THE EFFECT OF TEMPERATURE CYCLING ON THE PHYSICAL PROPERTIES OF FS-1 MAGNESIUM ALLOY SHEET METAL

Thesis by

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In Partial Fulfillment of the Requirements

For the Degree of

Aeronautical Engineer

California Institute of Technology

Pasadena, California

1953

ACKNOWLEDGEMENTS

The author wishes to acknowledge and to express his gratitude to his supervising committee, Drs. E. E. Sechler, D. S. Clark, and M. L. Williams, Jr., for their guidance and many helpful suggestions; to his co-worker, Lt. Winthrop P. Robinson, USN, for his cooperation; to Mr. Milton Wood for his assistance in carrying out the investigation; to Mr. Marvin Jessey for his assistance with the electrical problems; to Mr. C. A. Bartsch and his shop staff for their cooperation and skillful machine work; to Mrs. Betty Wood for her assistance in the presentation of the data; to Mrs. Virginia Boughton for the typing of the thesis, and to his wife for her patience and understanding throughout the preparation of this thesis.

ABSTRACT

An investigation was made of the effect of different cyclic heat treatments on the "high rate" creep and stress-strain characteristics of FS-1h magnesium alloy and 75S-T6 aluminum alloy sheet metal. The heat treatments to which the alloys were subjected consisted of a varying series of heating and cooling cycles such that the periods at heat treating temperature totaled 60 minutes. Temperatures investigated were 250° and 550°F. Following heat treatment, both tensile and creep tests were conducted at room temperature and at the temperature of heat treatment.

This thesis encompasses the investigation of FS-1h magnesium alloy, while the investigation of 75S-T6 aluminum alloy is covered in the current thesis of the author's co-worker[1].

It was found for the range of heat treatment cycling (1-20 cycles) and temperatures investigated, that there is no effect on the physical properties as a result of changing the number of heating cycles. Cyclic heat treatment at 550°F was the only treatment to result in a reduction of physical properties, and this only when tested at room temperature.

It was also found that, within the time range investigated, exposure time exhibits a negligible effect on the physical properties of the material. However, elevated temperatures affect the yield and ultimate properties.

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I. INTRODUCTION

Due to the high speeds being attained today by airplanes and missiles and the higher speeds foreseen for the near future, temperature effects are becoming a major factor which must be taken into account when considering the physical properties of the materials to be used.* Besides aerodynamic heating there are also structural heating effects to be considered from gas turbine and after-burner power plant installations. From the operational nature of aircraft, it can be seen that, in service, structural materials will be subjected to temperatures which cycle between comparatively wide limits, with the lower limit being the lowest atmospheric temperature expected. Over the useful life of the structure it can be expected to be subjected to many periods of alternate heating and cooling. The effect of heat on the structural elements subjected to a combination of high temperature and high working stresses must be known in order to design minimum weight structures. The most advantageous use of structural materials is accomplished when the load-carrying ability of the material being considered is known under the service conditions involved.

Past investigations on the effects of varying temperature have dealt with high temperature materials. Smith(2) brings together the results of the research work on metals at elevated temperatures, and References 3, 4, and 5 compile recent data on "short time" creep properties of many aircraft materials, but at the present time there seems to be little experimental work on cycling temperature effects

* At sea level the temperature rise (degrees F) is in the order of $(1.7 \times 10^{-4}) \times (V, \text{mph})^2$.

for light alloys.

The purpose of the investigation has been to determine the effects on the physical properties of FS-1h magnesium alloy sheet metal of different cycling heat treatments at 250° and 550°F. Tensile and creep tests were made on specimens which had been subjected to these heat treatments and comparisons made of moduli of elasticity, 0.2 per cent offset yield strengths, minimum creep rates, and tensile strengths where possible, at room temperature and at the temperature of heat treatment.

This investigation was conducted at the Guggenheim Aeronautical Laboratory of the California Institute of Technology, during the school year, 1952-53, under the supervision of Dr. E. E. Sechler.

II. MATERIALS FOR TEST

The material tested was commercial sheet, FS-1h* magnesium alloy 0.102 inches thick. The specimens were conventional sheet stock tensile specimens eight inches long by one inch wide with a reduced gage section one-half inch wide by approximately five inches from shoulder to shoulder. These details are shown in Figure 1. Creep and strain measurements were made over a three-inch gage length at the center of the reduced section. The specimen strips were sheared from the original sheet such that the longitudinal axis was parallel to the rolling direction of the sheet. The final contour was achieved by a milling machine operation. To decrease the transient heating and cooling time, the specimens were painted with a black, heat-resisting paint from shoulder to shoulder of the reduced section.

The tensile stress-strain curves for this material in the "as received" condition at room and elevated temperatures are given in Figure 2 with a curve of the properties obtained plotted against temperature shown in Figures 3 and 4. The "short time" creep curves for this material in the "as received" condition at room and elevated temperatures are given in Figure 5. The stress parameters for the creep curves are the stresses chosen for which cyclic heating effects are to be compared. The physical properties obtained from these curves are tabulated in Table I.

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* Dow Chemical Company designation.

III. EQUIPMENT

Three separate but similar furnaces were used for obtaining the required elevated temperatures for testing and heat treating the specimens. These were the same furnaces used in the investigations described in detail in Reference 4. Photographs of the furnaces appear in Figures 6, 7, and 8. Three basic furnace installations were required; heat treatment, tensile test, and creep test.

The heat treatment furnace installation is shown in the photograph of Figure 6. The furnace was mounted on top of a steel framework and was charged through a three-inch diameter hole in the bottom. The specimens to be heat treated were mounted on an extension to the piston rod of a three-inch diameter pneumatic cylinder mounted in a vertical position below the furnace. The furnace was charged with four specimens at one time, these being mounted in a light frame having a minimum of surface contact. Details of this mounting are shown in the photograph of Figure 9. Automatic positioning of the charging piston was obtained by two electrical air valves controlled by a tandem recycling timer. Power to the timer was controlled by an electric timer switch which enabled the cycling process to be terminated after any desired period of time. A counter actuated upon withdrawal of the specimens from the furnace was employed as a check on the actual number of heating cycles passed through. Temperature control was obtained by the use of an automatic controlling pyrometer with the controlling thermocouple placed between the heating coils at the mid-height of the furnace.

The tensile test furnace installation is shown in Figure 7. A

3000-pound compound-lever testing machine was modified to permit placement of the oven between the uprights for tensile testing at elevated temperatures.

The creep test installation is shown in Figure 8. The testing machine was a dead weight, single-lever type machine described in detail in Reference 4. This installation was used without change with the exception of a modification of the specimen grips. In both the tensile and creep furnace installations, temperature control was obtained by use of the same type of automatic controlling pyrometer used for the heat treatment furnace. The jaws used to grip the specimens in both the tension and creep tests are shown in Figures 10 and 11.

Creep strains at room and elevated temperatures and tensile strains at elevated temperatures were measured by means of two eight-power telescopes, permanently mounted four inches apart on the side of the furnaces as described in Reference 4. The light source, introduced through a hole centered between the telescopes, was a high intensity, six-volt bulb.

The telescopes were focused on two separate scales which were mounted on the specimen. These scales (1/100-inch divisions) were attached to spring clips which clipped to the specimen at points three inches apart, giving a three-inch gage length. Details of the scales are shown in Figure 12. The relative movement between the two scales, measured by the movement of each scale past a fixed hair line in the telescopes, determined the deformation of the specimen. The eight-power magnification of the scales permitted estimates to one-tenth of the smallest division making possible measurement of strains in the order of 0.0003 in./in.

Tensile strains at room temperature were measured by means of Huggenberger extensometers, with one mounted on each side of the specimen to permit an averaged strain reading. By estimating gage readings to one-tenth of the scale divisions, strains could be measured in the order of 1.66×10^{-5} in./in.

Due to interference of the specimen jaws with the end heating coils when assembled in the tensile test furnace, and to the restricted travel of the loading lever arm of the creep machine, the maximum total elongation possible was not sufficient to carry the elevated temperature tensile tests or the creep tests to rupture.

IV. EXPERIMENTAL TECHNIQUE

Heat Treatment

The heat treatment furnace was charged with four specimens at one time. The furnace was calibrated by checking temperature distribution over the specimens using thermocouples at each end and in the center of the specimen gage section. It was found that only with the furnace power control on the low setting could a satisfactory minimum temperature variation and gradient be obtained. The maximum temperature gradient measured over the gage width section on the surface of any specimen at heat treating temperatures was 2°F, while the maximum variation in temperature was 2°F. Prior to loading the specimens and charging the furnace, the furnace was heated for approximately one hour with the piston rod extension and loading frame inside the furnace. The heating and cooling curves for the heat treatment cycle are shown in Figures 13 and 14. Transient heating time was considered as that time necessary to bring the specimens up to within 2 per cent of the desired temperature. During the cooling part of the cycle the specimens were cooled to 100°F.

