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# EFFECT OF NUCLEAR RADIATION ON MATERIALS AT CRYOGENIC TEMPERATURES

by

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# FOREWORD

This quarterly report is submitted to the National Aeronautics and Space Administration, Lewis Research Center, by the Lockheed-Georgia Company in accordance with the requirements of NASA Contract NAS 3-7985.

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## 1 SUMMARY

This is the fifth quarterly report summarizing the work to date on Contract NAS 3-7985 entitled, "The Effect of Nuclear Radiation on Material at Cryogenic Temperatures." The studies under this contract include the effects of: (1) $10^{18}$ n/cm$^2$ (E > 0.5 MeV) at 30°R on tensile properties of titanium base alloys; (2) irradiation temperature (30°R to 540°R) on tensile properties of Aluminum 1099-H14 following irradiations up to 3 x $10^{17}$ n/cm$^2$ (E>0.5 MeV); (3) annealing following irradiation at 30°R to $10^{17}$ n/cm$^2$ (E > 0.5 MeV) on tensile properties of Aluminum 1099; and (4) irradiation at 30°R on axial, low-cycle fatigue properties of titanium base alloys.

The tensile testing phase of the contract is being performed with government owned test equipment which was available at the beginning of the contract. All out-of-pile tensile testing and in-pile testing of Aluminum 1099, Ti-55A, Ti-5Al-2.5 Sn (ELI), Ti-5Al-2.5 Sn (Std. I) and Ti-6Al-4V (Annealed) has been completed.

Increases in strength functions, accompanied by moderate reductions in ductility, were observed in the titanium alloys for which testing was complete. There was no evidence of saturation at $10^{18}$ n/cm$^2$ (E > 0.5 MeV).

The modification of two test loops for low-cycle fatigue, including calibration of the load monitoring instrumentation, has been completed. The modified loops have been used to perform out-of-pile fatigue tests at room temperatures; and at 30°R during irradiation, following irradiation to $10^{17}$ n/cm$^2$ and unirradiated. A total of forty-seven fatigue tests have been run at room temperature, fifty at 30°R. Cryogenic strengthening of Titanium 55A appears to have a greater effect on tensile properties than on fatigue life; irradiation at 30°R to $10^{17}$ n/cm$^2$ produces a greater effect at high load levels than at loads below 85% of the nominal $F_{tu}$. The effect of cryogenic strengthening of both interstitial levels of Titanium 5 Al-2.5 Sn on fatigue life is as pronounced as for tensile properties; no testing has as yet been conducted on irradiated specimens of this alloy.

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## 2 INTRODUCTION

The combination of a fast neutron and cryogenic environment encountered in the structural members of a liquid hydrogen nuclear rocket imposes service conditions dissimilar to those encountered in other engineering applications. Both fast neutron bombardment and extremely low temperatures affect the mechanical properties of engineering materials; therefore the magnitude of the combined effect must be determined to provide basic design information before materials for a reliable nuclear rocket system can be selected. Since the neutron irradiation effects will spontaneously anneal even at low temperatures, tests to provide the desired information concerning the combined effect must be conducted with the specimens held at the temperature of interest during the entire irradiation and testing period.

A screening program (ref. 1) was undertaken to assess the effect of fast neutron irradiation on selected engineering alloys at temperatures near the boiling point of liquid hydrogen (-423°F). Tensile tests on parallel sample sets of unnotched specimens for each alloy at room temperature unirradiated, at 30°R (-430°F) unirradiated and at 30°R irradiated to $1 \times 10^{17}$ n/cm$^2$ (E > 0.5 MeV), were performed at the NASA Plum Brook Reactor Facility using a helium refrigerator and testing equipment specially designed for in-pile testing under controlled temperature conditions.

Test results from the screening program indicated that titanium alloys possessed the highest strength-to-weight ratio following exposure to the combined nuclear-cryogenic environment as well as being among the least susceptible to deterioration of mechanical properties of the alloys tested. On the other hand, Aluminum 1099 (99.99% Aluminum) was found to be very sensitive to both irradiation and temperature of irradiation.

Based on the information obtained from the screening program, an in-pile test program (see section 5) has been initiated to study in greater detail the effects of a combined nuclear-cryogenic environment on the mechanical properties of metals. The objective of this program is to provide engineering data at higher integrated fluxes and/or under different load conditions than heretofore attained at cryogenic temperatures as well as data for more fundamental studies. Its scope consists of two general phases, tensile testing and low-cycle fatigue testing. The tensile testing phase includes irradiations at 30°R to $10^{18}$ n/cm$^2$ (E > 0.5 MeV), irradiations to $10^{17}$ n/cm$^2$ (E > 0.5 MeV) at temperatures between 30°R and room temperature (540°R), and irradiations to $10^{17}$ n/cm$^2$ (E > 0.5 MeV) at 30°R followed by specimen warm-up prior to fracture. The low-cycle fatigue testing phase includes both fatigue testing during irradiation at 30°R and fatigue testing following irradiation at 30°R to $10^{17}$ n/cm$^2$ (E > 0.5 MeV).

Standard test specimens cannot be used in this test program due to various restrictions on the test equipment imposed by the nuclear cryogenic environment. The tensile specimens being used represent a miniaturization of the standard ASTM E-8 specimen (ref. 3). The miniature fatigue specimens required in this program represent a departure from any commonly used design, but are similar in geometry to those used by other investigators (ref. 4), miniaturized to meet the requirements of the program.

Progress during the earlier reporting periods (refs. 5, 6, 7 and 8) consisted of necessary preparations, neutron flux mapping, temperature correlations, equipment modification, and some test results, including preliminary out-of-pile fatigue testing.

During this reporting period tensile testing of Titanium 5 Al-2.5 Sn was completed and low-cycle fatigue testing of Titanium 55A, Titanium 5 Al-2.5 Sn (ELI) and Titanium 5 Al-2.5 Sn (Std. I) was initiated.

## 3 TEST EQUIPMENT

The test equipment (figure 1) for in-pile and out-of-pile testing under controlled temperature and load conditions permits the test program to be performed wholly by remote operations. Most of this equipment had undergone major overhaul and modification (ref. 2) in preparation for the nominal 140 hours irradiation period to obtain $10^{18}$ n/cm$^2$ ($E > 0.5$ MeV) exposures. Maintenance and calibration schedules, established during this overhaul effort, have kept the equipment operating reliably. This equipment and its operation, described previously (refs. 1, 5, 6, 7 and 8), is summarized in the following sections for purposes of discussing information pertinent to the design, modification, and performance characteristics.

### 3.1 TEST LOOPS

The test loops are stainless steel cylindrical envelopes, six inches OD by about nine feet long, containing all necessary equipment for irradiating a test specimen under controlled temperature conditions and fracturing the specimen, at temperature, in tension or compression without removal from the irradiation field. At the aft end of the test loops, fittings are provided to connect the refrigeration system, the load control system, and the instrumentation and data recording system. Other fittings are provided for test loop cooling using deionized water (which must be isolated from the helium refrigerant).

To perform the test program, five tension-compression test loops are currently being used as follows:

Test Loop 201-001 (the prototype loop): design and maintenance studies,

Test Loop 201-002: leak in inner helium was repaired during this period (section 3.7.1) and loop was placed in semi-permanent storage in hot laboratory area due to service replacement with loop modified for fatigue testing (Loop 201-004),

Test Loop 201-003: Used for tensile testing in Reactor Cycle 49P, early in reporting period. Replaced in service with loop modified for fatigue testing (Loop 201-005) and placed in semi-permanent storage in hot laboratory area during Reactor Cycle 50S, Test Loop 201-004: Used during this reporting period for low cycle fatigue testing and tensile testing. During Reactor Cycle 54S, near the end of the reporting period, this loop was removed from service and placed in temporary storage in the lead shielded cask due to the temporary lack of a second pair of operable transfer lines (section 3.7.2),

Test Loop 201-005: Used during this reporting period for low cycle fatigue testing and tensile testing. A loss of dynamometer signal resulted in an aborted test in this loop during Reactor Cycle 50P after an irradiation exposure of $0.2 \times 10^{17}$ n/cm$^2$. The loop was removed from the quadrant by remote techniques and repaired and returned to service during the same reactor power cycle. Loop 201-005 has been used without incident for 14 irradiations for a total exposure of $22 \times 10^{17}$ n/cm$^2$ since repair.

Low-cycle axial tension-compression fatigue tests as well as tensile tests are being performed using existing tension-compression test loops. The original specifications to which the test loops were constructed required that they be capable of exerting tensile or compressive loads, but not both in a cyclic manner. Therefore, the existing self-aligning features have been replaced by a more complex arrangement and considerable analysis and some modification was required before reliable tensile-compressive fatigue data could be obtained. The modified specimen holder design is shown in figure 2.

The prototype tensile test loop (201-001) and tensile test loop 201-005 were used to experimentally determine the extent of modification required. After completion of the detail design and experimental evaluation of the fatigue loop concepts in these test loops, similar modifications were performed on loop 201-004. Results of these efforts were summarized in a previous report (ref. 8).

## 3.2 REFRIGERATION SYSTEM

The test specimen temperature is maintained at temperatures between 30° - 540° Rankine using a gaseous phase helium refrigerator system. This system (ref. 5) contains an electrically driven positive displacement compressor, counterflow heat exchanger and four reciprocating expansion engines. The system was specifically designed and fabricated for this application to provide a minimum of 1150 watts of refrigeration for maintaining any specified specimen temperature from 30°R to 540°R by varying engine speed, expansion engine pressure ratio, and the heat input from manually controlled electrical resistance heaters installed in the refrigerant distribution manifold. As is reported elsewhere (section 3.7.2), the manually controlled heater was inoperative during much of this reporting period. This did not affect the operation of the system at 30°R, the desired test temperature for all the specimens scheduled during this reporting period.

The refrigeration system was operated for approximately 1520 hours during this reporting period. The system operated without incident except for a piston rod failure which did not cause loss of refrigeration on the specimen under test at the time. The rod was replaced and the affected engines returned to service without loss of refrigerator time.

## 3.3 LOAD CONTROL SYSTEM

The existing specimen loading system for tensile testing utilizes a positive displacement pump with demineralized water as the working fluid to provide the pressure required by the hydraulic actuator positioned in the test loop. Strain rate can be controlled through a variable speed drive connected to the pump.

The load transducer is located in the test loop and the extensometer is positioned directly on the specimen to measure only the strain which occurs between the gage marks. The pump and recording instrumentation are located in appropriate cabinets positioned on the grating above the quadrant at the 0'-0" level.

To perform the low-cyclic fatigue studies this system has been modified (refs. 5 and 6) to provide a closed loop servo system as shown in figure 3. The modified system includes an oil operated actuator mechanically coupled to a demineralized water operated actuator, which in turn provides the required flow and pressure to the actuator installed in the test loop.

Installation, check-out and calibration of the system was completed during the previous reporting period (ref. 8).

## 3.4 TRANSFER SYSTEM

To permit insertion and withdrawal of the test loops into the reactor, during reactor operation, a transfer system was designed and installed in quadrant D of the Plum Brook Reactor Facility. In addition, provision to change test specimens was incorporated by the installation of a hot cave with an access port in line with the assigned reactor beam port HB-2, as shown in figure 1.

To position the test loop for insertion or withdrawal from either the beam port or hot cave, the supporting tables, which are submerged approximately twenty feet in quadrant water, are aligned remotely using hydraulic pressure provided from an axial piston pump using demineralized water as a working fluid. After positioning, the loop carriage is coupled to the access port and the loop is inserted or withdrawn by a worm-drive screw arrangement driven by a hydraulic motor.

During this reporting period. the transfer system was used for a total of thirty-one cycles* of test loop insertion and removal. The system performed satisfactorily during this operational period. Except for routine maintenance discussed in section 3.7.4, the system operated without incident.

## 3.5 SPECIMEN CHANGE EQUIPMENT

Due to the high activity level of the test loops after several in-pile exposures, remote handling techniques are required for changing specimens. A hot cave provides adequate shielding for this operation. This cave is provided with manipulators, support fixtures and special tools to permit change-over of the specimen. In addition, minor repairs on the forward end of the test loop have been performed in this hot cave.

During this reporting period, the specimen change equipment was used for installation and removal of over one hundred test specimens. No specimen change equipment difficulties were encountered.

* For cycle definition, see section 3.7.

## 3.6 MISCELLANEOUS TEST EQUIPMENT

During this reporting period, the test loop transfer cask and associated equipment were used to move a test loop to the hot laboratory area in accordance with approved procedures. Loops 201-002 and 201-003 were placed in semi-permanent storage in the hot laboratory area, with Loop 201-003 requiring transfer from the containment vessel.

Loop 201-004 was placed in the shielded cask for temporary storage, where it remained at the end of the reporting period.

## 3.7 TEST EQUIPMENT MAINTENANCE AND CALIBRATION

Projected maintenance schedules for the test equipment and refrigerator system define the major sub-systems associated with the test equipment and the components contained therein that require periodic scheduled inspection, adjustment, repair and overhaul. The maintenance and calibration program previously developed by a reliability analysis provided a use cycle and a common criterion for maintaining records of the use and performance of scheduled maintenance on the test equipment. The cycle is as follows:

- Insertion into hot cave for specimen installation.
- Removal from hot cave after specimen installation.
- Insertion into reactor beam port for test irradiation.
- Withdrawal from beam port after completing test, and positioning the loop for insertion into the hot cave for specimen change-over.

Normal operation of the test equipment follows this cycle. However, most of the equipment operates submerged in the quadrant water, and with the exception of the carriages and test loops, is accessible for maintenance only when the quadrant is drained. Some deviations from the projected schedules are therefore necessary.

The projected refrigerator maintenance schedule is related to the hours of operation which are recorded cumulatively on a time meter which operates when the expansion engines are operating. Operating time is maintained by recording start-up and shut-down time on the refrigerator operation logs. Maintenance logs are used to record normal and abnormal maintenance and repair.

During this reporting period, the projected maintenance schedules were adhered to, inasmuch as possible, to perform routine inspection and repair. A number of repairs and adjustments were performed, some being a continuation of effort previously reported (refs. 6, 7 and 8) and some required due to equipment malfunction during performance of the test program.

### 3.7.1 Test Loop Repairs

As previously reported (refs. 5, 6, 7 and 8) leakage had been observed in one of the refrigerant lines of test Loop 201-002. This leak was between the refrigerant line and the coaxial annular vacuum insulating space in the aft end of the helium inlet.

This leak was successfully repaired during this reporting period. First, the precise location of the leak was determined by connecting a mass spectrometer type leak detector to the annular space and introducing helium into the aft end of the refrigerant line in small incremental volumes, the forward portion of the refrigerant line being blanked-off with a movable barrier during the test. After the site of the leak was known, the annular space was evacuated and a commercially available metal filled epoxy sealant (Devcon B - liquid type) was applied to the inner wall of the refrigerant line in the area of the leak using a specially built pneumatically operated tool. The effectiveness of the repair was tested by thermal shock accomplished by filling the line with liquid nitrogen and boiling it off. No leak was detectable, after five thermal cycles, using a mass spectrometer leak detector at maximum sensitivity. After testing, Loop 201-002 was placed in semi-permanent storage.

