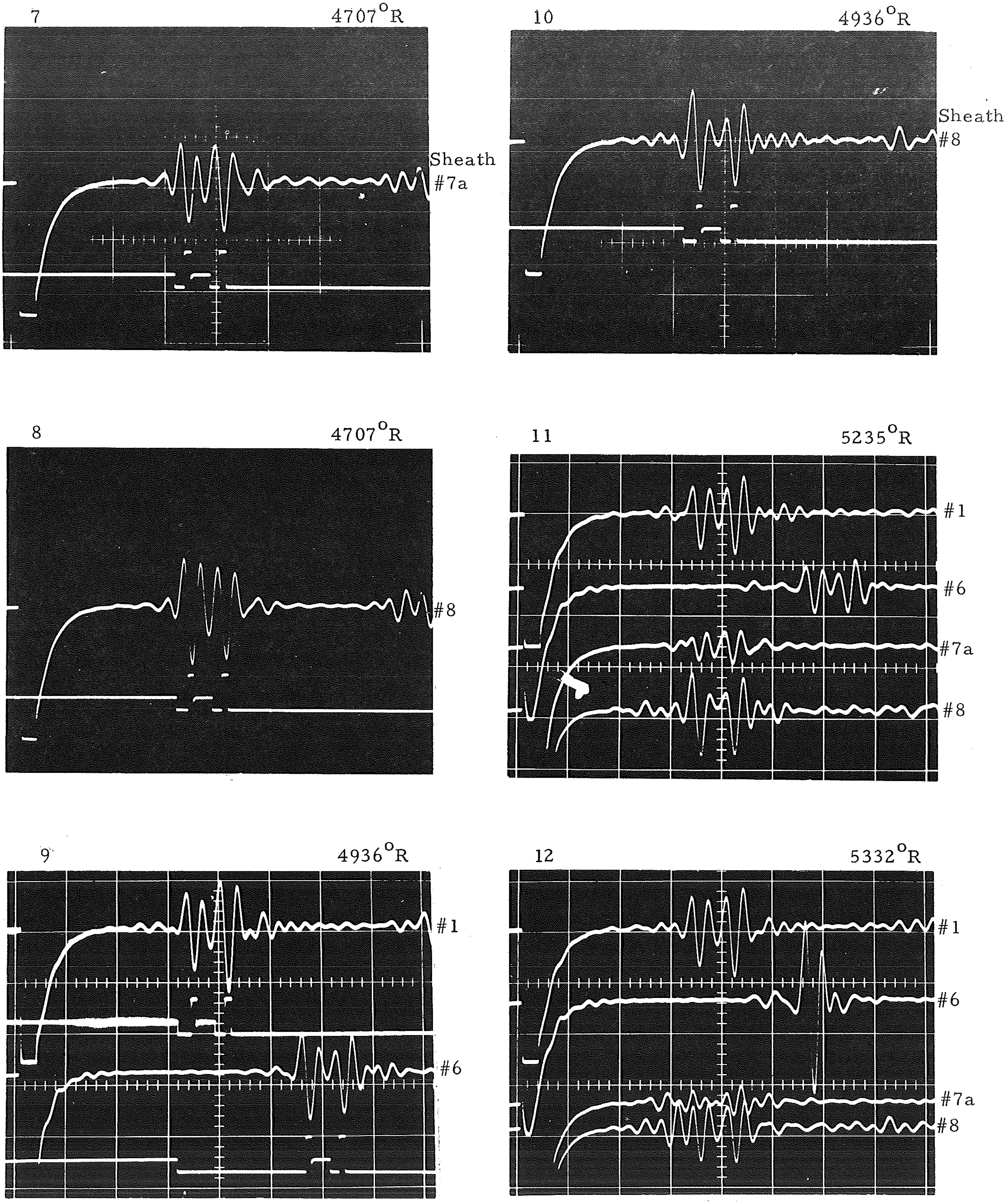


Fig. 21. Oscillograms 7 to 12 for Run 6, Sheath # as shown, 27 June 1969.