The following heat treatment cycles were used, with the total time at heat treating temperature being 60 minutes in each case:

Treatment	Time During Cycle at Heat Treatment Temperature	Number of Cycles
(a)	60 minutes	1
(b)	12 minutes	5
(c)	5 minutes	12
(d)	3 minutes	20

Tensile Tests

Due to the large mass of metal in the jaws gripping the specimen, difficulty was encountered in holding to a minimum the temperature gradient across the gage section. Lowest temperature gradients were obtained with the heat control on low. At the top and bottom of the furnace there were small heating coils mounted in each half which were wired separately from the main heating coils. Power to these coils was controlled by means of a Variac and a selection between parallel or series wiring, thus making possible some control over the heat distribution through the furnace. At 550°F these four coils were wired in parallel, while at 250°F only the bottom coils were used, and these in series.

Prior to each test the furnace was brought up to test temperature with a dummy specimen mounted in the jaws and held for approximately 30 minutes in order to heat up the jaws and furnace. The jaws and adaptors were then removed from the furnace and by means of an assembly fixture the specimen to be tested was secured in the hot jaws. This made possible an accurate alignment of the specimen, jaws and adaptor, permitted a lower temperature gradient, and reduced the heating time required. The scales were clipped to the specimen at the proper gage length and the test assembly was mounted in the testing machine. In all tests the pyrometer thermocouple and a thermocouple for temperature check were attached on opposite sides of the specimen at its center. During heating, compensation was made for thermal expansion by adjustment of the testing machine to maintain zero load on the specimen. After reaching test temperature, loads were evenly applied as extension readings were made.

The maximum gradients over the specimen gage length during test were 6°F at 250°F and 10°F at 550°F. Times to bring the specimen up to test temperature were approximately four minutes at 250°F and approximately nine minutes at 550°F. Approximately four minutes elapsed after the test temperature was reached before testing was begun in order to permit stabilization of the temperature gradient and to make final adjustments.

In a similar manner tensile tests at room temperature were set up using Huggenberg extensometers with the furnace removed.

Creep Tests

The stresses at which the creep tests were conducted were obtained by varying the stress until creep curves were obtained. These showed definite development of a minimum creep rate in a six minute test period. The procedure in making creep tests up to the application of the load was the same as for tensile tests. The only load on the specimen during heating was that due to the dead weight of the lower jaw and adaptor which produced a stress of approximately 200 psi. Upon reaching test temperature, a slight tension was placed on the specimen by finger-tightening of the ball-seat joint on the lower adaptor in the bottom plate of the test frame. Final positioning of the furnace was checked and adjusted to insure proper field of view in the telescopes and the load was applied as evenly as possible by releasing a jack from beneath the loaded pan at the end of the loading lever. Due to the large initial deformations encountered and the type of optical measurements used, accurate determination of the initial deformation was impossible in most cases. Therefore, all data on strain were taken following initial deformation which occurred upon the application of load, and this time dependent strain ($\epsilon_t = \epsilon - \epsilon_o$) was recorded at 15-second intervals over a test time of six minutes.

Maximum gradients over the specimen gage length, the time to bring the specimen up to test temperature, and stabilization time were the same as for the tensile test.

V. RESULTS AND DISCUSSION

Stress-strain and creep tests were conducted according to the procedures previously given for test temperatures of 550°, 250°F and room temperature. The results of these tests are given in the curves of Figures 2, 5, and 15 through 22. The physical properties obtained from these curves are tabulated in Table I. There were four primary conditions of testing; (1) room temperature test, 250°F heat treatment, (2) 250°F test, 250°F heat treatment, (3) room temperature test, 550°F heat treatment, and (4) 550°F test, 550°F heat treatment. Tests were also run on untreated specimens at room temperature, 250°F, and 550°F.

The results of the stress-strain tests on untreated specimens for the three test temperatures are given in the form of stress-strain curves in Figure 2. As an indication of the effect of test temperature on yield strength and modulus of elasticity, the curves of Figures 3 and 4 were constructed from values obtained from Figure 2.

The results of the creep tests on untreated specimens for the three test temperatures at the stresses chosen for comparison of cyclic heating effects are given in the creep curves in Figure 5.

The results of the stress-strain tests for the four primary test conditions are shown in the four sets of curves in Figures 15 through 18. The curves have offset origins to facilitate plotting and interpretation. The values of modulus of elasticity and 0.2 per cent offset yield strength obtained from these curves and the tensile strength are tabulated in Table I, and are cross-plotted against heat treatment cycles in Figure 23 to illustrate the variation of these physical properties with heating cycles. Tensile strength at 550°F could not be obtained due to the limitations previously mentioned.

The results of the "fast rate" creep test for the four primary test conditions are shown in the four sets of curves in Figures 19 through 22. Where necessary, the time origins have been offset to facilitate plotting and interpretation. The values of minimum creep rate (MCR) obtained from these curves are tabulated in Table I and are cross-plotted against heat treatment cycles in Figure 24 to illustrate the variation with heating cycles.

The primary concern of the investigation was to determine whether or not there would be any difference in effect on the physical properties as the result of varying the cyclic heating and cooling treatments. From the presentation of the data it appears that there is a negligible effect due to the difference in the cyclic heat treatments under any of the testing conditions. It may appear that there is considerable variation in the minimum creep rates for the 250°F creep tests (250°F heat treatment), but when it is considered that the stresses used to obtain the "fast rate" creep are in the low slope portion of the stress-strain curve, close to the ultimate tensile strength, some variation should be expected because of slight differences in material. For the 550°F tensile tests (550°F heat treatment) there appears to be more of a variation of modulus of elasticity among the different heat treatments, but it is felt that this is probably due to errors in experimental technique magnified by the high temperature conditions.

The specimens were kept at elevated temperatures in the transient heating period for a considerable length of time, and therefore, the specimens subjected to the 20 cycle heating were at elevated temperatures much longer than the one cycle specimens. Since there was a negligible difference in the physical properties of the specimens subjected to the different cyclic heat treatments, it appears that at elevated temperatures for periods of time greater than that corresponding to the 60 minute (one cycle) treatment and less than that corresponding to the three minute (20 cycle) treatment, time at elevated temperatures has no effect on the reduction of properties, and that the effects of temperature are dependent only on the maximum temperature. This is in agreement with the results of Reference 6, where a thorough investigation was conducted on the effect of temperature and exposure time on the physical properties for periods of time from 30 minutes to 1000 hours.

It is of interest to note from the stress-strain tests that the only conditions of testing where there is any difference between the heat treated specimens and the untreated specimens is that of room temperature test (550°F heat treatment), where there is considerable reduction in yield and tensile strength as the result of the heat treatment, although all curves start to break at about the same stress (Figure 17). At the other test conditions, the cyclic heat treatment has had no effect on the stress-strain characteristics.

It is also evident that there is a similar observation to be made from the creep tests. For all of the test conditions, with the exception of room temperature test (550°F heat treatment), the cyclic heat treatment has had no effect on the minimum creep rates. At room temperature test

(550°F heat treatment) there has been a considerable increase in the minimum creep rate as the result of the heat treatment.

VI. CONCLUSIONS

1. There is no effect on the physical properties of FS-1h magnesium alloy as a result of changing the number of cycles of heating, in the range of 1 to 20 cycles, where the periods at heat treating temperature total 60 minutes.

2. There is a period of time at elevated temperature, from 60 minutes and greater, for which the resulting reduction in physical properties apparently does not vary with increase in time.

3. Cyclic heat treatments investigated at 250°F have no effect on the physical properties of FS-1h tested either at room temperature or at 250°F. Cyclic heat treatments investigated at 550°F have no effect on the physical properties of FS-1h at 550°F. Heat treated specimens at these test conditions show only the normal reduction of properties due to the elevated test temperatures.

4. Cyclic heat treatments at 550°F result in a reduction of 27 per cent in yield strength, 9 per cent in tensile strength, and a reduction in creep resistance of FS-1h when tested at room temperature.

VII. RECOMMENDATIONS

1. This investigation should be extended to determine what minimum heat treatment is required to bring about a reduction in room temperature physical properties found to result from the 550°F cyclic heat treatment. Since all of the cycles investigated resulted in the same reduction, it is recommended that a one-cycle heating be investigated, where the period at temperature is varied, starting from a practicable minimum, and increased until the period is reached where there is a negligible change in the properties with change in time, as indicated in Reference 6. This should be done over the temperature range of 250°F to 550°F. Thus, it would be possible to determine how the reduction of properties varies with increase in temperature, and depends on time at temperature.

2. With the present equipment, tensile strength at elevated temperature could be determined using specimens with a shorter gage length, although no strain measurement could be obtained.

3. One of the undesirable variables in the present set up for creep tests is the uneven application of loading resulting from the manual operation of a hydraulic or screw type jack. A much desired improvement would be possible by use of a jack which retracts at a predetermined, fixed rate.