As reported elsewhere (section 3.1) Loop 201-005 developed a degradation of dynamometer during Reactor Cycle 50P. This loop had received its initial irradiation in this cycle. At the time of the instrumentation failure, the total irradiation received was less than six hours, for a total accumulated dose of less than $10^{16}$ n/cm$^2$ (thermal). The specific activity resulting from this exposure was of a sufficiently low level to permit repairs to be made in the containment vessel with a shielding of movable lead sheets. Authorization to perform this repair was obtained and the dynamometer was replaced, using approved procedures and health safety monitoring. The repairs were completed during the following specimen irradiation, in Loop 201-004, and the loop was returned to service before the next irradiation scheduled to use Loop 201-005.

### 3.7.2 Refrigeration System Repairs

The transfer lines terminate in a thermally isolated enclosure containing refrigerant shut-off and by-pass valves normally used to isolate the test loop from the refrigerant stream and to permit circulation of the refrigerant in the transfer lines to maintain them at low temperature during specimen change-over. These valves, in all three transfer line assemblies, have frequently malfunctioned or leaked so severely that they could not be used for their intended application, thus requiring the utilization of manually operated valves in the manifold to isolate the test loops. In addition, leaks occurred in the flexible portion of the transfer lines (refs. 5, 6, 7 and 8) resulting in a heat leak into the lines for exceeding the permissible rate.

One set of transfer lines was at the manufacturer's factory undergoing modification and repair at the start of this reporting period. These lines had not been returned to Plum Brook at the close of this period.

About midway in the reporting period, an additional set of transfer lines, previously modified by the manufacturer, developed a low temperature leak. The leak was of sufficient magnitude to render the lines unusable at 30°R. They were removed from service and the leak was located using low temperature techniques. The lines have been de-contaminated and removed from the containment vessel. Methods of effecting repairs of these lines, either by Lockheed personnel at Plum Brook or the manufacturer, are being investigated.

This development limits the experiment to the operation of a single test loop at cryogenic temperatures in place of the concurrent two loop operation previously used.

Early in the reporting period a piston rod in expansion engine No. 3, pod No. 1, failed during refrigerator system operation necessitating the shut down of both engines in this pod. The test chamber temperature (30°R) was maintained with the other pair of expansion engines and the test was continued without loss of refrigeration time during disassembly of the engines, replacement of the failed rod, and re-assembly and re-start of the engines.

During the long scheduled reactor shut-down, Cycle 54S, the refrigeration system was thoroughly overhauled. Re-built expansion engines were installed in both pods. Engines 5 and 6 were installed in pod 1; 3 and 4 in pod 2. The manifold was opened to allow inspection and repair of the 2500 w main line heater (ref. 8). A short circuit was discovered to be the cause of malfunction. This was corrected and the heater is now in service. The heat exchanger was flushed with Freon No. 11 TF to remove accumulated carry-over oil and other contaminants to improve the primary to secondary side heat transfer characteristics. The Freon was thoroughly pumped from the system after cleaning was complete. The high pressure control valve (V 6) pilot was cleaned and refurbished. All set point and control gages were checked and calibrated. The refrigeration system was returned to service on October 11 and has run continuously without incident since that date.

### 3.7.3 Corrosion of Test Equipment

As previously reported, evidence of corrosion had been observed in test head assembly 201-010. The principal corrosion, discussed in detail in reference 6, occurred at the welded peripheral seams of the stainless steel actuator bellows assemblies which separate helium from cooling water in the test loops. To alleviate this problem, the welded bellows in all of the test loops will be replaced by two sections of two-ply hydraulically formed bellows, welded end-to-end and welded to suitable adapters at the ends. The end-to-end welding is required because the section lengths are limited by the forming technique. The spring rate of the new bellows assembly is about 22 lb/in.

The first replacement was made in the prototype loop to determine the best installation procedures before replacement of the bellows in the other loops. Following the replacement of a bellows in the prototype loop, a new bellows was installed in the non-irradiated test loop 201-005, to permit further refinement of the techniques. The methods developed provided leak-free joints in these loops. Since this repair, Loop 201-005 has withstood in-pile exposures to over $22 \times 10^{17}$ n/cm$^2$ with no evidence of bellows leakage.

However, it remains to be determined if these methods are adequate for fabrication of leak-free welds in the limited working exposures permitted on the irradiated test loops. Replacement of the bellows in the irradiated test loops will not be attempted until necessitated by a bellows failure.

### 3.7.4 Miscellaneous Repairs and Adjustments

The principal miscellaneous maintenance work during this reporting period consisted of routine activities such as replacement of carriage drive motors and gears, rebuilding the load actuators in Loop 201-001 (after some 100,000 cycles) and replacement of the seals and changing the oil in the 10HP high pressure water pump (after 75.1 hours running time).

## 3.8 EXPERIMENT DESIGN MANUAL AND HAZARDS ANALYSIS

As previously reported (ref. 5), revisions to the Experiment Design Manual and Hazards Analysis were required, since:

- The present test program includes irradiation exposures at 140°R, 320°R and 540°R, as well as at 30°R. Prior experiment approval from the Plum Brook Reactor Facility was predicated on operation at 30°R, freezing-out gaseous impurities in the refrigerant prior to irradiation of the gas.

- The present test program includes cyclic loading from tension to compression, thus changing the stress pattern on the test loop head from that used as a basis of the stress analysis on which prior experiment approval was based.

The modification of the Experiment Design Manual and Hazards Analysis required by the increased irradiation temperature was completed and reported in the preceding reporting period (ref. 6). These changes consisted of an activation analysis of the possible impurities in the refrigerant and determination of the degree of hazard incurred in the event of the maximum credable incident. The calculations and conclusions were included in the Experiment Design Manual and Hazards Analysis and the Plum Brook Reactor Facility has granted approval for experiment operation at all temperatures up to 540°R.

A refined hazards analysis, including various components in the test loop, operating in tension and compression in cyclic loading up to a maximum of 3500 pounds load, was completed during the last reporting period (ref. 8). The results, discussed in detail in reference 8, have been included in the Experiment Design Manual and Hazards Analysis. They show that the possibilities of heat bolt, ring seal and head assembly end cap failure are remote even under the most severe operating conditions and that even if such a failure should occur it would not constitute a hazard to the reactor or test facility operation.

# 4 TEST PROCEDURES

The test procedures discussed in the following sections are required for the acquisition of data under the carefully controlled test program environmental conditions and the reduction, analyses and interpretation of the data thus generated. Brief discussions of test specimen designs, flux mapping, tensile test methods, fatigue test methods, and post-exposure structural studies follow.

## 4.1 TEST SPECIMEN DESIGN

The test specimens used in this program are miniaturized due to various restrictions on the test equipment imposed by the nuclear cryogenic environment. Two specimen designs, one for the tensile test program and one for the fatigue test program, are required.

The tensile specimen, shown in figure 4 and discussed in detail in reference 5, represents a miniaturization of the standard ASTM E-8 specimen (ref. 3). It is essentially a cylindrical tensile coupon, approximately two inches overall length, with threaded ends. The specimen gage length is 0.5 inch with a nominal diameter of 0.125 inch at the mid-point in the gage length, which conforms to the standard 4:1 gage length to diameter ratio. There is a slight taper to the mid-point of the gage length to ensure fracture in that area.

The fatigue specimen design is shown in figure 5. Fatigue specimen design is not as standardized as tensile specimen design and the fatigue specimen used in this program represents a departure from any commonly used design. However, the specimen geometric configuration is similar to that used by other investigators, such as Coffin (ref. 4). This allows some comparison between this data and data from other laboratories and no deficiencies in the design have been indicated.

## 4.2 FLUX MAPPING

Accurate knowledge of the fast flux available in HB-2, both spectral shape and level, is necessary to determine the irradiation exposure required to provide the desired integrated flux for each specimen.

The fast flux was measured at various reactor operational parameters during the preceding reporting period (ref. 6) using fast neutron threshold foils (table 1). The results of these measurements are reported in detail in reference 6 and shown in figure 6.

A meeting of NASA and Lockheed personnel was held during an earlier reporting period for a discussion of the flux mapping activities. It was concluded that there was no significant change in flux level or spectral shape since the conclusion of the screening program. The flux curves used in the earlier program (ref. 1 and ref. 6) are still in use as the basis of exposure calculations.

## 4.3 TEST METHODS

Tensile testing requires the measurement and recording of several data for post-testing evaluation. These data include:

- Measurement and recording of the load on the specimen continuously from the initial application until specimen failure.

- Measurement and recording of the elongation of the specimen continuously from initial application of the load until a point after the total elongation represents more than 0.2 percent permanent strain.

- Measurement of specimen temperature throughout irradiation and testing.

- Measurement of elongation (a measure of total permanent strain) and reduction of area (a measure of non-uniform strain) on failed specimens as a post-irradiation examination.

The test methods required to provide accurate records of these parameters have been discussed in some detail in a previous report (ref. 5). A brief summary of these methods follows.

Load measurements are monitored with a ring type dynamometer, using a linear variable differential transformer (LVDT) to measure the ring deflection resulting from the applied load. Elongation is measured using an extensometer in which a LVDT measures the incremental separation between two knife edges initially 0.50 inch apart on the gage length of the specimen.

For load-elongation recording, the monitoring instruments convert the load or elongation into electrical signals, of which the strength is a function of the magnitude of the measured parameter. The electrical impulse from each of these instruments is amplified and plotted automatically by an X-Y recorder. Load appears as the Y plot, elongation as the X plot and the resultant load-elongation curves are recorded on graph paper as a permanent record of these test data. The extensometer is capable of measuring only about 0.010 inch elongation with reliable accuracy. After this limit of approximately two percent total strain has been reached, the recorder is switched to a load-time plot traveling at a rate of 0.02 in/sec.

The load-elongation curve developed during testing on the X-Y recorder and the initial specimen dimensions provide data for the determination of the ultimate tensile strength ($F_{tu}$) and the tensile yield strength ($F_{ty}$). The modulus of elasticity may be approximated from these curves, but an exact determination of this value is unobtainable due to the method of extensometer installation imposed by the necessity of using remote handling techniques.

Elongation and reduction of area values are obtained by fitting the broken specimens together and measuring the fractured gage length and minimum diameter by means of a micrometer stage and hair line apparatus accurate to $\pm$ 0.0001 inch. These values are reported as the change in magnitude from original specimen dimensions expressed as a percentage of the original value.

All of these methods conform to the requirements of ASTM Specification E-8 (ref. 3), with an extensometer installation classification of B-2 under ASTM Specification E-83 (ref. 3).

Fatigue testing requires less measurement and data recording than tensile testing, although the fatigue test methods are decidedly more complex than the tensile test methods. Data from an individual fatigue test consist simply of the load amplitude and cycles to failure. To obtain this data, a closed loop electro-hydraulic servo system is used to automatically apply a sinusoidal cyclic load to the specimen. The maximum tensile load is equal to the maximum compression load and is predetermined by the specimen dimension (at its minimum diameter) and by the desired percentage of $F_{tu}$.

The same dynamometer as is used in the tensile testing, described earlier, is used by the servo system as the load sensing element. Through error detector circuitry the system amplifies small differences between the load and a sinusoidal input signal and matches the load to the signal by way of a servo valve in the hydraulic loading system. The system, with the exception of the dynamometer, is calibrated prior to each test. The dynamometer is very stable and requires calibration only at extended intervals which generally include several tests.

At the beginning of a test, the cyclic load amplitude is gradually increased, in about 10 cycles, at 6 cpm, to the test load amplitude. This ramp is required to accurately set the test load amplitude without over-shooting. At the end of the ramp the cyclic rate is increased to 15 cpm and automatic cycle counting is started.

The cyclic load is recorded versus time during the ramp and at intervals during the testing as a check on the performance of the control system. The test stops automatically on reaching 10,000 cycles or when the specimen fails.

The direct measurement of specimen temperatures using thermocouples or other temperature measuring transducers was not considered practicable when performing a series of these tests (ref. 1). An alternate method of establishing the temperature of the specimen was incorporated into the refrigerator. This involves measurement of the temperature at the manifold inlet and return using platinum resistance type thermometers. These temperature measurements are averaged to give the specimen temperature. The temperature is controlled at a set point by automatic variation of the heater load.

The calibration technique consisted of calibration of three copper-constantan thermocouples attached to a Titanium 6Al-4V test specimen against a NBS calibrated platinum resistance thermometer and using this instrumented specimen as a working standard to establish refrigeration system operating parameters required for the maintenance of the desired specimen temperature both in-pile and out-of-pile. This activity for the tensile specimen is reported in detail in reference 5 and reference 6 and the important results are summarized in table 2 along with results from similar measurements for a fatigue specimen of Titanium 5Al-2.5 Sn (Std. I).

## 4.4 STRUCTURAL STUDIES

Failed tensile specimens of each test material and environmental condition in that phase of the test program have been delivered to the NASA Plum Brook Hot and Metallurgical Laboratories Section for metallographic preparation for structural studies using optical microscopy techniques. The specimens have been prepared and given preliminary examination; however, no photomicrographs had been completed at the end of the reporting period. Discussion of the structural observation will be deferred until illustrative photographs are available for publication.

## 5 TEST PROGRAM

The materials, pure aluminum, titanium and titanium alloys, to be tested during this program were selected on the basis of their potential usefulness in nuclear-cryogenic space hardware and their ability to yield fundamental information in terms of basic mechanisms occurring in metals and alloys during and following fast neutron irradiation at cryogenic temperatures. The scope of the test program, including the basis for material selection, has been previously reported (ref. 5 and 6) and consists of the following major items of investigation:

- Effects of cryogenic irradiation and annealing on tensile properties of Aluminum 1099-H14.
- Effects of irradiation at 30°R on tensile properties of titanium and titanium alloys.
- Effects of irradiation on low-cycle rate fatigue properties of titanium and titanium alloys.

All test specimens used in the program are fabricated from materials manufactured using extraordinary precautions and provided with complete chemical and metallurgical pedigrees. A summary of the pedigree information is given in tables 3 and 4.

The portion of the testing program related to the tensile testing of Aluminum 1099-H14 had been completed in an earlier reporting period and the results are reported fully in reference 8.

### 5.1 EFFECTS OF IRRADIATION AT 30°R ON TENSILE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS

The titanium alloys of primary alpha structure usually exhibit good cryogenic properties due to the hexagonal close-packed structure of this phase. They have a high modulus of rigidity and a high strength-weight ratio, which is comparable with the best aluminum alloys. Also, they have allowable working temperatures which are higher than the aluminum alloys. This makes them more suitable for rocket components, initially at cryogenic temperatures, which may see elevated temperatures during rocket firing.

The tensile testing phase of the program, shown in table 5, consists of three investigations:

- Effects of irradiation at 30°R on commercially pure titanium (Ti-55A).

- Effects of interstitial content in Ti-5Al-2.5 Sn on changes due to irradiation at 30°R.

- Effects of initial heat treatment of Ti-6Al-4V on changes due to irradiation at 30°R.