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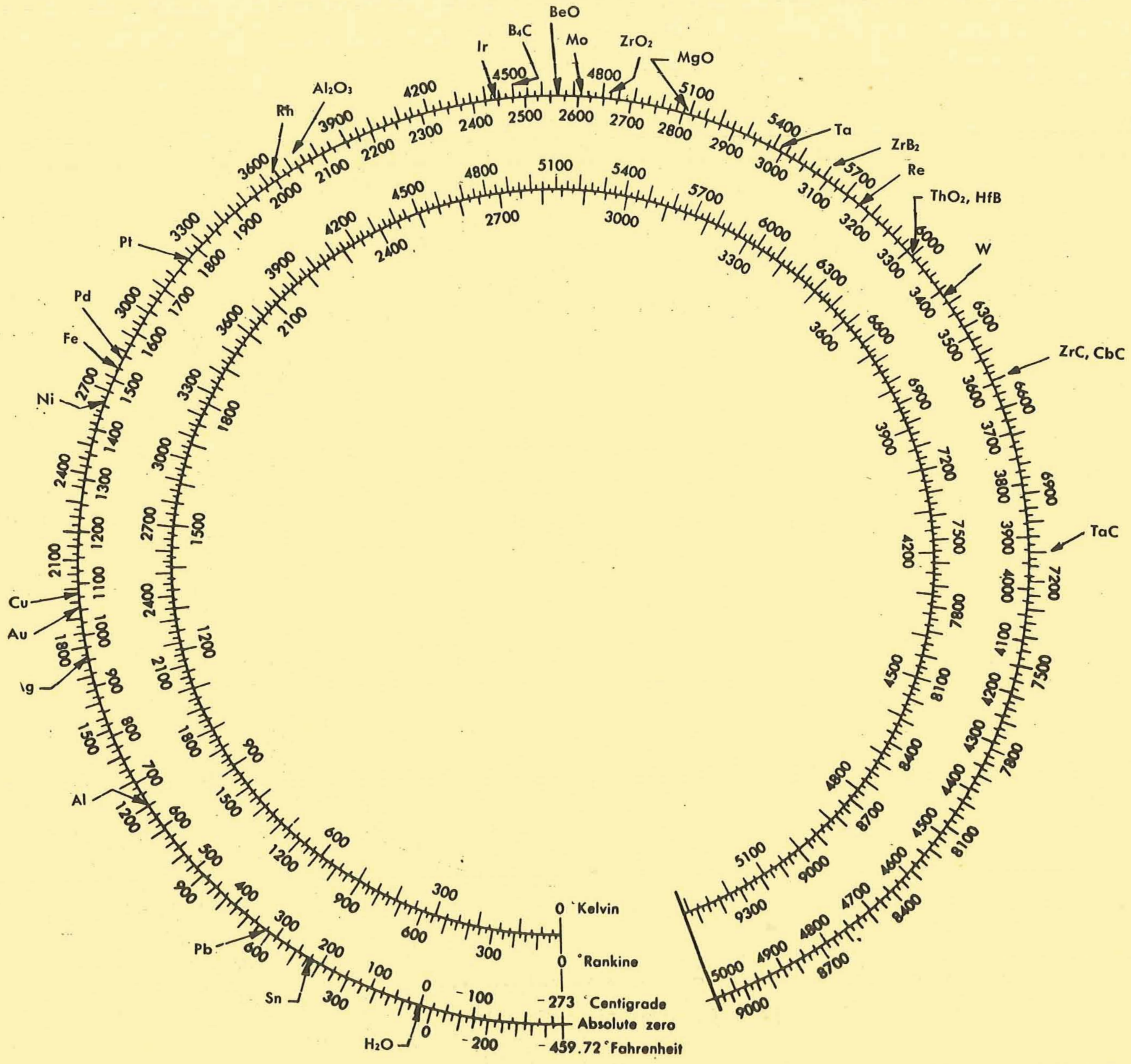
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