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TABLE I. FS-1h Magnesium Physical Properties - Cyclic Heat Treatment

Heat Treatment Temp.	Heat Treatment No. Cycles	Modulus of Elasticity, E (lb/sq. in.)	Yield Strength 0.2°/o Offset (lb/sq. in.)	Tensile Strength	Minimum Creep Rate (in./in./HR)	Modulus of Elasticity, E (lb/sq. in.)	Yield Strength 0.2°/o Offset (lb/sq. in.)	Minimum Creep Rate (in./in./HR)
		Room Temperature Test				250°F Test		
					(σ = 39,600)			(σ = 25,000)
None		6.6×10^6	29,500	40,700	0.16	3.3×10^6	19,900	0.70
250°F	1	6.2×10^6	30,000	40,600	0.19	3.2×10^6	19,500	0.91
	5	6.2×10^6	29,800	40,600	0.16	3.3×10^6	20,500	0.99
	12	6.2×10^6	28,800	40,300	0.22	3.3×10^6	20,300	0.80
	20	6.2×10^6	29,700	40,700	0.16	3.2×10^6	20,000	0.94
		Room Temperature Test				550°F Test		
					(σ = 36,500)			(σ = 4,300)
None		6.6×10^6	29,500	40,700	negligible	0.68×10^6	4,600	0.40
550°F	1	6.4×10^6	21,600	37,300	0.22	0.79×10^6	4,400	0.38
	5	6.4×10^6	21,800	37,500	0.16	0.73×10^6	4,500	0.35
	12	6.4×10^6	21,000	36,600	0.21	0.88×10^6	4,500	0.35
	20	6.4×10^6	21,400	37,000	0.17	0.76×10^6	4,300	0.37

(All specimens 60 minutes total time at heat treatment temperature)