Out-of-pile test data and in-pile test data for irradiations to 1 x $10^{17}$ n/cm$^2$ (E> 0.5 MeV) were obtained in the screening program (ref. 1). The mechanical testing phase of the in-pile portion of the present program is nearing completion. All tests for Ti-55A, Ti-5Al-2.5 Sn (ELI) and (Std. I), and Ti-6Al-4V (Annealed) have now been completed. Tensile testing of Ti-6Al-4V (Aged) has been started, but insufficient data to warrant reporting has been obtained. Structural studies have been initiated during this reporting period but are not as yet completed.

### 5.1.1 Effects of Irradiation At 30°R On Titanium 55A

Titanium 55A, although of only moderate strength, has good forming characteristics and meets the requirements for some nuclear rocket application; however, it was selected for study in this program primarily because it is essentially commercially pure elemental titanium and may yield important fundamental information. It has exhibited a small but measurable increase in yield strength due to fast neutron irradiation of $10^{17}$ n/cm$^2$ (E> 0.5 MeV) at 30°R in an earlier test program (ref. 1).

This phase of the test program was completed in a previous period and reported in reference 8. The test results are repeated in table 6 and plotted as a function of integrated neutron flux at 30°R in figure 7. Figure 8 shows typical load-elongation curves for the various irradiation levels included in the testing phase of the investigation.

The data plotted in figure 7 show that there is a direct dependence of $F_{tu}$ and $F_{ty}$ on irradiation level (to $10^{18}$ n/cm$^2$ (E> 0.5 MeV)) accompanied by a significant but not critical reduction in ductility parameters. No degradation of any mechanical property of sufficient magnitude to compromise engineering integrity after exposures to $10^{18}$n/cm$^2$ (E> 0.5 MeV) was observed.

Titanium 55A is essentially a polycrystalline titanium of commercial purity. This material was tested in the annealed condition, but with standard interstitial content; therefore, the population of "foreign" substitutional solute atoms should be small but the number and distribution of interstitial atoms should be similar to the interstitial populations in alloyed materials. Since alpha titanium is a hexagonal close packed lattice material, slip might be expected to be fairly laminar -- particularly with a relatively small population of substitutional atoms. The presence of interstitials might be expected to increase turbulence of the flow during slip. Since the reported

$F_{ty}$ is based on 0.2% offset rather than on divergence from Hookes Law, the relatively low (for titanium alloys) $F_{ty}/F_{tu}$ ratio of about 0.7 at 30°R, both unirradiated and at $1 \times 10^{17}$ indicate a rather laminar behavior; the increase of this parameter to 0.75 at $6 \times 10^{17}$ and 0.78 at $1 \times 10^{18}$ indicates an increase in turbulence resultant from lattice imperfections induced by increased irradiation levels.

### 5.1.2 Effects of Interstitial Content in Ti-5Al-2.5 Sn On Changes Due To Irradiation At 30°R

Titanium - 5% Al - 2.5% Sn is a fairly high strength alpha phase alloy ($F_{tu} \approx 120$ Ksi at room temperature). It is now commercially available in the extra low interstitial grade (less than 0.125% interstitials, and designated ELI) and possibly would be specified in this grade by designers for use in shells, pressure vessels and pump parts of nuclear rockets. However, recent nuclear cryogenic tests to $10^{17}$ n/cm$^2$ (E>0.5 MeV) at 30°R, (ref. 1), indicate that the ultimate strength of the ELI material may be adversely affected by the neutron irradiation. It is conceivable that higher irradiations might cause adverse effects on various properties, including fatigue strength, which would negate any inherent advantages of the ELI material.

The tensile testing of the low interstitial grade were completed and reported at an earlier date (ref. 8). The test results obtained for this material are re-published in table 7 and figure 9.

The tensile testing of Titanium 5 Al-2.5 Sn (Std. I) was completed in this reporting period and the resultant test data are shown in table 8 and figure 10. The most prominent discrepancy between the data plotted in figure 10 and that in figures 7 and 9 is the increase in the spread of the range of values apparent for the standard interstital material after irradiation to $10^{18}$ n/cm$^2$. The initial view of this "data scatter" might be that it is due to some random test error rather than to an observable randomization of actual material behavior. However, examination of the test data in table 8 shows that the relationship among the test parameters remains consistent for the individual specimens tested after exposures of $10^{18}$ n/cm$^2$; i.e., the specimen with higher strength values had lower reduction of area values. This might indicate that a random interaction between neutrons and interstitial atoms may result in less uniform, and so less predictable, effects of irradiation at higher levels on the greater interstitial atom population. Any neutron-interstitial atom interaction would be expected to be most pronounced when light atoms were involved; the neutron would exercise a greater effect through collision with a hydrogen nucleus of approximately its own mass than with an oxygen nucleus of about sixteen times its mass.

Two other titanium alloys so-far tested, Titanium 55A and Titanium 6Al-4V (annealed) were also manufactured using standard interstitial control methods rather than special low-interstitial techniques. However, as is shown in the pedigree data in table 3, both of these particular alloy heats happened to be low in hydrogen content. The absence of this large range of values in Titanium 55A, with a rather high oxygen content, tends to confirm the importance of light atoms as a causative agent in the irradiation generated random variation in mechanical properties.

Except for the above mentioned "data scatter" in the standard interstitial material, Titanium 5 Al-2.5 Sn (ELI) and (Std. I) show similar effects of irradiation at both levels. Examination of the data in tables 7 and 8 and figures 9 and 10 show an essential similarity in behavior.

A comparison of the data shown in figures 9 and 10 with that shown for unalloyed titanium in figure 7 indicates that the larger population of substitutional solute atoms in the two grades of Titanium 5 Al-2.5 Sn imparts a more turbulent flow pattern during slip occurring in late elastic and early plastic behavior. This is observable, particularly, through the comparison of $F_{ty}/F_{tu}$ ratios.

### 5.1.3 Effects of Initial Heat Treatment of Ti-6Al-4V on Changes Due to Irradiation at 30°R.

Titanium - 6% Al - 4% V is an alpha-beta alloy in which the beta phase is metastable in the annealed condition and largely transformed to alpha by aging. The ultimate strength of the aged materials is about 170 Ksi at room temperature with favorable cryogenic characteristics and it is very likely to be specified for shells and pressure vessels in space hardware. Irradiation to $10^{17}$ n/cm$^2$ (E > 0.5 MeV) at 30°R causes measurable increases in the strength of the aged material but not the annealed material. Higher irradiations at the same temperature may confirm this effect and may possibly yield fundamental information regarding the effects of nuclear irradiation on precipitation processes. Such effects are still not very well understood although they are of wide general interest to both basic researchers and applications people.

The tests required for the in-pile tensile test program, table 5, had been completed and reported (ref. 8) at an earlier date for the annealed material. Test results are presented, along with data previously obtained (ref. 1), in table 9. The test data are presented graphically in figure 11, where straight lines are used, rather than curves, due to the lack of data between $10^{17}$ and $10^{18}$ n/cm$^2$ (E > 0.5 MeV).

Comparison of figure 11 with figures 7, 9 and 10 shows the annealed Ti-6Al-4V to change in a manner similar to the commercially pure material and the 5 Al-2.5 Sn alloy.

Testing of the aged material has been initiated but is not sufficiently advanced to warrant reporting at this time. No further evaluation of test results will be made until test data from the aged material is available for comparison.

## 5.2 LOW-CYCLIC FATIGUE TESTING

The fatigue testing phase of the testing program is, currently, concentrating on three materials: Titanium 55A, Titanium 5 Al-2.5 Sn (Std. I) and Titanium 5 Al-2.5 Sn (ELI). These materials are tested axially in compression and tension with a test ratio of unity. The tests are performed at 15 cpm with various load values determined by the test results obtained. The load is cycled between compression and tension until failure of the specimen occurs or a specimen life of 10,000 cycles has been obtained. The tests are performed under the following environmental conditions:

- Room temperature, unirradiated,
- 30°R, unirradiated,
- 30°R, following irradiation at 30°R to $10^{17}$ n/cm$^2$,
- 30°R, during irradiation.

The load is increased incrementally over the initial ten cycles until the full amplitude of cyclic oscillation has been obtained. Thus, although the materials were initially in the annealed condition, the ramp effect during loading causes a degree of work hardening prior to application of the full load.

Additional tests to ascertain the effect of variation of cyclic rate and of the ramp function will be conducted.

The test results for the fatigue testing completed at the close of this reporting period are shown in tables 10 through 17. Statistical analysis of the fatigue test data now available has not been undertaken. This activity will be deferred until all test results are available to ensure the selection of an analytical technique adequate to analyze the variability in the fatigue data obtained. The studies of the curves connecting the points in figures 12, 13 and 14, sketched in to indicate probable trends, may be altered slightly in the final report on the basis of additional data and more sophisticated analysis.

### 5.2.1 Fatigue Testing of Titanium 55A

This material is essentially an unalloyed poly-crystalline titanium. Although the testing of this material has not been completed, sufficient test data has been generated to warrant publication. Tables 10 through 13 present the existing test data, figure 12 shows tentative S-N curves for this material in three of the test conditions.

Examination of the data shows that the cryogenic increase in ultimate tensile strength is greater than the accompanying increase in fatigue life. Irradiation to $10^{17}$ n/cm$^2$ appears to increase the fatigue life at higher load ranges but seems to have little effect at load levels below 85% of the $F_{tu}$.

This material was tested at various cyclic rates at 30°R, 90% of $F_{tu}$, unirradiated. Examination of the test data does not show a statistically significant variation in fatigue life, both extreme values are for the same cyclic rate (15 cpm). Further testing will be performed at various rates.

### 5.2.2 Fatigue Testing of Titanium 5 Al-2.5 Sn (ELI)

This titanium alloy has not been tested after irradiation. The test results for this material at room temperature (540°R) and at 30°R, unirradiated, are shown in tables 14 and 15 and S-N curves from these data are shown in figure 13. Unlike the unalloyed titanium, the cryogenic increase in mechanical properties was at least as great in fatigue as in the tensile parameters.

### 5.2.3 Fatigue Testing of Titanium 5 Al-2.5 Sn (Std I)

This titanium alloy was tested rather extensively at room temperature (540°R), unirradiated. Only fragmentary data are available at 30°R, unirradiated and none for irradiated specimens.

The data shown in table 16 and plotted on an S-N curve in figure 14 show that the presence of interstitial atoms seem to increase fatigue life at room temperatures. The total interstitial content (the elements C, N, H & O) in the standard interstitial grade is 0.179%; the total in the extra low grade is 0.103% by weight (table 3). Although the difference seems small, due to the low atomic weight of interstitial elements the Standard grade has about 1.1 total atomic percent as opposed to about 0.6 for the ELI. Thus, the dislocation blocking effect of the interstitial atoms during cyclic loading could vary considerably between the two grades of material.

The increase in fatigue life at a given load in terms of the percentage of a nominal $F_{tu}$ caused by a 30°R, unirradiated, temperature is of a surprisingly large magnitude (table 17). This observation is based on the very small specimen population of test data currently available, and may not appear as pronounced after further testing.

However, one specimen loaded to 100% of the nominal $F_{tu}$ at 30°R withstood 10,000 cycles without failure. It should be noted that the ramp approach to initial loading, mentioned in section 4.3, imparts a degree of work hardening which was not present in the specimens used to determine the nominal $F_{tu}$.

## 6 REFERENCES

Reference

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# 7 TABLES

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TABLE 1 FLUX MAPPING FOILS

| Type of Foil | Nuclear Reaction | Threshold Energy, $E_T$ (Mev) | Cross Section ($\times 10^{-24}$ $cm^2$) |
|---|---|---|---|
| Indium | $In^{115}(n,n')In^{115m}$ | 0.45 | 0.20 |
| Neptunium | $Np^{237}(n,f)\ Ba^{140}$ | 0.75 | 1.52 |
| Uranium | $U^{238}(n,f)\ Ba^{140}$ | 1.45 | 0.54 |
| Sulfur | $S^{32}(n,p)\ P^{32}$ | 2.9 | 0.284 |
| Nickel | $Ni^{58}(n,p)Co^{58}$ | 5.0 | 1.67 |
| Magnesium | $Mg^{24}(n,p)Na^{24}$ | 6.3 | 0.0715 |
| Aluminum | $Al^{27}(n,\alpha)\ Na^{24}$ | 8.6 | 0.23 |

TABLE 2 TEMPERATURE CORRELATION DATA

| Test Run | Specimen Temperatures (°R) Fwd. | Mid. | Aft | Refrig. Temperatures (°R) Loop Inlet | Loop Return | Return Manifold | Heater Load (watts) | Engine Speed (RPM) |
|---|---|---|---|---|---|---|---|---|
| Tensile* In-Pile | 30.5 | 30.0 | 30.4 | 29.1 | 32.5 | 32.5 | 230 | 320 |
| Tensile* Out-Of-Pile | 29.9 | 29.6 | 29.8 | 29.5 | 30.9 | 31.0 | 410 | 290 |
| Fatigue** In-Pile | 28.4 | 28.1 | 28.4 | 27.2 | 30.5 | *** | 80 | 280 |
| Fatigue** Out-Of-Pile | 28.1 | 27.7 | 28.1 | 27.5 | 30 | *** | 60 | 250 |

* Mean of 3 runs on Titanium 6 Al - 4V tensile specimen

** One run on Titanium 5 Al - 2.5 Sn fatigue specimen

*** Main heater out, by-pass valve partly open

TABLE 3 MATERIAL COMPOSITIONS (PEDIGREE DATA)

| Alloy and Temper | Lockheed Code | Percent of Element (remainder: Titanium) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Fe | Al | Other | C | | N | | H | | O | |
| | | wt% | wt% | wt% | wt% | At% | wt% | At% | wt% | At% | wt% | At% |
| 55A Annealed | 1 Aa | 0.19 | - | - | 0.032 | 0.13 | 0.023 | 0.08 | 0.006 | 0.29 | 0.218 | 0.65 |
| 5 Al-2.5 Sn (ELI) Annealed | 3 Aa | 0.028 | 5.43 | 2.41 Sn | 0.033 | 0.13 | 0.011 | 0.04 | 0.006 | 0.29 | 0.053 | 0.16 |
| 5 Al-2.5 Sn (Std) Annealed | 8 Aa | 0.110 | 5.10 | 2.50 Sn | 0.032 | 0.13 | 0.019 | 0.07 | 0.012 | 0.57 | 0.116 | 0.35 |
| 6 Al - 4 V Annealed | 2 Ac | 0.170 | 5.95 | 4.00 V | 0.010 | 0.040 | 0.022 | 0.08 | 0.006 | 0.29 | 0.065 | 0.19 |
| 6 Al - 4 V Sol. Treated Aged | 2 Aa | 0.150 | 5.80 | 3.90 V | 0.010 | 0.040 | 0.035 | 0.12 | 0.010 | 0.48 | 0.102 | 0.31 |