Nominal Published Values: E : 6.25×10^6 psi Yield Strength : 26 - 33,000 psi

Tensile Strength : 38 - 43,000 psi

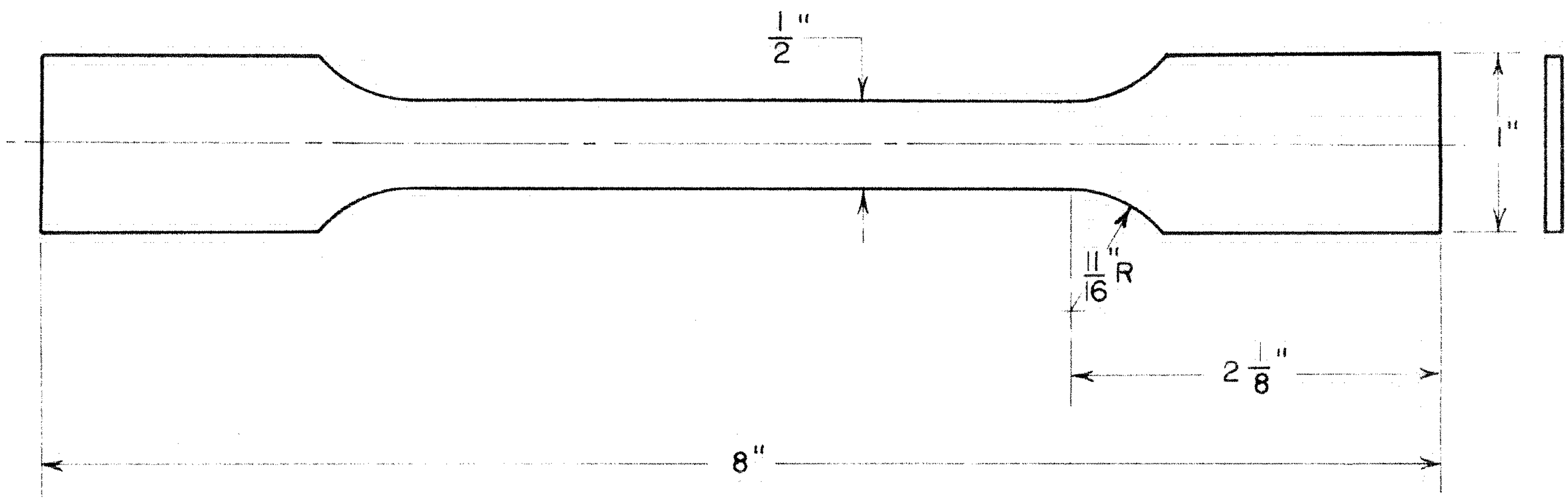


FIG. I — TEST SPECIMEN

FIG. 2

FS-1h MAGNESIUM TENSILE STRESS-STRAIN CURVES
ROOM AND ELEVATED TEMPERATURE TESTS
NO HEAT TREATMENT PRIOR TO TESTING

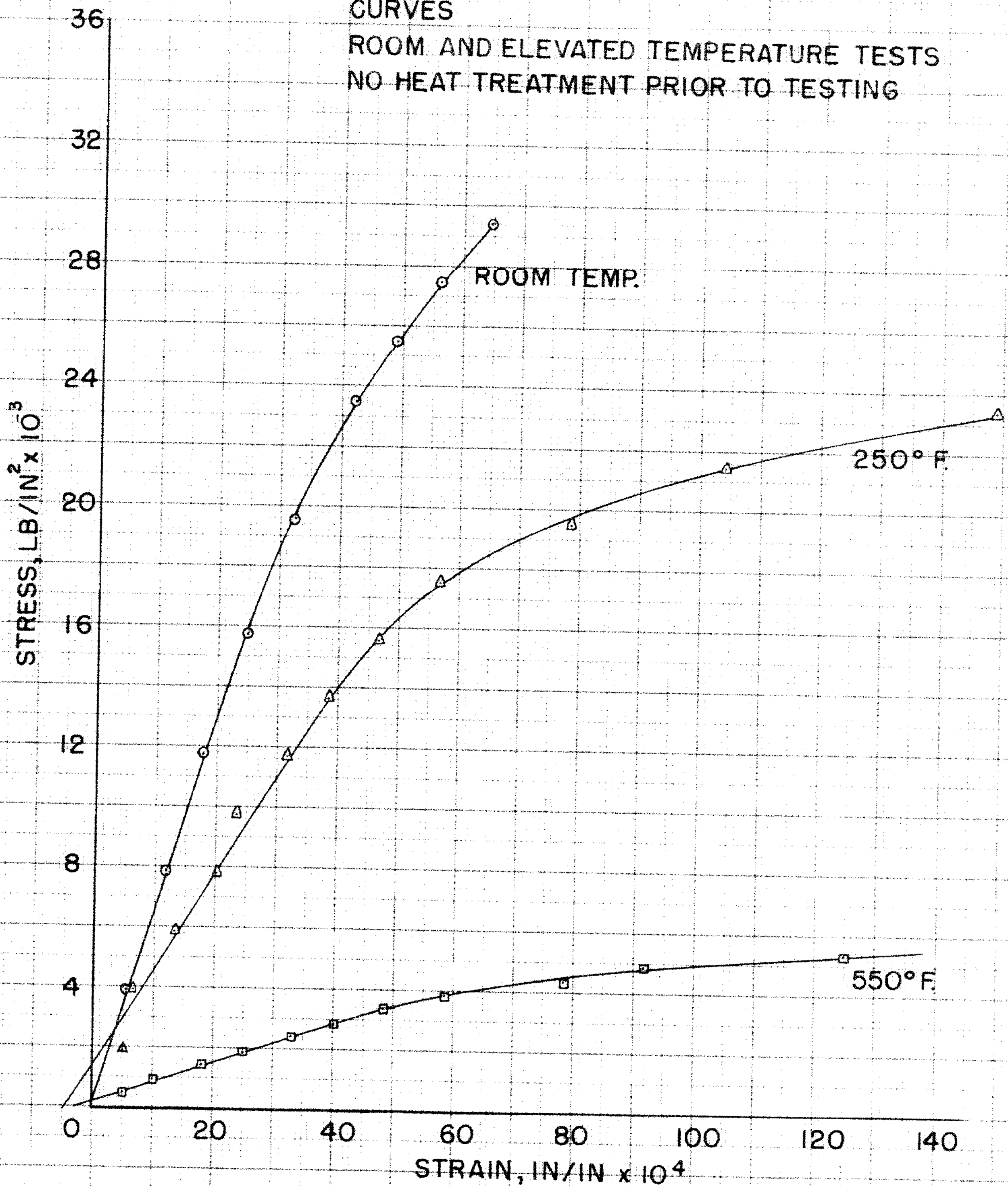


FIG. 3

VARIATION OF YIELD STRENGTH IN TENSION WITH TEMPERATURE
NO HEAT TREATMENT PRIOR TO TESTING

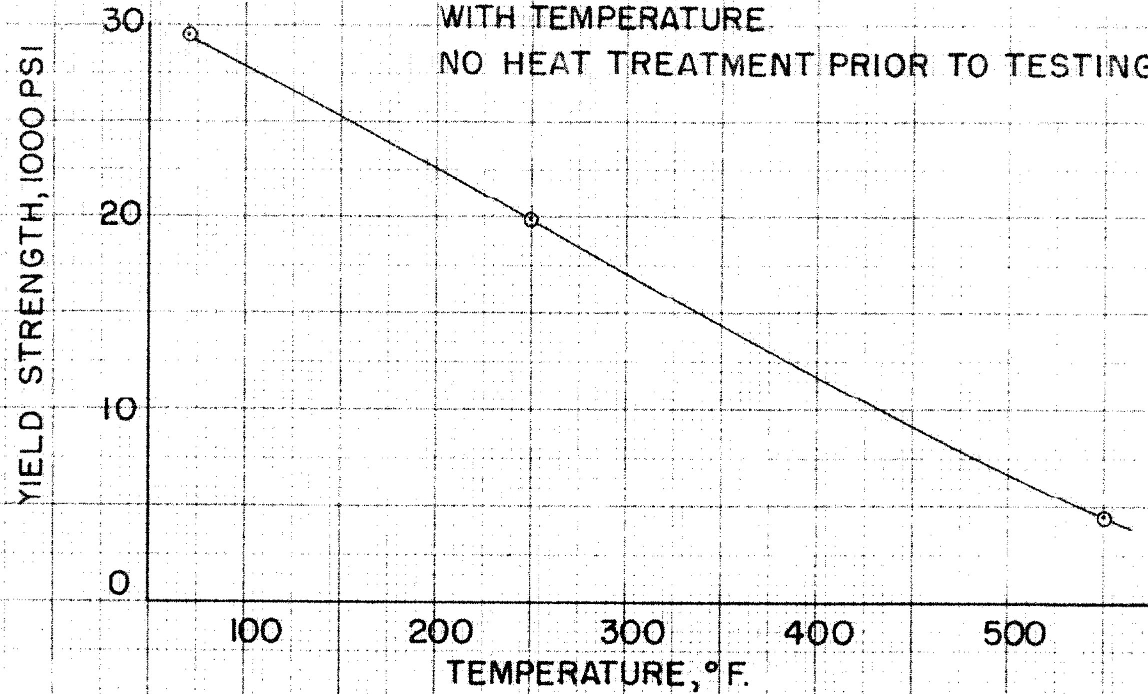
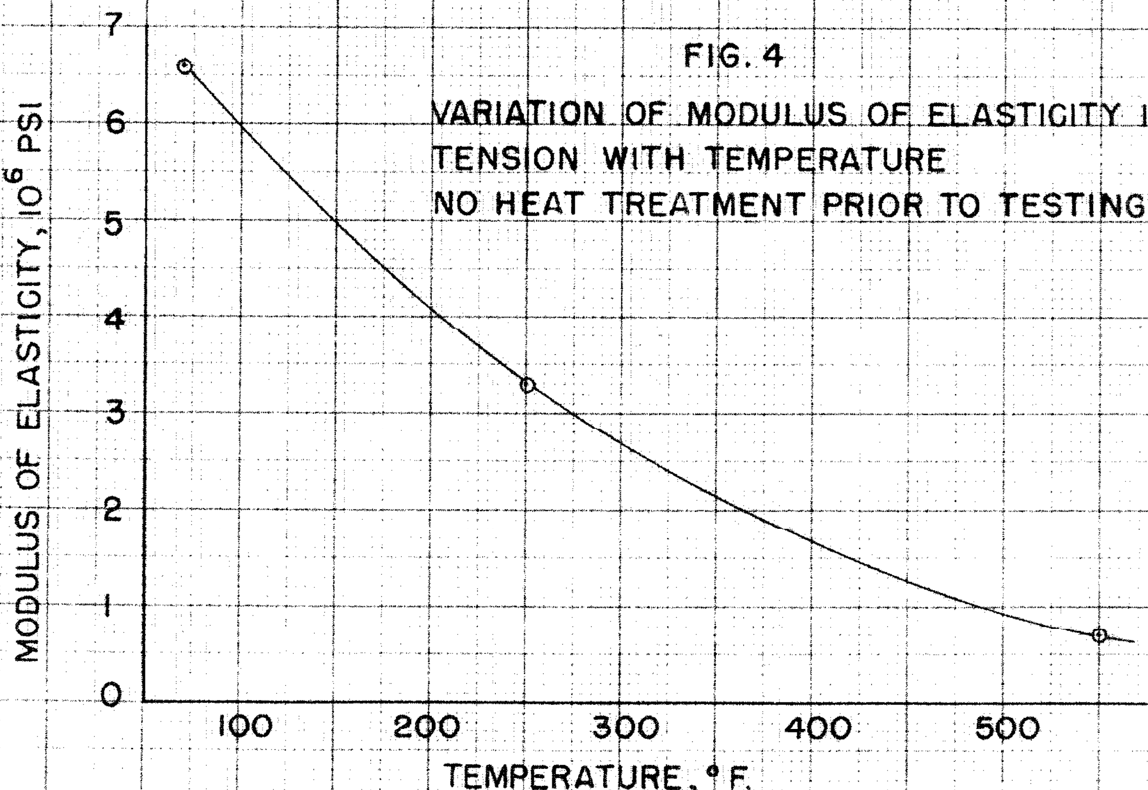


FIG. 4

VARIATION OF MODULUS OF ELASTICITY IN TENSION WITH TEMPERATURE
NO HEAT TREATMENT PRIOR TO TESTING



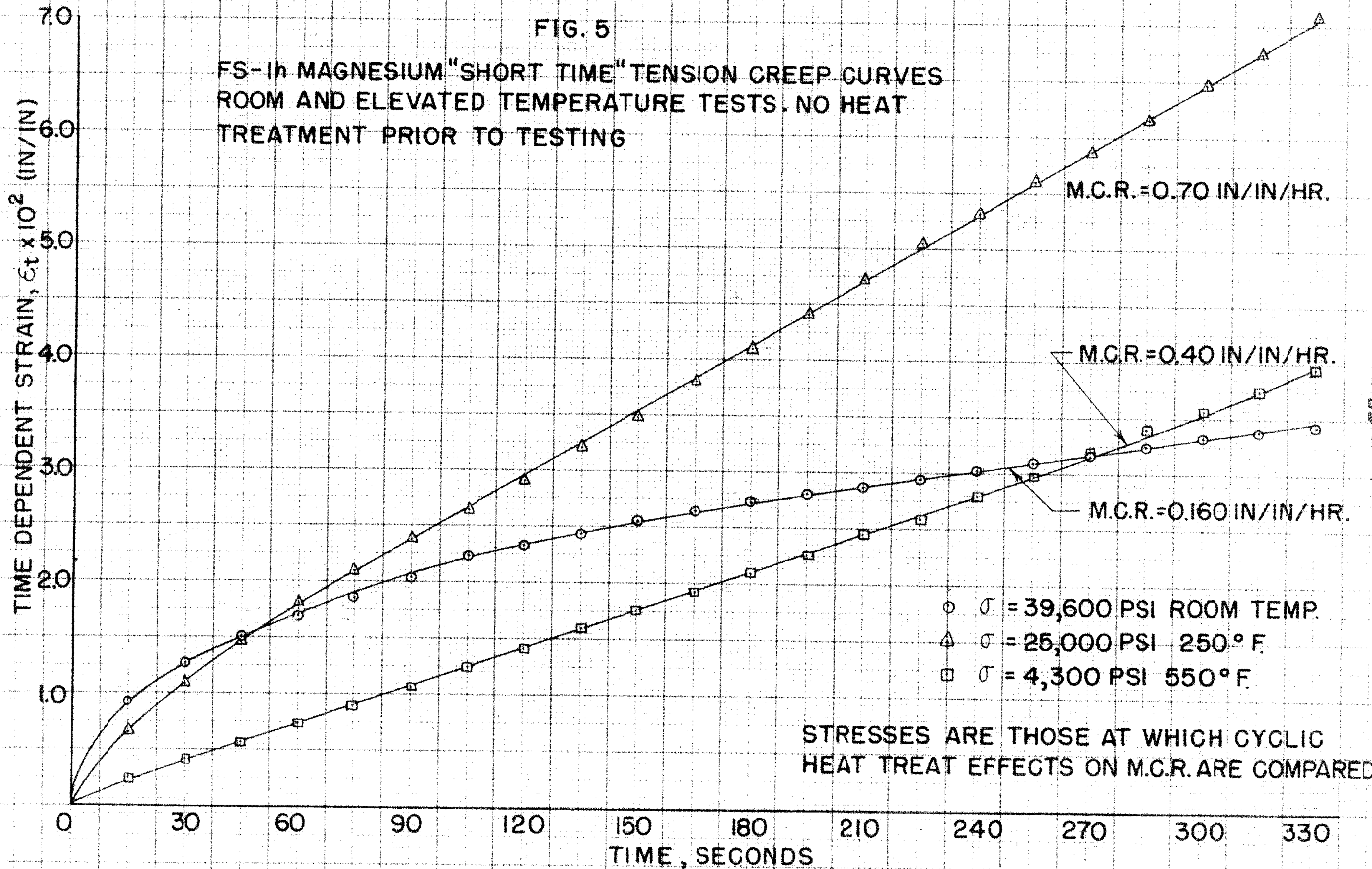


FIG. 5

FS-1h MAGNESIUM "SHORT TIME" TENSION CREEP CURVES ROOM AND ELEVATED TEMPERATURE TESTS. NO HEAT TREATMENT PRIOR TO TESTING

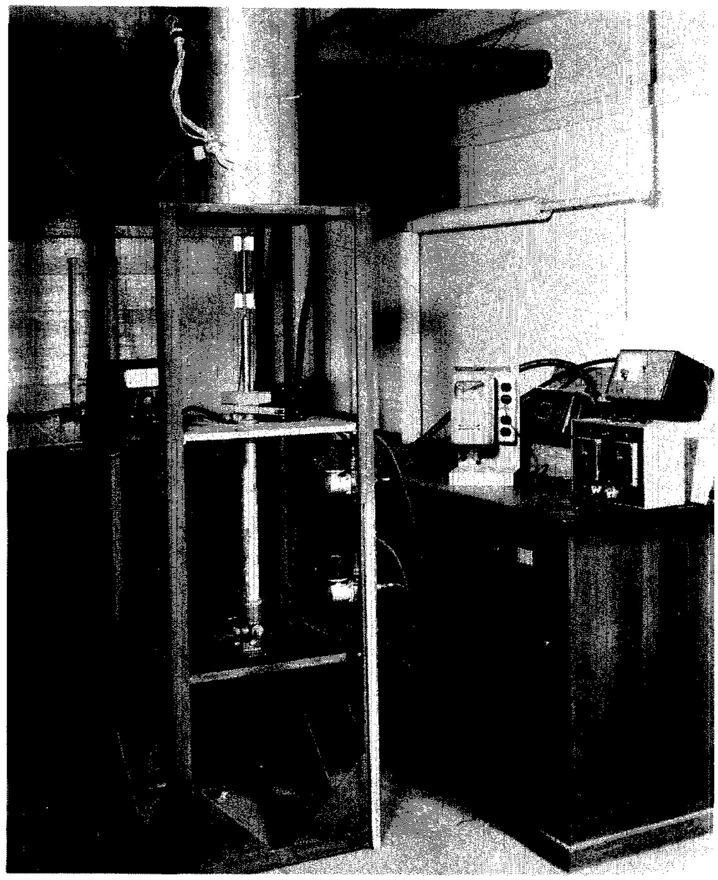


Figure 6 Heat Treatment Furnace

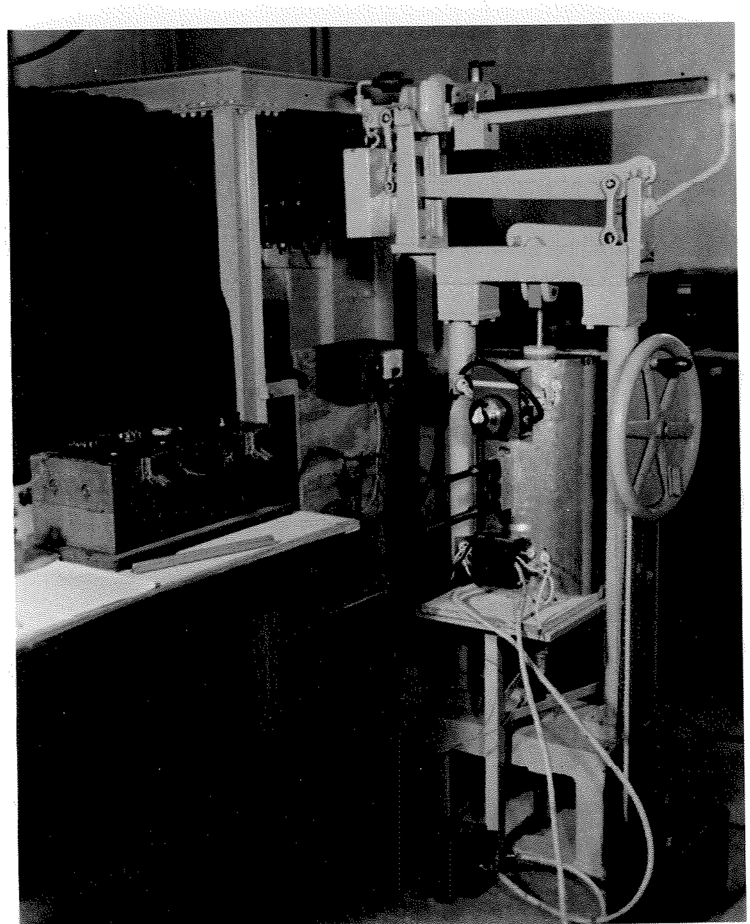


Figure 7 Tensile Testing Machine and Furnace

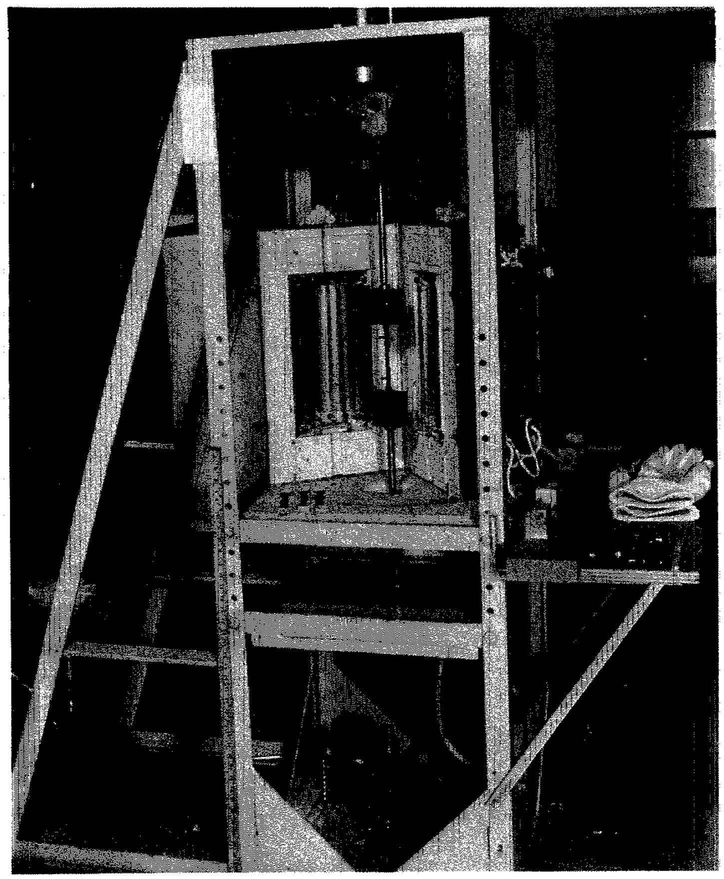


Figure 8 Creep Loading Frame and Furnace

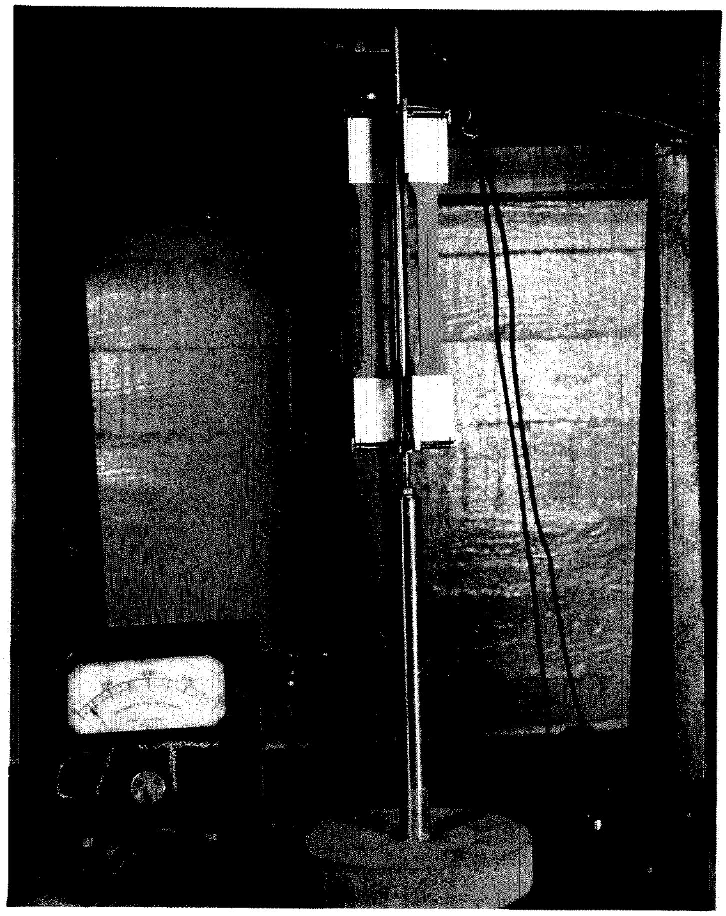
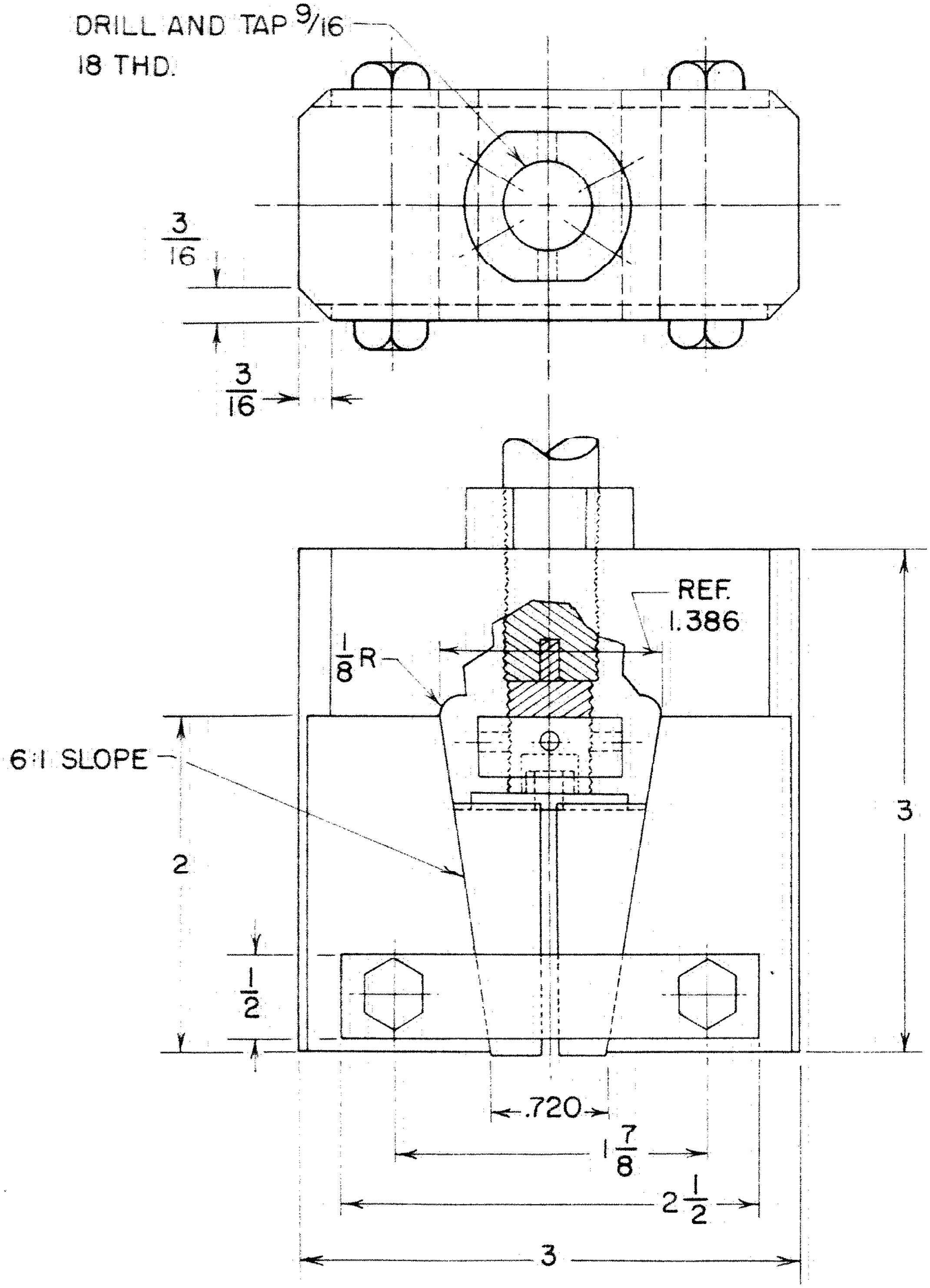