TABLE 4 MATERIAL PHYSICAL CHARACTERISTICS (PEDIGREE DATA)

| Alloy / Temper | Lockheed Code | Form | Spec. | Vendor Code / Vendor Lot or Heat No. | $F_{tu}$ (Ksi) | $F_{ty}$ 0.2% offset (Ksi) | Elongation in 4D (%) | Hardness | Grain Size * |
|---|---|---|---|---|---|---|---|---|---|
| Titanium 55A Annealed | 1 Aa | 0.5" Round Bar | Mil-T-7993A Class II | (1) M-9186 | 70.5 | 60.5 | 35 | Rockwell B 87 | 5 |
| Ti-5Al-2.5 Sn (ELI) Annealed | 8 Aa | 0.5" Round Bar | Vendor 49021-1 | (1) V-2402 | 119.3 | 101.2 | 17 | Rockwell C 24.9 | ** |
| Ti-5Al-2.5 Sn (Std. I) Annealed | 3Aa | 0.5" Round Bar | AMS-4910 | (1) M-7888 | 131.0 | 127.0 | 22 | Rockwell C 31-33 | 8 |
| Ti-6Al-4V Annealed | 2 Ac | 0.5" Round Bar | Mil-T 9047C | (1) M-8574 | 146.0 | 138.0 | 15.5 | Rockwell C 30-33 | ** |
| Ti-6Al-4V Solution Treated And Aged | 2 Aa | 0.5" Round Bar | Mil-T 9047C | (1) M-9812 | 173.0 | 165.0 | 13 | Rockwell C 33-36 | ** |

* ASTM No. E112-58T

** Not Measured

(1) Titanium Metals Corporation of America

TABLE 5 SCOPE OF TENSILE TEST PROGRAM FOR STUDYING THE EFFECTS OF IRRADIATION AT 30°R ON TITANIUM AND TITANIUM ALLOYS

| MATERIAL | CONDITION | NUMBER SPECIMENS | EXPOSURE $n/cm^2$ (E>0.5 MeV) | REMARKS |
|---|---|---|---|---|
| Ti-55A | Annealed | 3 | $6 \times 10^{17}$ | (1) (2) |
| Ti-55A | Annealed | 3 | $1 \times 10^{18}$ | (1) (2) |
| Ti-5Al-2.5 Sn (ELI) | Annealed | 3 | $1 \times 10^{18}$ | (1) (2) |
| Ti-5Al-2.5 Sn (STD) | Annealed | 3 | $1 \times 10^{18}$ | (1) (2) |
| Ti-6Al-4V | Annealed | 3 | $1 \times 10^{18}$ | (1) (2) |
| Ti-6Al-4V | Aged | 3 | $1 \times 10^{18}$ | (1) |

(1) Data from tests at 30°R and 540°R without irradiation and at 30°R with $1 \times 10^{17}$ $n/cm^2$ (E> 0.5 MeV) irradiation available from screening program (ref. 1).

(2) These tests completed at the end of this reporting period.

TABLE 6 TENSILE TEST RESULTS, TITANIUM 55A-ANNEALED[b]

| Specimen | Temp. (°R) | Irradiation ($n/cm^2$, E > 0.5 MeV) | $F_{tu}$ (Ksi) | $F_{ty}$ 0.2% offset (Ksi) | $F_{ty}/F_{tu}$ | Elongation in 0.5 in (4D) (%) | Reduction of Area (%) | Fracture Stress (Ksi) | Modulus[#] ($10^3$ Ksi) |
|---|---|---|---|---|---|---|---|---|---|
| Range of 5[a] | 540 | None | 65.1-69.4 | 47.5-63.3 | 0.73-0.91 | 25-33 | 59-65 | - | 12-14 |
| Mean of 5[a] | 540 | None | 67.0 | 53.5 | 0.798 | 30.0 | 62.3 | - | 14 |
| Range of 5[a] | 30 | None | 167-172 | 118-124 | 0.71-0.73 | 33-34 | 51-54 | - | 16-20 |
| Mean of 5[a] | 30 | None | 169.4 | 122.0 | 0.722 | 33.3 | 53.0 | - | 18 |
| Range of 3[a] | 30 | $1 \times 10^{17}$ | 180-216 | 128-136 | 0.63-0.73 | 32-36 | 52-54 | - | 17-25 |
| Mean of 3[a] | 30 | $1 \times 10^{17}$ | 192.3 | 131.7 | 0.690 | 34.0 | 53.0 | - | 18 |
| 1 Aa 200 | 30 | $6 \times 10^{17}$ | 203 | 154 | 0.75 | 29 | 45 | 370 | 20 |
| 1 Aa 203 | 30 | $6 \times 10^{17}$ | 204 | 158 | 0.78 | 29 | 46 | 380 | 19 |
| 1 Aa 153 | 30 | $6 \times 10^{17}$ | 211 | 154 | 0.73 | 27 | 38 | 341 | 15 |
| Mean | 30 | $6 \times 10^{17}$ | 206.0 | 155.3 | 0.753 | 28.3 | 43.0 | 363.7 | 18 |
| 1 Aa 152 | 30 | $1 \times 10^{18}$ | 213 | 159 | 0.74 | 30 | 49 | 420 | 12 |
| 1 Aa 205 | 30 | $1 \times 10^{18}$ | 216 | 171 | 0.79 | 29 | 44 | 387 | 22 |
| 1 Aa 206 | 30 | $1 \times 10^{18}$ | 223 | 181 | 0.81 | 19 | 38 | 358 | - |
| Mean | 30 | $1 \times 10^{18}$ | 217.3 | 170.3 | 0.780 | 26.0 | 43.7 | 388.3 | 17 |

(a) From screening program (ref. 1)
(b) Previously reported (ref. 7)
(#) For comparison purposes only
\- Not determinable

TABLE 7 TENSILE TEST RESULTS, TITANIUM-5Al-2.5 Sn (ELI)-ANNEALED [b]

| Specimen | Temp. (°R) | Irradiation ($n/cm^2$ E>0.5 MeV) | $F_{tu}$ (Ksi) | $F_{ty}$ 0.2% offset (Ksi) | $F_{ty}/F_{tu}$ | Elongation in 0.5 in (4D) (%) | Reduction of Area (%) | Fracture Stress (Ksi) | Modulus[#] ($10^3$ Ksi) |
|---|---|---|---|---|---|---|---|---|---|
| Range of 5[a] | 540 | None | 118-133 | 104-119 | 0.88-0.91 | 12-19 | 39-46 | 169-171 | 15-20 |
| Mean of 5[a] | 540 | None | 126.4 | 113.4 | 0.896 | 16.0 | 42.2 | 170.0 | 15 |
| Range of 5[a] | 30 | None | 225-236 | 203-225 | 0.92-0.96 | 8-11 | 32-33 | - | - |
| Mean of 5[a] | 30 | None | 228.4 | 214.2 | 0.948 | 9.7 | 32.3 | - | - |
| Range of 3[a] | 30 | $1 \times 10^{17}$ | 222-225 | 211-215 | 0.95-0.96 | 11 | 31 | - | 17-18 |
| Mean of 3[a] | 30 | $1 \times 10^{17}$ | 223.3 | 213.0 | 0.953 | 11.0 | 31.0 | - | 18 |
| 8 Aa 49 | 30 | $1 \times 10^{18}$ | 268.0 | 262.1 | 0.98 | 6 | 22 | - | 22 |
| 8 Aa 55 | 30 | $1 \times 10^{18}$ | 270.9 | 263.1 | 0.97 | 6 | 25 | 359 | 23 |
| 8 Aa 60 | 30 | $1 \times 10^{18}$ | 252.6 | 250.0 | 0.99 | 6 | 27 | 347 | 22 |
| Mean | 30 | $1 \times 10^{18}$ | 263.8 | 258.4 | 0.980 | 6.0 | 24.7 | 353.0 | 22 |

(a) From screening program (ref. 1)

(#) For comparison purposes only

(b) Previously reported (ref.8)

\- Not determinable

TABLE 8 TENSILE TEST RESULTS, TITANIUM 5 Al - 2.5 Sn (Std. 1), Annealed

| Specimen | Temp. (°R) | Irradiation ($n/cm^2$ E>0.5 MeV) | $F_{tu}$ (Ksi) | $F_{ty}$ 0.2% offset (Ksi) | $F_{ty}/F_{tu}$ | Elongation in 0.5 in (4D) (%) | Reduction of Area | Fracture Stress (Ksi) | Modulus (# ($10^3$ Ksi) |
|---|---|---|---|---|---|---|---|---|---|
| Range of 5 (a) | 540 | None | 124-127 | 112-121 | 0.90-0.98 | 23-24 | 48-52 | - | 12-16 |
| Mean of 5 (a) | 540 | None | 125.2 | 114.8 | 0.920 | 23.3 | 50.7 | - | 15 |
| Range of 5 (a) | 30 | None | 213-231 | 200-215 | 0.87-0.95 | 12-18 | 21-35 | 298 | 18-18 |
| Mean of 5 (a) | 30 | None | 224.8 | 205.2 | 0.915 | 13.8 | 30.0 | 298 | 18 |
| Range of 3 (a) | 30 | $1 \times 10^{17}$ | 222-257 | 211-231 | 0.82-0.97 | 9-14 | 36 | - | 15-21 |
| Mean of 3 (a) | 30 | $1 \times 10^{17}$ | 239.0 | 218.0 | 0.913 | 11.5 | 36 | - | 18 |
| 3 Aa 64 (x) | 30 | $5 \times 10^{17}$ | 254.9 | - | - | 8 | 29 | 360.0 | - |
| 3 Aa 71 (x) | 30 | $8.5 \times 10^{17}$ | 240.8 | 231.6 | 0.96 | 6 | 25 | 322.9 | 20 |
| 3 Aa 63 | 30 | $1 \times 10^{18}$ | 289.2 | 279.9 | 0.97 | 6 | 21 | 368.2 | 25 |
| 3 Aa 73 | 30 | $1 \times 10^{18}$ | 211.8 | 205.3 | 0.97 | 8 | 34 | 322.0 | 20 |
| 3 Aa 167 | 30 | $1 \times 10^{18}$ | 250.8 | 248.2 | 0.99 | 7 | 27 | 342.1 | 16 |
| Mean | 30 | $1 \times 10^{18}$ | 250.6 | 244.5 | 0.976 | 7.0 | 27.3 | 344.1 | 20 |

(a) From screening program (ref. 1)

(x) Irradiation terminated prematurely by irrecoverable reactor scram

(#) For comparison purposes only

\- Not determinable

TABLE 9

TENSILE TEST RESULTS, TITANIUM 6 AL - 4V (ANNEALED) [b]

| Specimen | Temp. (°R) | Irradiation ($n/cm^2$, E>0.5 MeV) | $F_{tu}$ (Ksi) | $F_{ty}$ 0.2% offset (Ksi) | $F_{ty}/F_{tu}$ | Elongation in 0.5 in (4D) (%) | Reduction of Area-(%) | Fracture Stress | Modulus(#) ($10^3$ Ksi) |
|---|---|---|---|---|---|---|---|---|---|
| Range of 5 (a) | 540 | None | 142-145 | 134-141 | 0.94-0.97 | 13-14 | 42-48 | - | 14-15 |
| Mean of 5 (a) | 540 | None | 144.0 | 137.8 | 0.957 | 13.8 | 45.0 | - | 15 |
| Range of 5 (a) | 30 | None | 249-265 | 228-255 | 0.87-0.97 | 7-10 | 27-36 | - | 17-17 |
| Mean of 5 (a) | 30 | None | 260.4 | 243.2 | 0.934 | 7.6 | 30.4 | - | 17 |
| Range of 3 (a) | 30 | $1 \times 10^{17}$ | 265-290 | 254 | 0.95 | 5-6 | 37-38 | - | - |
| Mean of 3 (a) | 30 | $1 \times 10^{17}$ | 273.7 | 254 | 0.95 | 5.7 | 37.3 | - | - |
| 2 Ac 54 | 30 | $1 \times 10^{18}$ | 302.7 | 289.4 | 0.96 | 4 | 34 | 456 | 20 |
| 2 Ac 55 | 30 | $1 \times 10^{18}$ | 332.9 | 314.5 | 0.95 | 4 | 34 | 506 | 25 |
| 2 Ac 56 | 30 | $1 \times 10^{18}$ | 325.0 | 294.7 | 0.91 | 6 | 34 | 494 | 32 |
| Mean | 30 | $1 \times 10^{18}$ | 320.2 | 299.5 | 0.940 | 4.7 | 34.0 | 485.3 | 26 |

(a) From screening program (ref. 1)
(b) Previously reported (ref. 8)
(#) For comparison purposes only

\- Not determinable

TABLE 10 PARTIAL FATIGUE TEST RESULTS, TITANIUM 55A-ANNEALED
(Axial Load; Test Ratio = -1; 15 cpm)
Tested at 540°R, No Irradiation

| STRESS Ksi | STRESS $\%F_{tu}$* | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 67.0 | 100 | 1 Aa 275 | 1643 |
| | | 1 Aa 274 | 1476 |
| | | 1 Aa 276 | 1395 |
| 63.6 | 95 | 1 Aa 240 | 4945 |
| | | 1 Aa 241 | 2905 |
| | | 1 Aa 264 | 2006 |
| 60.3 | 90 | 1 Aa 263 | 3764 |
| | | 1 Aa 223 | 3198 |
| | | 1 Aa 273 | 1498 |
| 57.0 | 85 | 1 Aa 242 | 6873 |
| | | 1 Aa 239 | 6587 |
| 55.3 | 82 1/2 | 1 Aa 277 | 10000 (not failed) |
| | | 1 Aa 278 | 9051 |
| 52.3 | 78 | 1 Aa 238 | 10000 (not failed) |
| 50.3 | 75 | 1 Aa 220 | 10000 (not failed) |
| | | 1 Aa 224 | 10000 (not failed) |

* Mean at 540°R, unirradiated (ref. 1)

TABLE 11 PARTIAL FATIGUE TEST RESULTS, TITANIUM 55A - ANNEALED (Axial Load; Test Ratio = -1; 15 cpm except as noted) Tested at 30°R, No Irradiation

| STRESS Ksi | $\%F_{tu}$* | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 160.9 | 95 | 1 Aa 256 | 1619 |
| | | 1 Aa 267 | 1260 |
| | | 1 Aa 268 | 903 |
| 152.5 | 90 | 1 Aa 248 | 3589 |
| | | 1 Aa 288 | 3458 (30 cpm) |
| | | 1 Aa 286 | 3234 (6 cpm) |
| | | 1 Aa 282 | 2265 (6 cpm) |
| | | 1 Aa 261 | 2224 (6 cpm) |
| | | 1 Aa 250 | 1322 |
| | | 1 Aa 287 | 1261 (30 cpm) |
| | | 1 Aa 266 | 1001 |
| 144.0 | 85 | 1 Aa 265 | 5576 |
| | | 1 Aa 257 | 4035 |
| | | 1 Aa 255 | 3725 |
| 139.8 | 82 1/2 | 1 Aa 269 | 5912 |
| 135.5 | 80 | 1 Aa 249 | 10000 (not failed) |
| 127.1 | 75 | 1 Aa 247 | 10000 (not failed) |