Figure 9 Specimen Mounting for Heat Treatment


DRILL AND TAP 9/16
18 THD.
3/16
3/16
REF. 1.386
1/8 R
6:1 SLOPE
2
1/2
3
.720
1 7/8
2 1/2
3

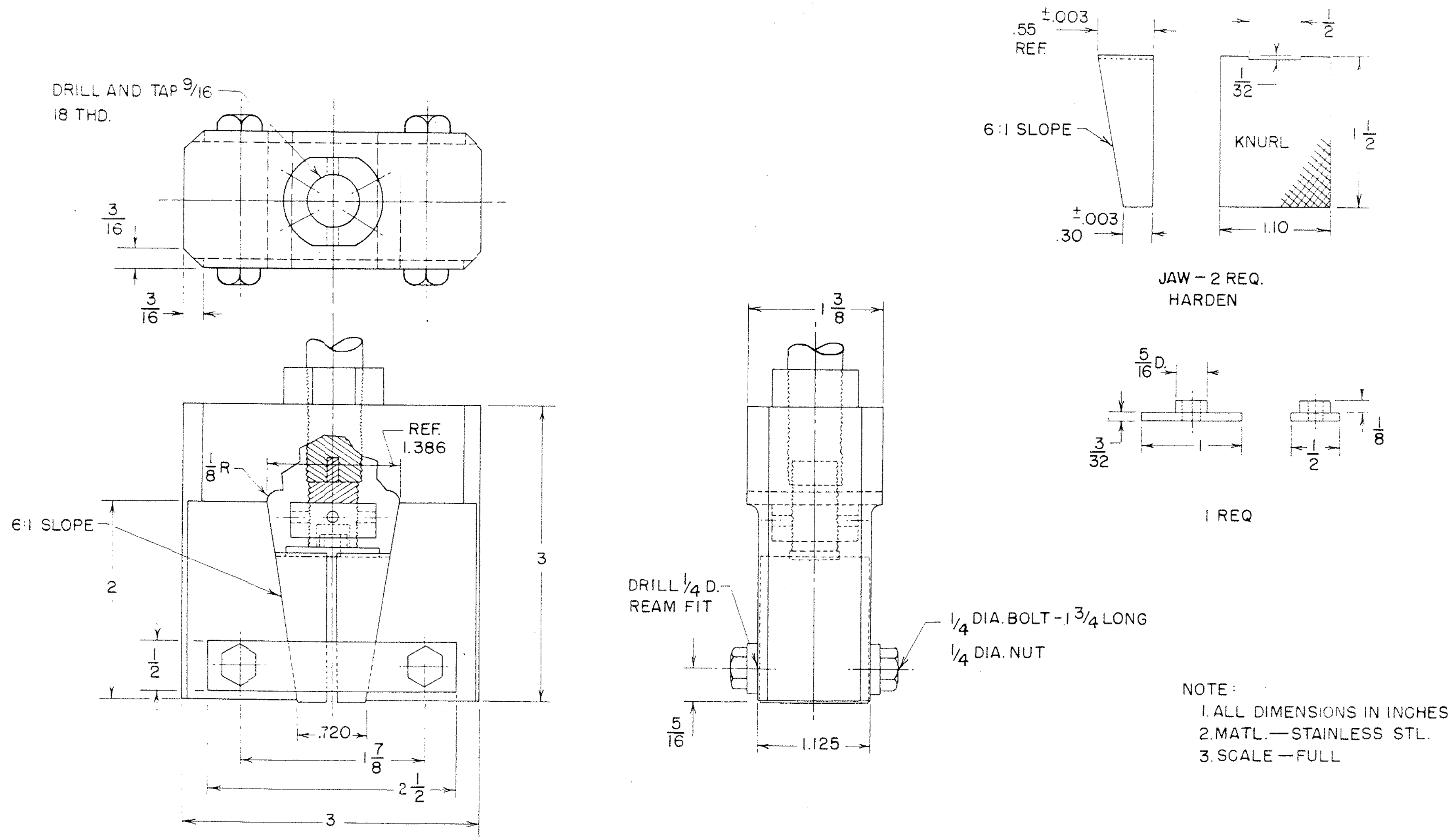


FIG. 10 — TENSILE TEST GRIPS

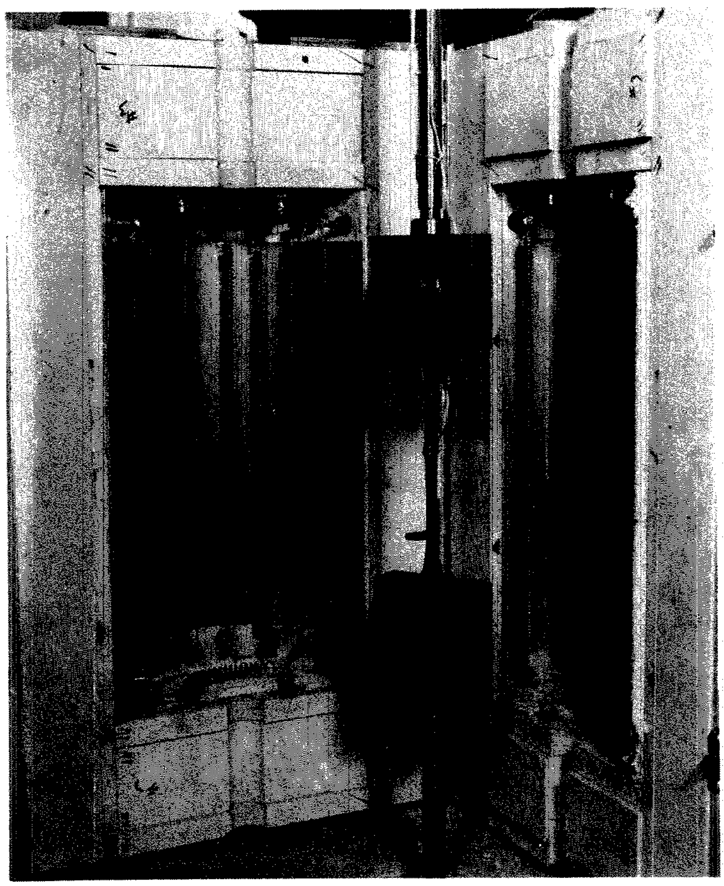


Figure 11 Specimen Mounted in Grips for Test

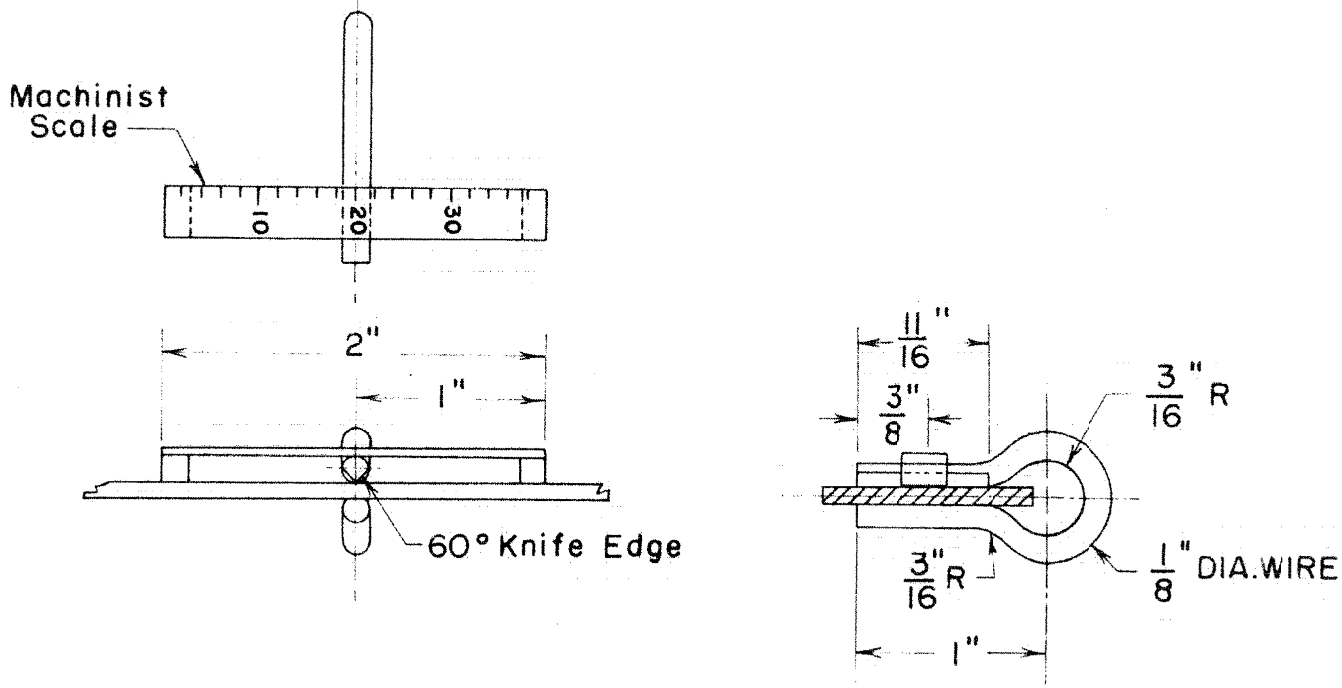


FIG. 12 — SPECIMEN SCALE CLIP

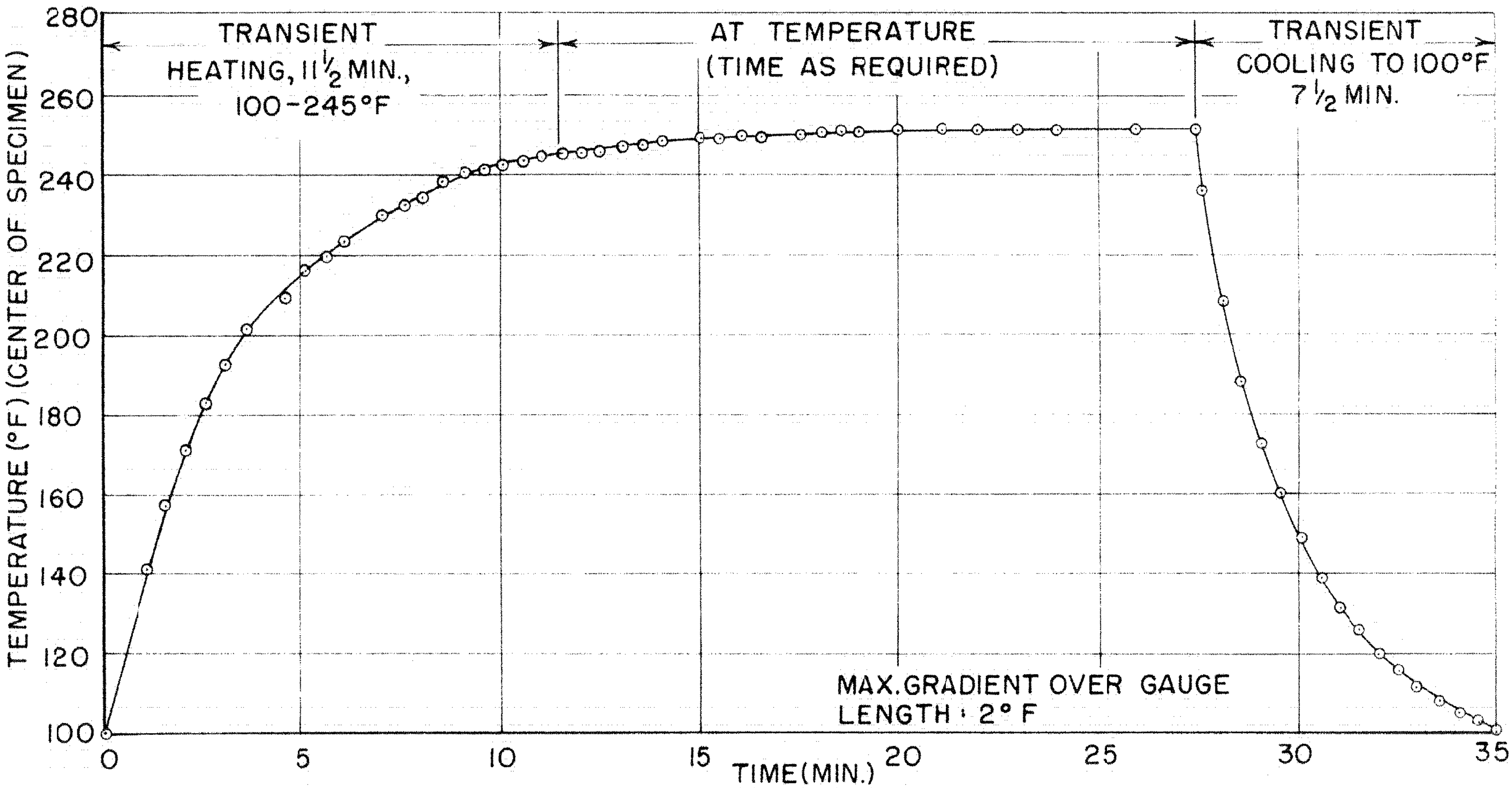


FIG. 13-TEST SPECIMEN HEAT TREAT CYCLE (250°)

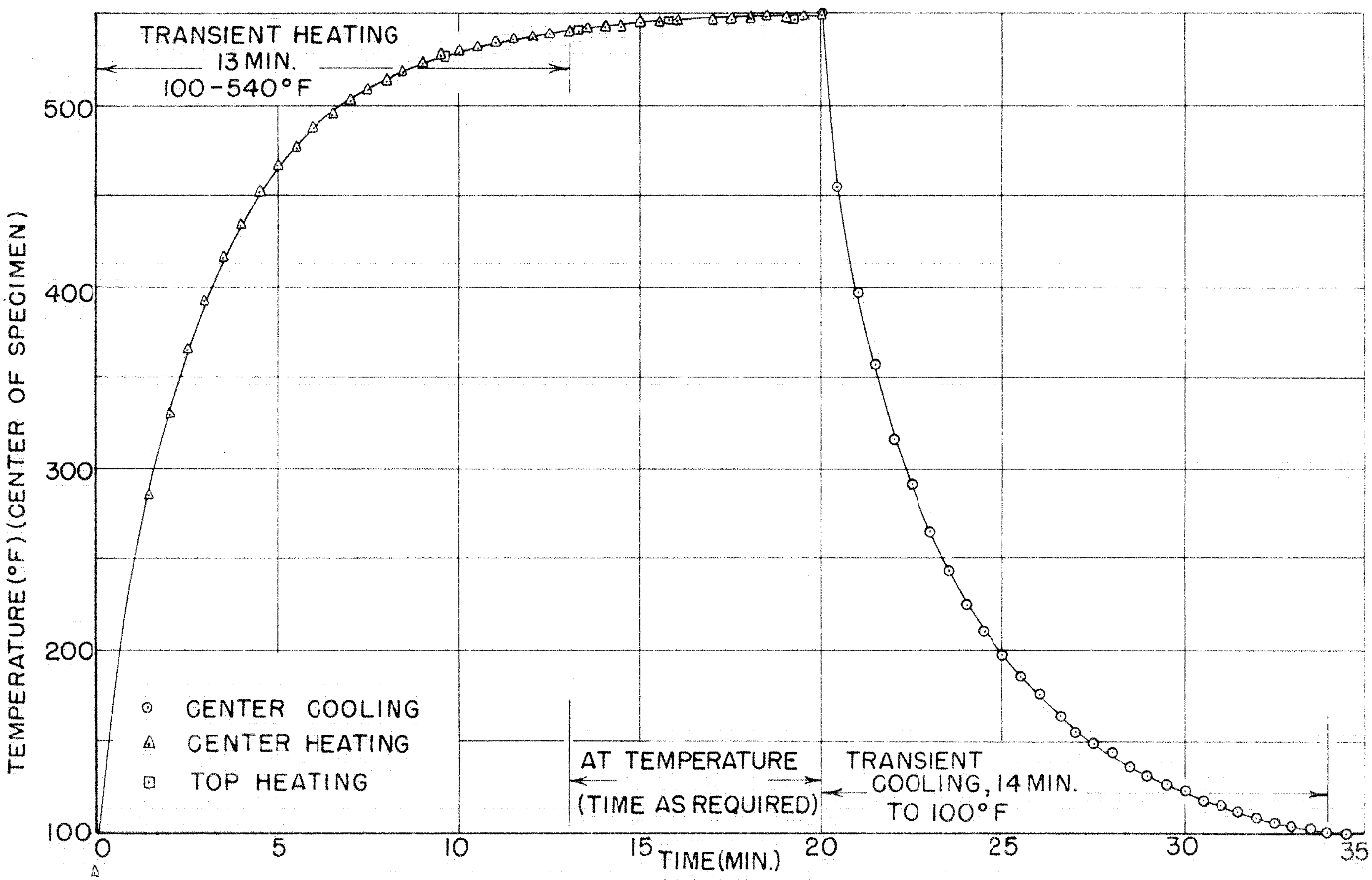


FIG. 14 - TEST SPECIMEN HEAT TREAT CYCLE (550°)