* Mean at 30°R, unirradiated (ref. 1)

TABLE 12 PARTIAL FATIGUE TEST RESULTS, TITANIUM 55A, ANNEALED
(Axial Load; Test Ratio = -1; 15 cpm)
Tested at 30°R Following Irradiation to $10^{17}$ n/$cm^2$ (E>0.5 MeV) at 30°R

| STRESS Ksi | $\%F_{tu}$* | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 169.4 | 100 | 1 Aa 290 | 2740 |
| | | 1 Aa 260 | 799 |
| | | 1 Aa 279 | 740 |
| 160.9 | 95 | 1 Aa 280 | 2401 |
| | | 1 Aa 253 | 1748 |
| | | 1 Aa 271 | 1568 |
| 152.5 | 90 | 1 Aa 270 | 5498 |
| | | 1 Aa 244 | 3436 |
| | | 1 Aa 252 | 2329 |
| 144.0 | 85 | 1 Aa 258 | 6562 |
| | | 1 Aa 245 | 4279 |
| | | 1 Aa 251 | 2964 |
| 135.5 | 80 | 1 Aa 259 | 10000 (not failed) |
| | | 1 Aa 289 | 10000 (not failed) |
| | | 1 Aa 291 | 10000 (not failed) |

* Mean at 30°R, unirradiated (ref. 1)

TABLE 13 PARTIAL FATIGUE TEST RESULTS, TITANIUM 55A, ANNEALED
(Axial Load; Test Ratio = -1, 15 cpm)
Tested at 30°R During Irradiation

| STRESS | | SPECIMEN | CYCLES TO FAILURE | NEUTRON FLUX | |
|---|---|---|---|---|---|
| Ksi | $\%F_{tu}$* | | | Rate** (1) | Accum. (2) |
| 152.5 | 90 | 1 Aa 254 | 2709 | 2.2 | 2.5 |
| 135.5 | 80 | 1 Aa 281 | 5762 | 2.1 | 5.2 |

* Mean at 30°R, unirradiated (ref. 1)

** Average over irradiation period

(1) $\times 10^{-12}$ n/cm$^2$/ Sec

(2) $\times 10^{-16}$ n/cm$^2$

TABLE 14 PARTIAL FATIGUE TEST RESULTS, TITANIUM 5 Al-2.5 Sn (ELI)
(Axial Load; Test Ratio = -1; 15 cpm)
Tested at 540°R, No Irradiation

| STRESS Ksi | STRESS $\%F_{tu}$* | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 121.3 | 96 | 8 Aa 63 | 446 |
| 113.8 | 90 | 8 Aa 70 | 764 |
| | | 8 Aa 69 | 557 |
| | | 8 Aa 71 | 394 |
| 107.4 | 85 | 8 Aa 72 | 2306 |
| | | 8 Aa 67 | 1991 |
| | | 8 Aa 73 | 1249 |
| 101.1 | 80 | 8 Aa 65 | 2928 |
| | | 8 Aa 68 | 2387 |
| | | 8 Aa 74 | 2036 |
| 98.0 | 77 1/2 | 8 Aa 75 | 5653 |
| | | 8 Aa 76 | 4674 |
| 94.8 | 75 | 8 Aa 64 | 10000 (not failed) |

* Mean at 540°R, unirradiated (ref. 1)

TABLE 15 PARTIAL FATIGUE TEST RESULTS, TITANIUM 5 Al-2.5 Sn (ELI)
(Axial Load; Test Ratio = -1; 15 cpm)
Tested at 30°R, No Irradiation

| STRESS Ksi | STRESS $\%F_{tu}$* | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 239.8 | 105 | 8 Aa 94 | 1180 |
| | | 8 Aa 85 | 593 |
| | | 8 Aa 92 | 433 |
| 228.4 | 100 | 8 Aa 93 | 1551 |
| | | 8 Aa 84 | 1374 |
| | | 8 Aa 88 | 1114 |
| 217.0 | 95 | 8 Aa 89 | 6502 |
| | | 8 Aa 87 | 4965 |
| | | 8 Aa 83 | 3147 |
| 205.6 | 90 | 8 Aa 86 | 10000 (not failed) |
| | | 8 Aa 91 | 10000 (not failed) |
| | | 8 Aa 82 | 7152 |
| 194.1 | 85 | 8 Aa 81 | 10000 (not failed) |

* Mean at 30°R, unirradiated (ref. 1)

TABLE 16 TITANIUM 5 Al-2.5 Sn (Std. I)
(Axial Load; Test Ratio = -1; 15 cpm)
Tested at 540°R, No Irradiation

| STRESS Ksi | STRESS $\%F_{tu}$ | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 128.3 | 102 1/2 | 3 Aa 95 | 327 |
| 125.2 | 100 | 3 Aa 90 | 949 |
| | | 3 Aa 91 | 875 |
| 122.1 | 97 1/2 | 3 Aa 86 | 1333 |
| 118.9 | 95 | 3 Aa 87 | 2132 |
| | | 3 Aa 85 | 2024 |
| | | 3 Aa 75 | 1937 |
| 112.7 | 90 | 3 Aa 80 | 3758 |
| | | 3 Aa 76 | 3756 |
| | | 3 Aa 77 | 3544 |
| 106.4 | 85 | 3 Aa 78 | 7724 |
| | | 3 Aa 82 | 6716 |
| | | 3 Aa 79 | 6066 |
| 103.3 | 82 1/2 | 3 Aa 89 | 7229 |
| | | 3 Aa 88 | 6968 |
| 100.2 | 80 | 3 Aa 84 | 10000 (not failed) |
| | | 3 Aa 83 | 10000 (not failed) |
| | | 3 Aa 93 | 8308 |

* Mean at 540°R, unirradiated (ref. 1)

TABLE 17 PARTIAL FATIGUE TEST RESULTS, TITANIUM 5 Al-2.5 Sn (Std. I)
(Axial Load; Test Ratio = -1; 15 cpm)
Tested at 30°R, No Irradiation

| STRESS Ksi | STRESS $\%F_{tu}$* | SPECIMEN | CYCLES TO FAILURE |
|---|---|---|---|
| 247.3 | 110 | 3 Aa 96 | 4272 |
| 224.8 | 100 | 3 Aa 92 | 10000 (not failed) |
| 202.3 | 90 | 3 Aa 97 | 10000 (not failed) |

* Mean at 30°R, unirradiated (ref. 1)

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# 8 FIGURES

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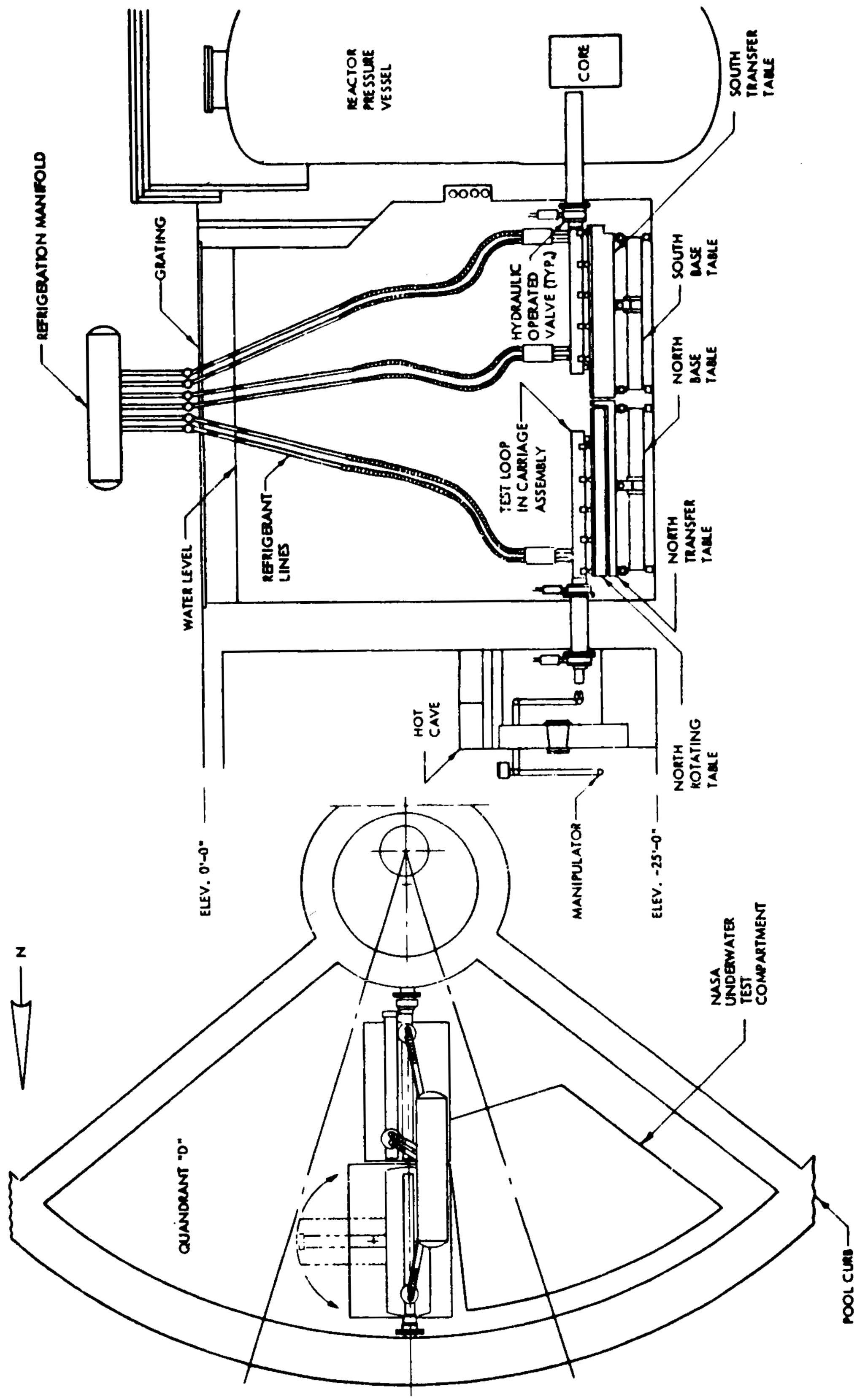



FIGURE 1 SYSTEM LAYOUT

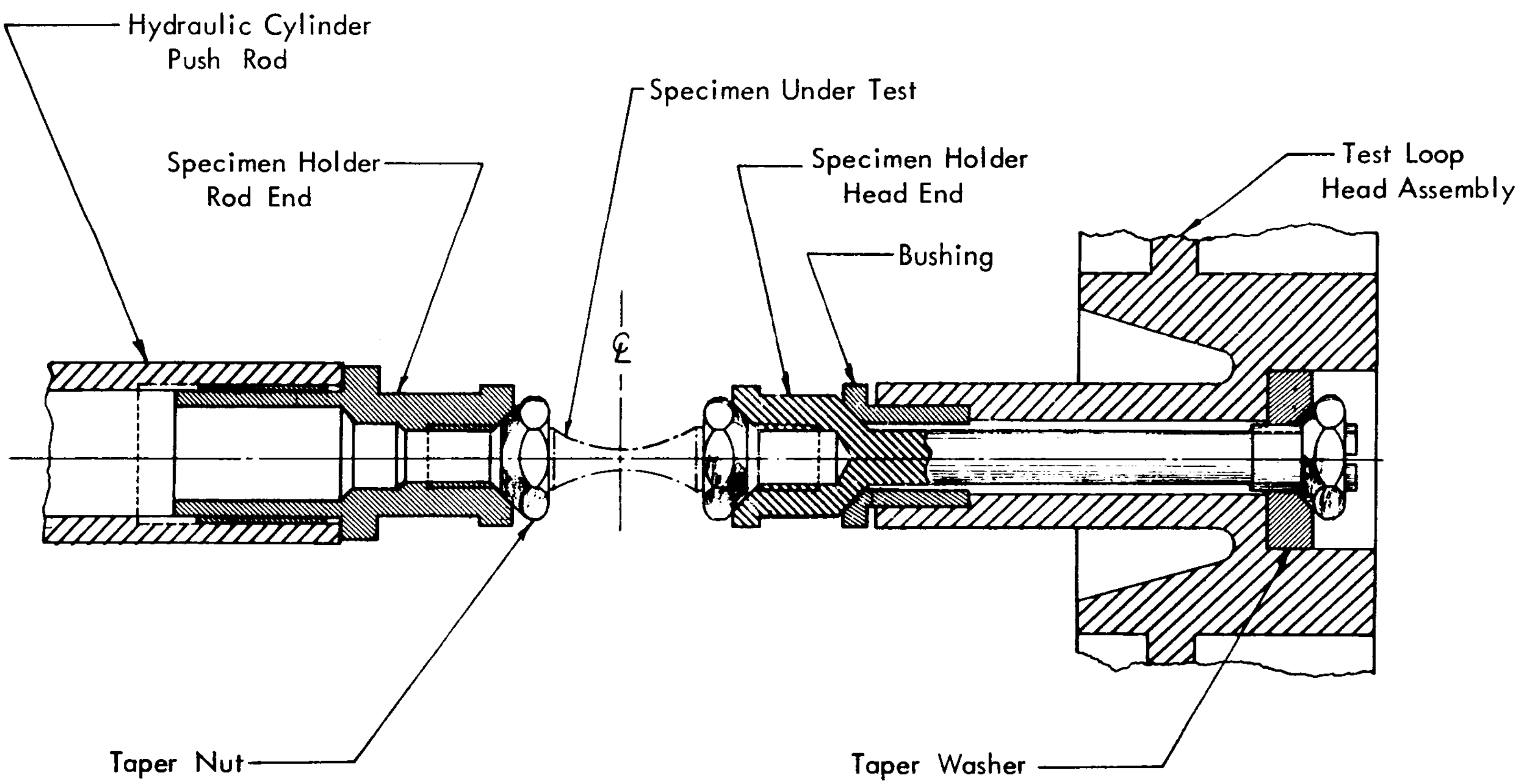



FIGURE 2 SPECIMEN HOLDER DESIGN

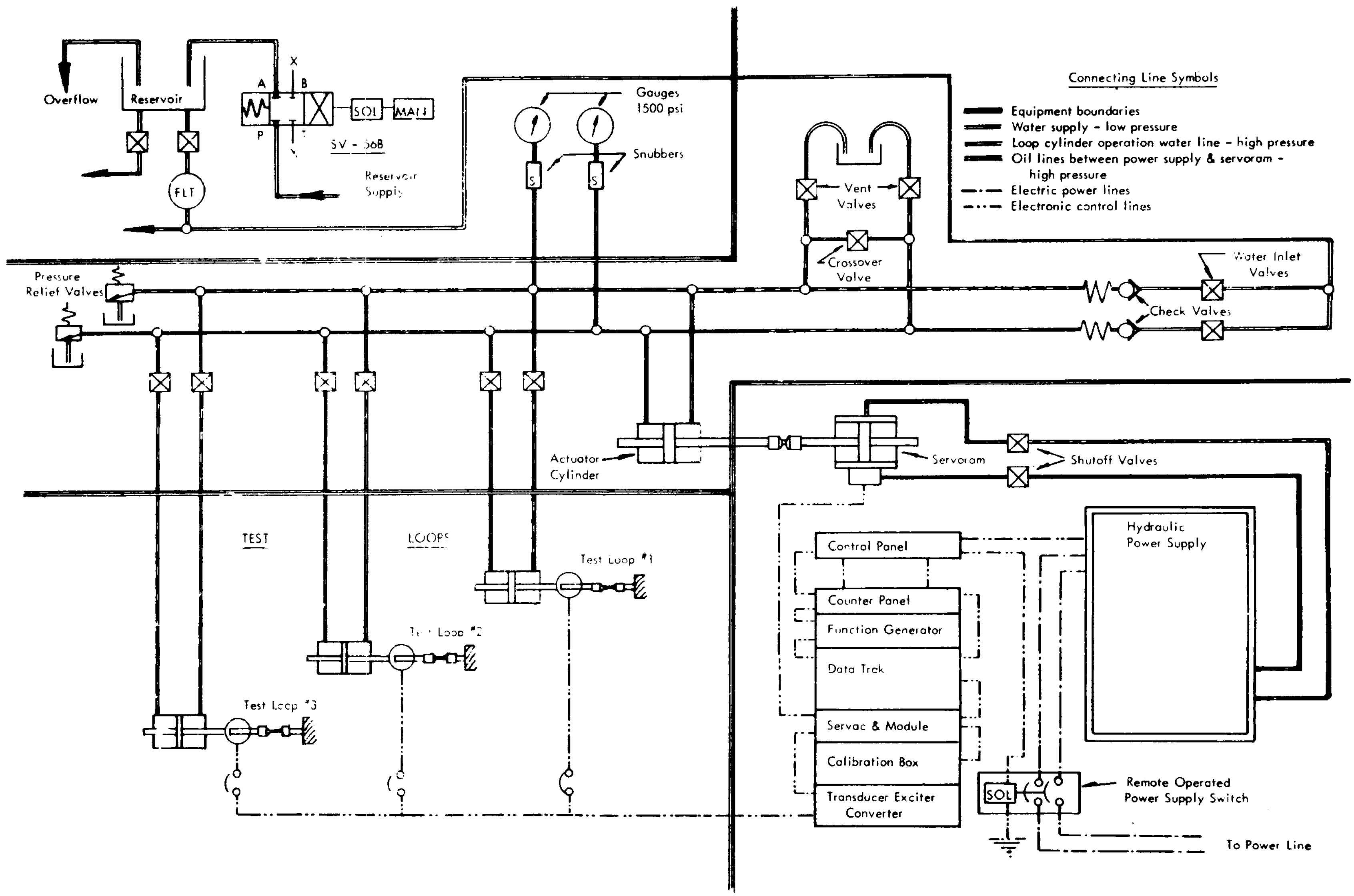
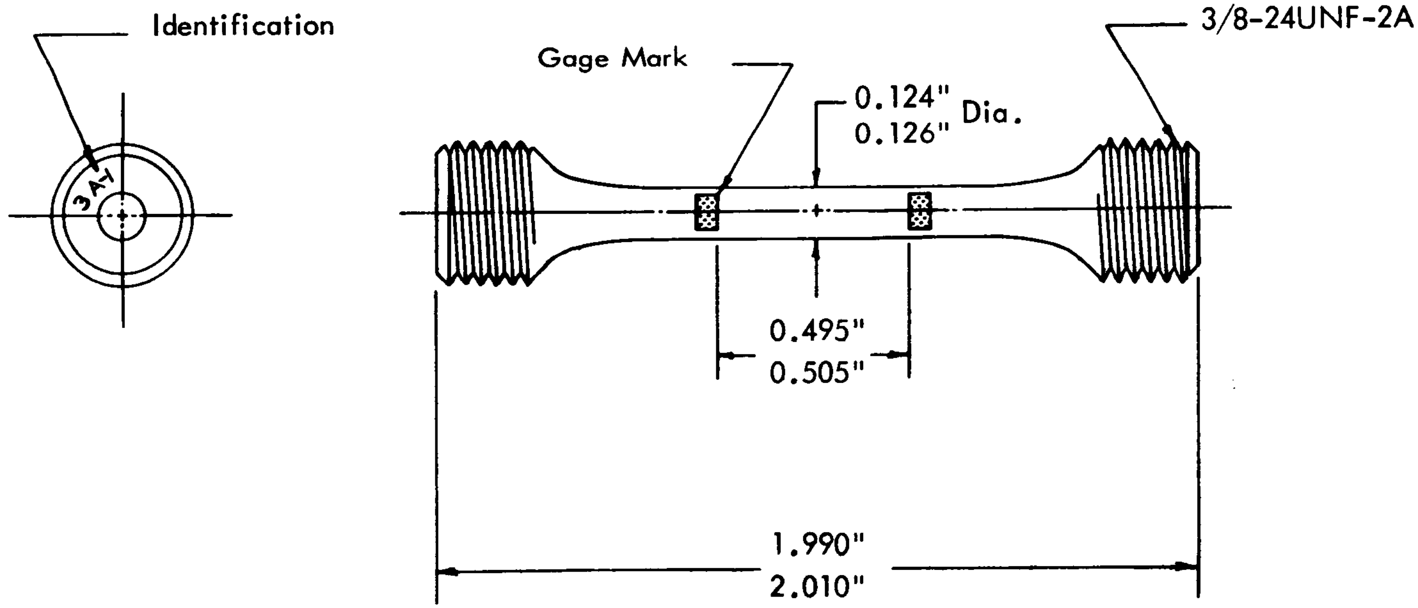



FIGURE 3 LOAD CONTROL SYSTEM (SCHEMATIC)




Note: Diameter at gage marks shall be center diameter + $\frac{0.002''}{0.004''}$.

FIGURE 4 TENSILE SPECIMEN

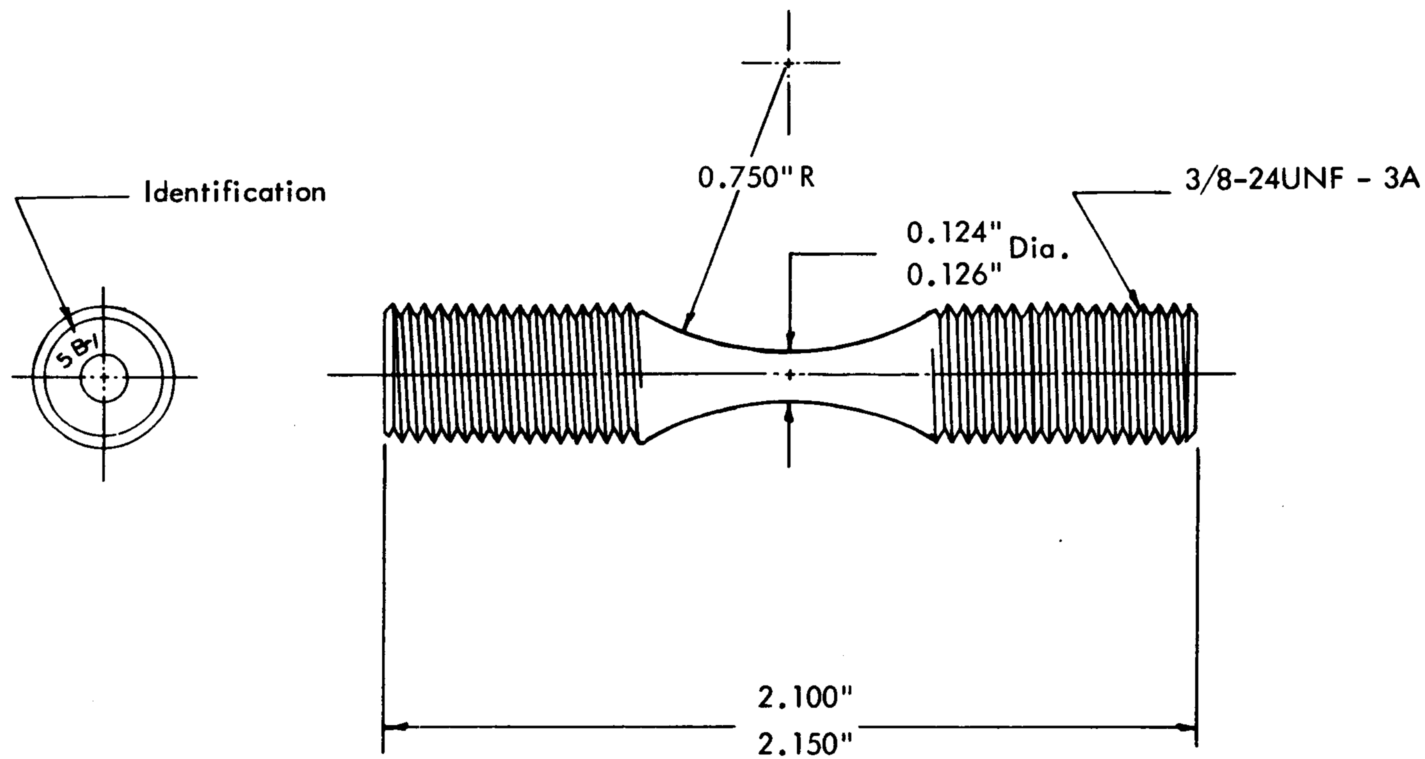



FIGURE 5 FATIGUE SPECIMEN

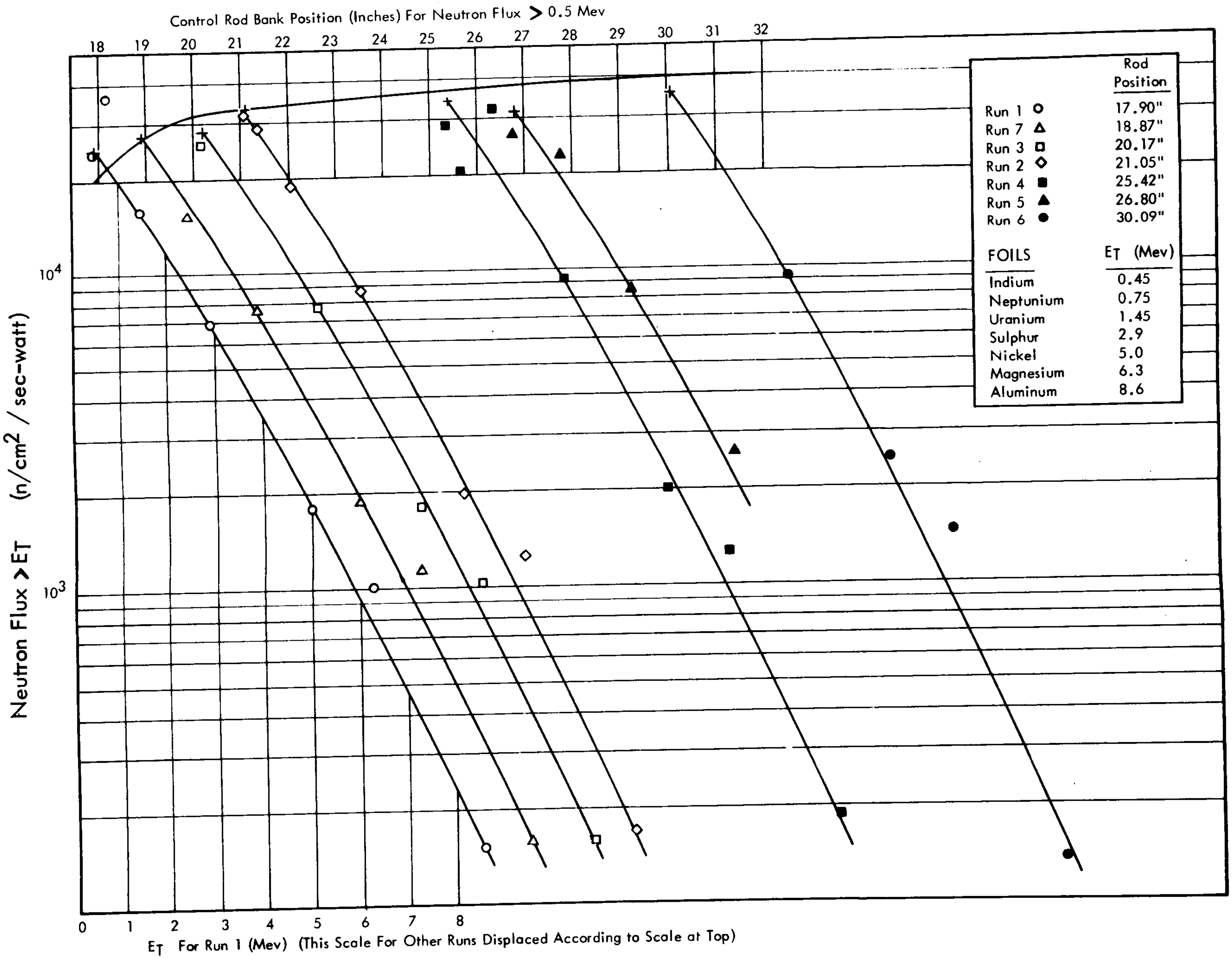



FIGURE 6. NEUTRON FLUX>$E_T$ VS. $E_T$ AND NEUTRON FLUX >0.5 MEV VS. CONTROL ROD BANK POSITION

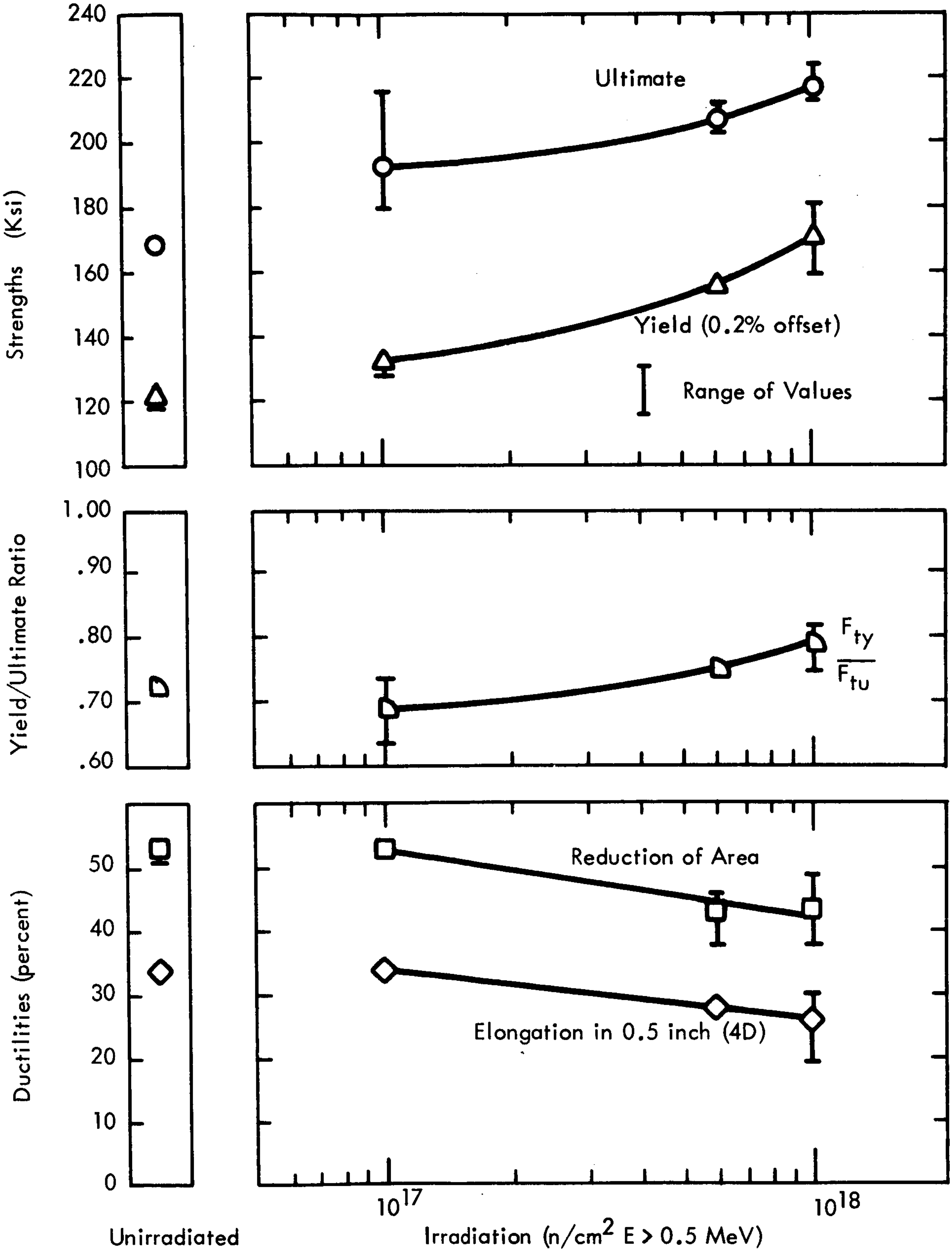



FIGURE 7 EFFECTS OF IRRADIATION AT 30°R ON TITANIUM 55A

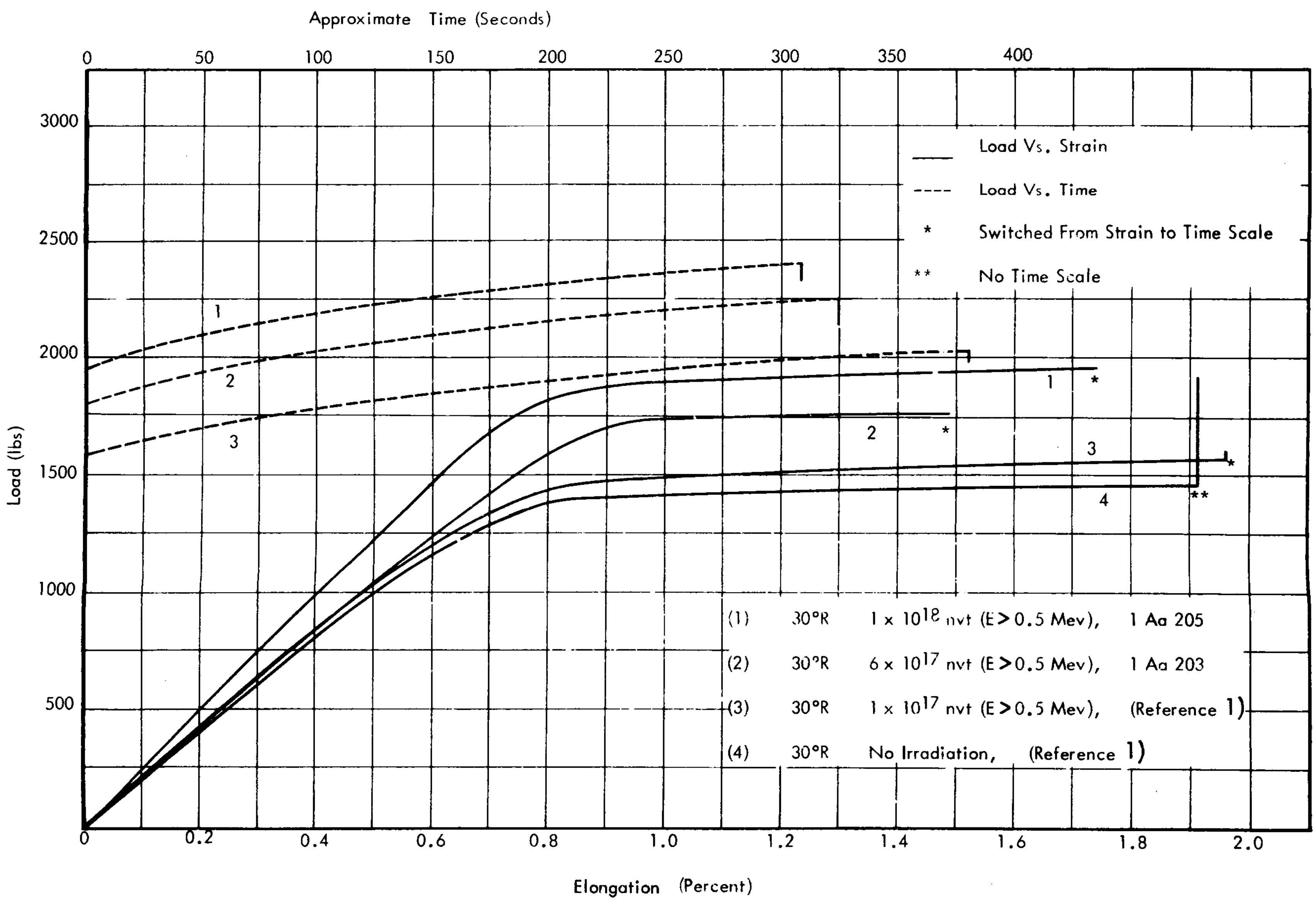



FIGURE 8 TYPICAL LOAD-ELONGATION CURVES FOR TITANIUM 55A

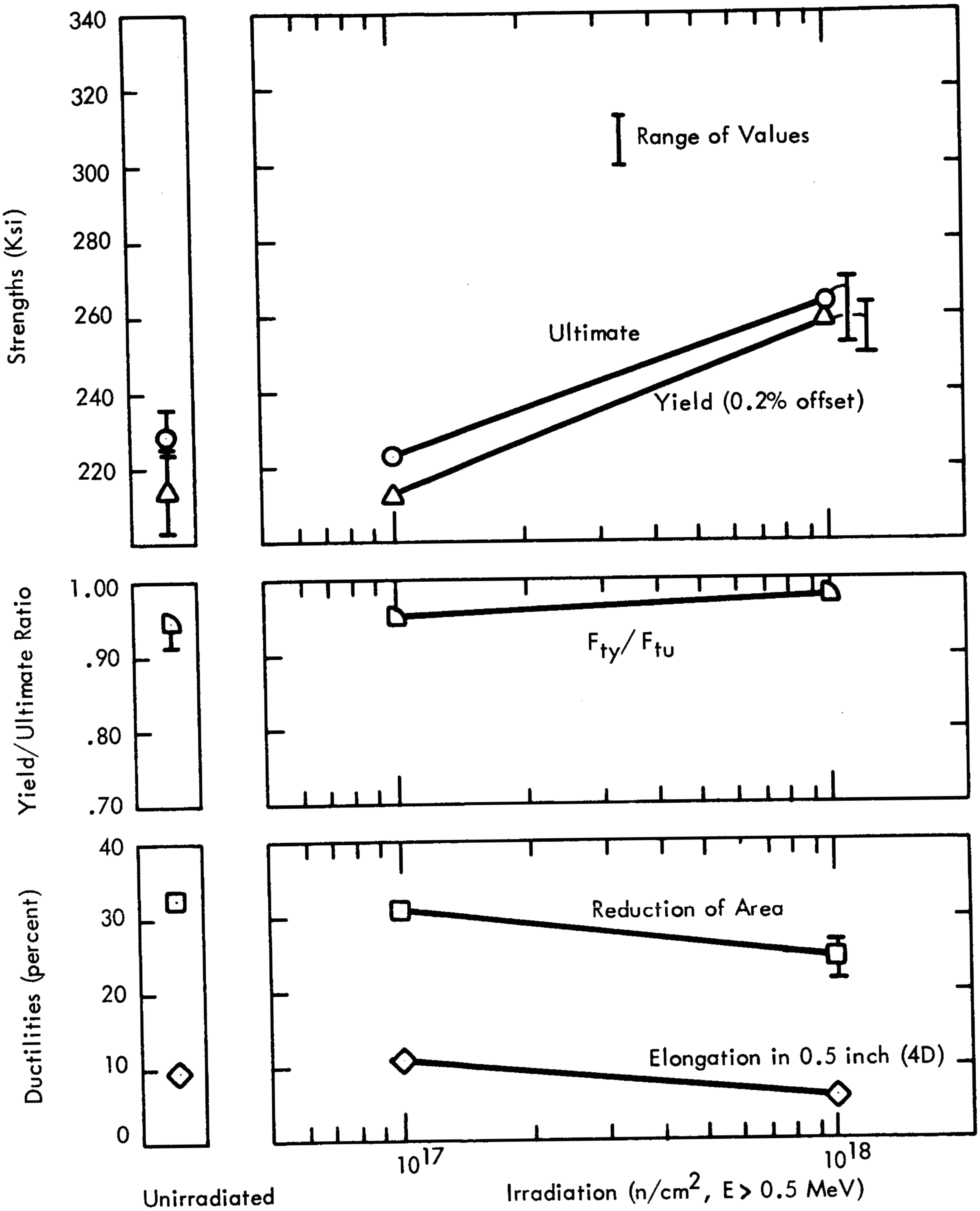



FIGURE 9 EFFECTS OF IRRADIATION AT 30°R ON TITANIUM 5 Al-2.5 Sn (ELI)

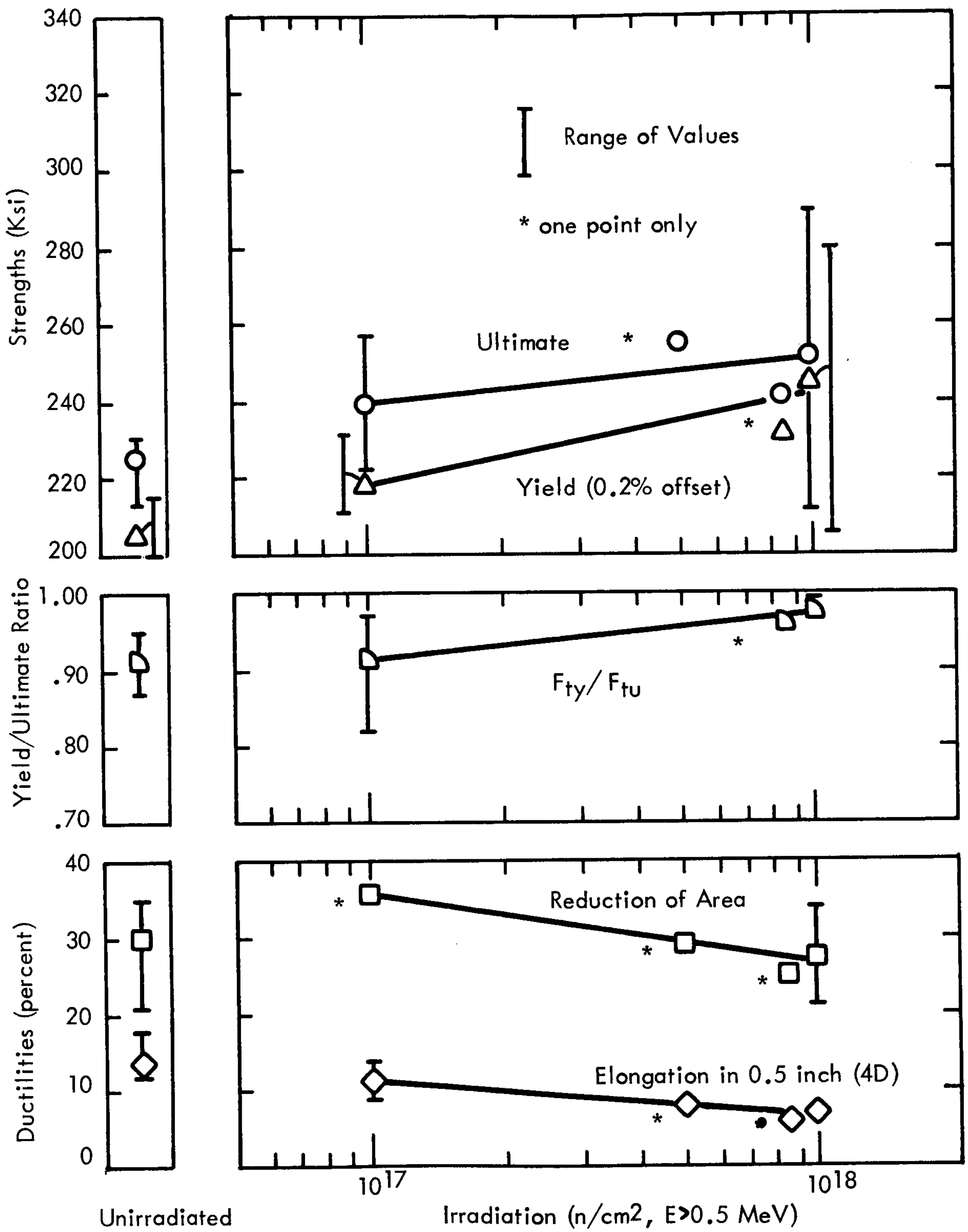



FIGURE 10 EFFECTS OF IRRADIATION AT 30°R ON TITANIUM 5 Al-2.5 Sn (Std. I)

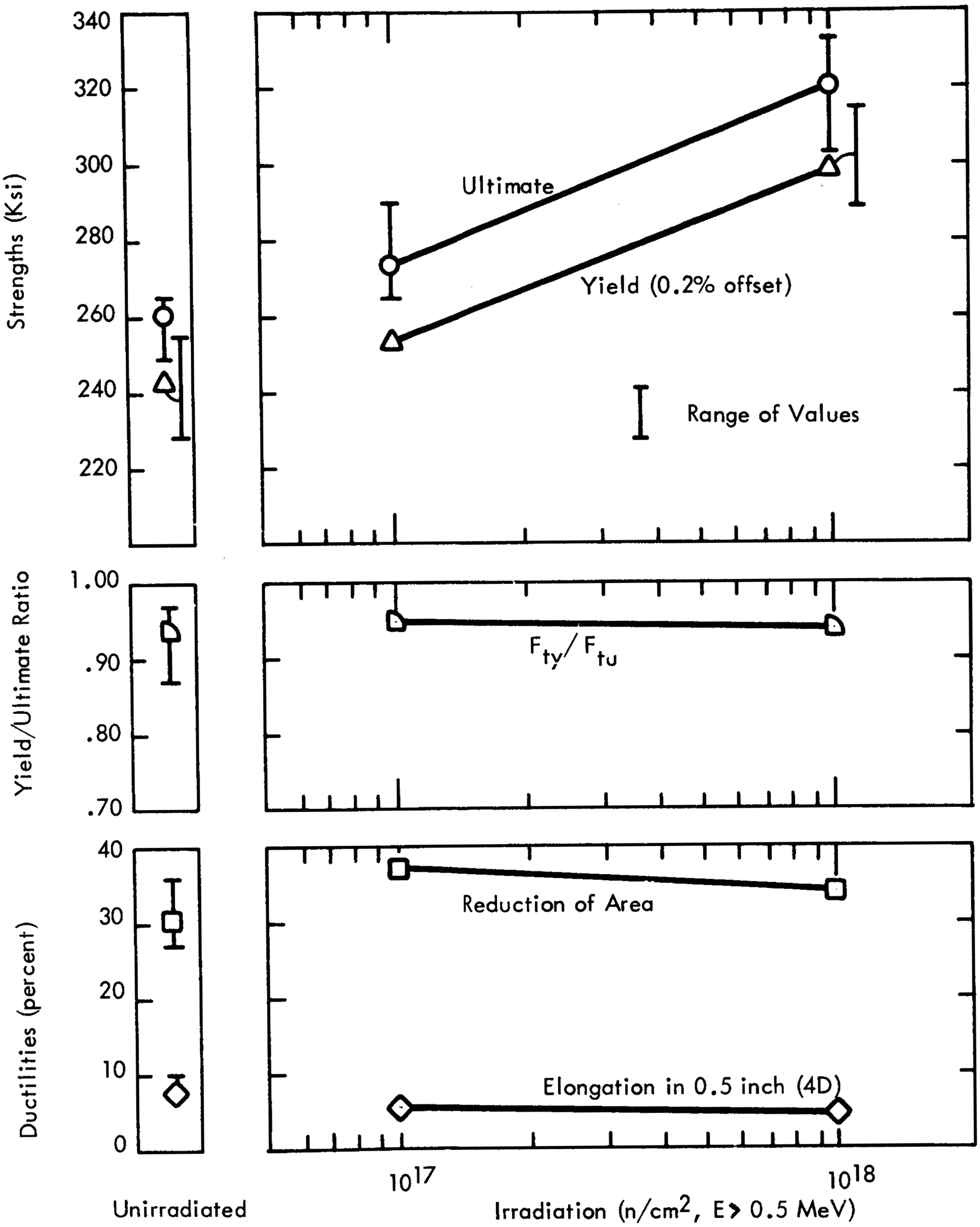



FIGURE 11 EFFECTS OF IRRADIATION AT 30°R ON TITANIUM 6 Al-4V (ANNEALED)

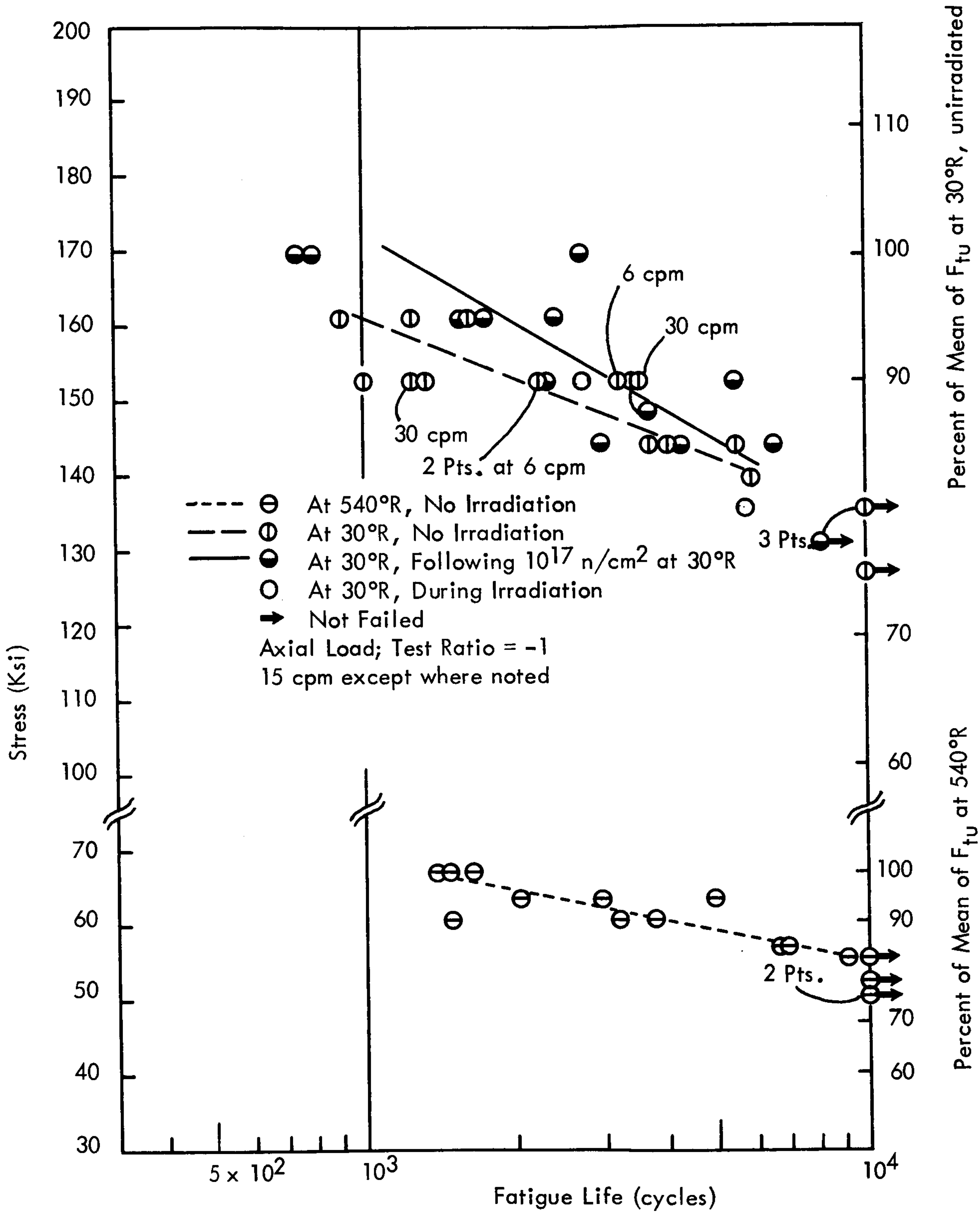



FIGURE 12 LOW-CYCLE FATIGUE STRENGTH OF TITANIUM 55A AT VARIOUS TEST CONDITIONS

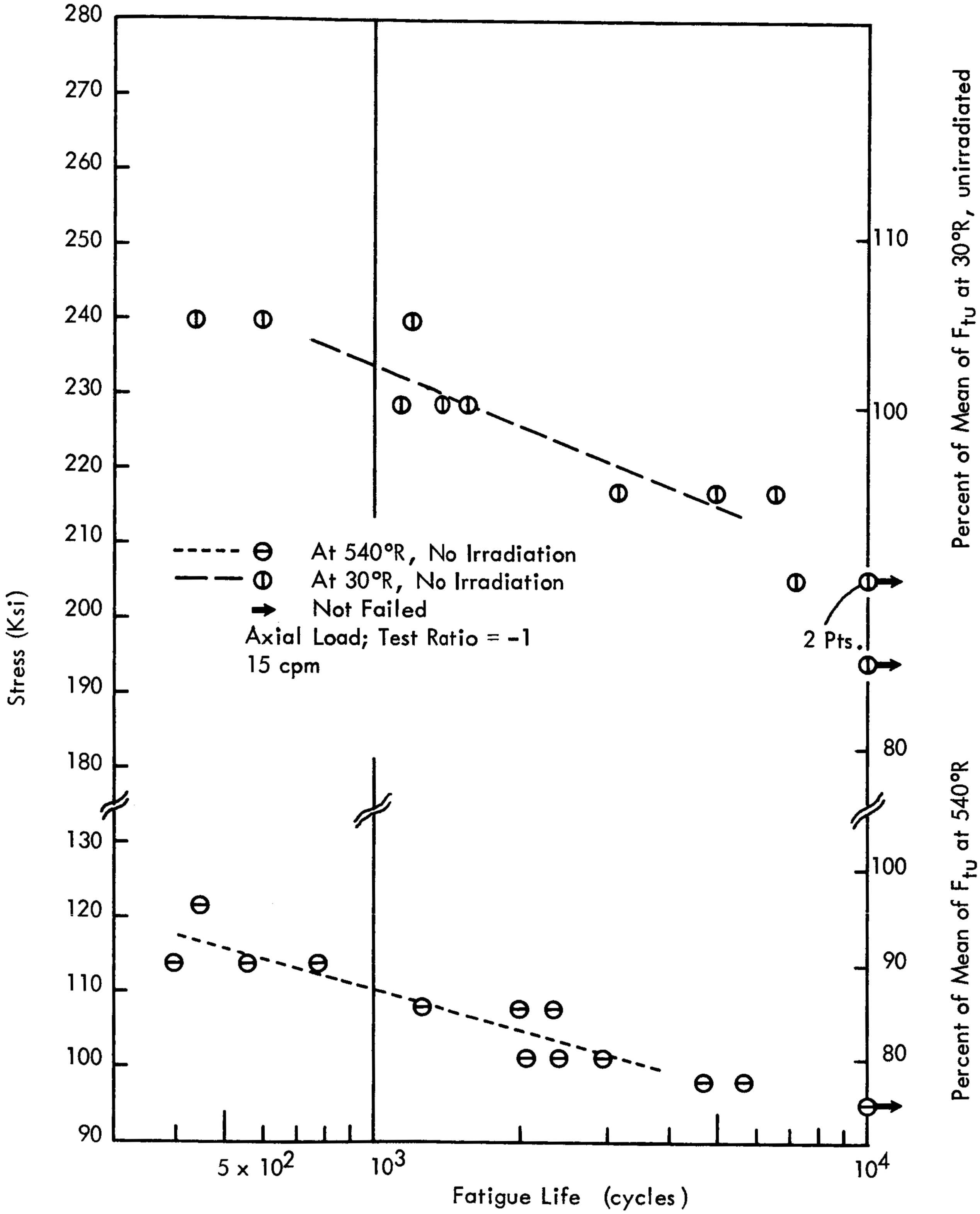



FIGURE 13 LOW-CYCLE FATIGUE STRENGTH OF TITANIUM 5 Al-2.5 Sn (ELI) AT VARIOUS TEST CONDITIONS

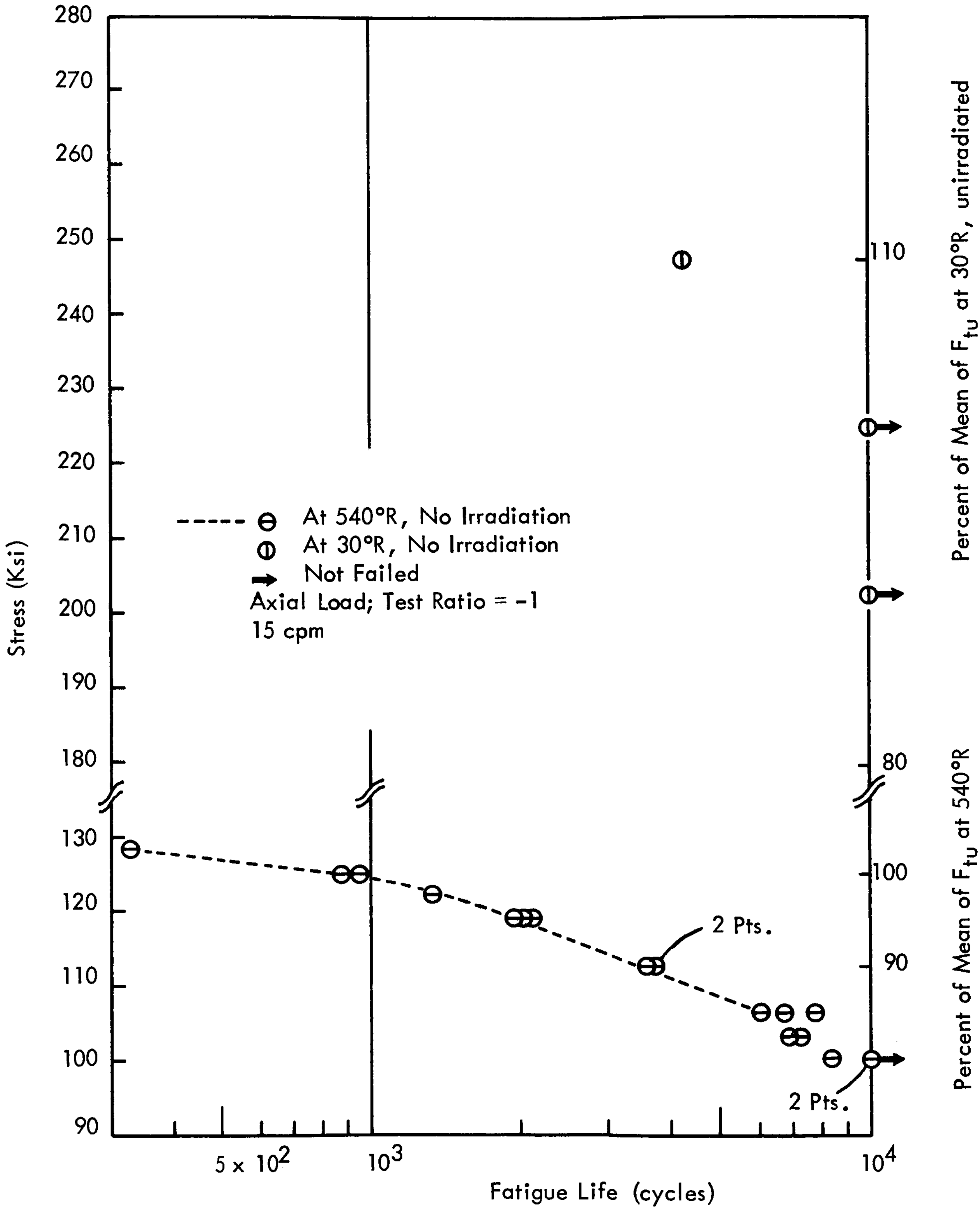



FIGURE 14 LOW-CYCLE FATIGUE STRENGTH OF TITANIUM 5 Al-2.5 Sn (Std. I) AT VARIOUS TEST CONDITIONS

APPENDIX A

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## ABSTRACT

This is the fifth Quarterly Report summarizing, to date, studies (under Contract NAS 3-7985) of the effects of nuclear radiation on materials at cryogenic temperatures. The test data herein reported include the effects of $10^{18}$ n/cm$^2$ ($E > 0.5$ MeV) at 30°R on the tensile properties of Titanium 55A, Titanium 5 Al-2.5 Sn (ELI), Titanium 5 Al-2.5 Sn (Std. I) and Titanium 6 Al-4V (annealed) and the effects of $10^{17}$ n/cm$^2$ at 30°R on the low-cycle fatigue life of Titanium 55A.