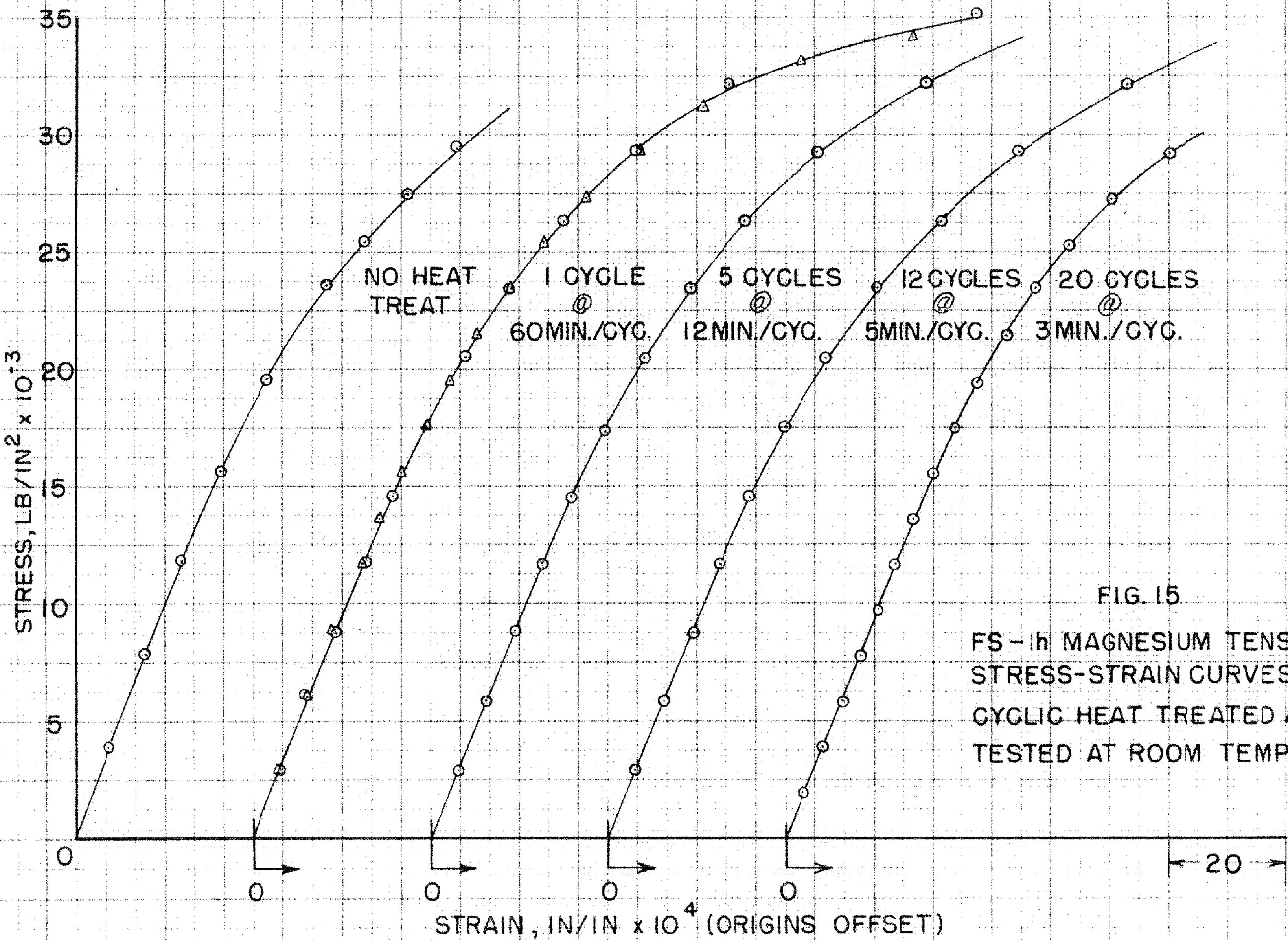


FIG. 15

FS-1h MAGNESIUM TENSION STRESS-STRAIN CURVES.
CYCLIC HEAT TREATED AT 250° F.
TESTED AT ROOM TEMP.

FIG. 16

FS-1h MAGNESIUM TENSION STRESS-STRAIN CURVES
CYCLIC HEAT TREATED AT 250° F.
TESTED AT 250° F

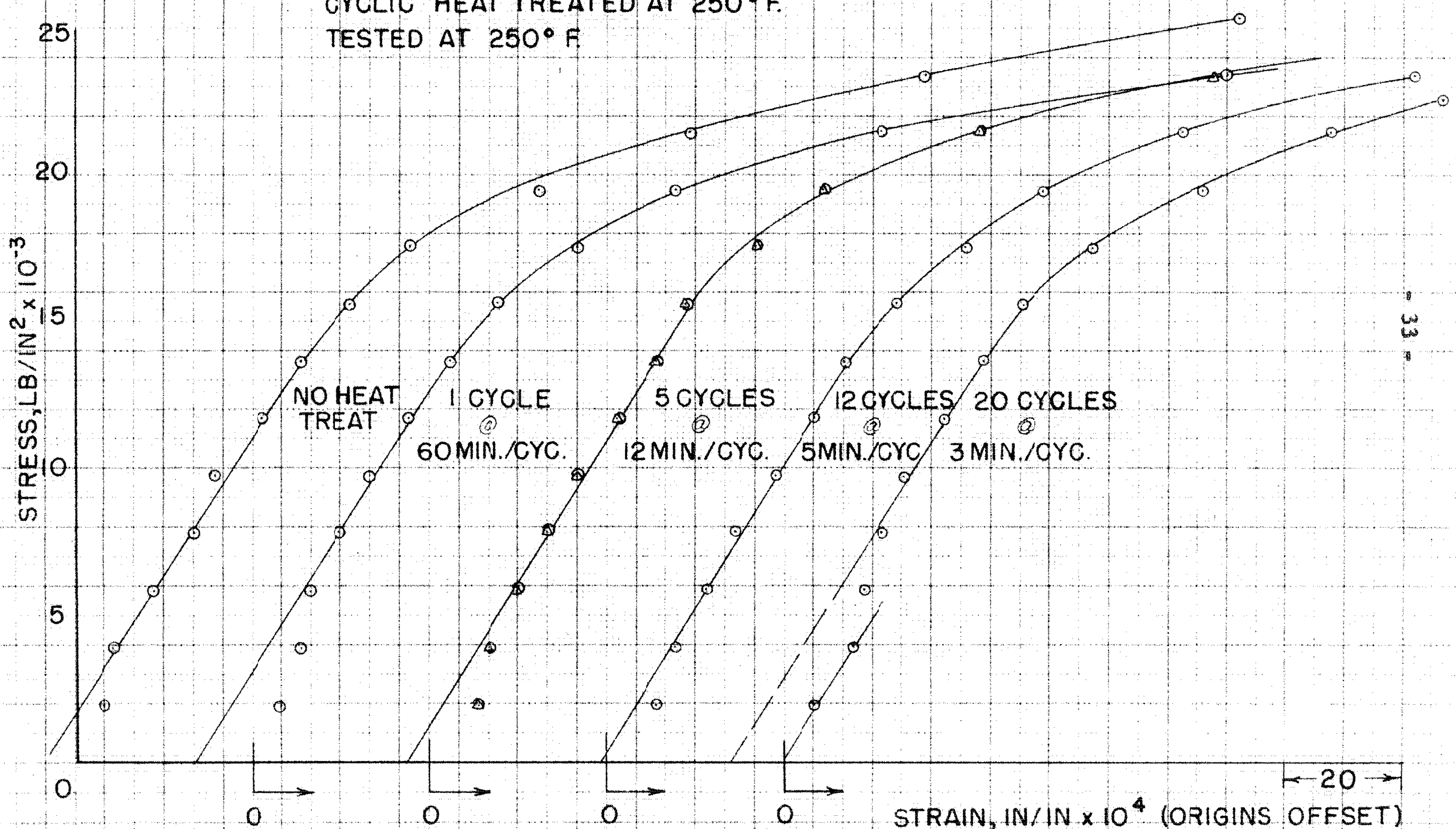


FIG. 17

FS-1h MAGNESIUM TENSION STRESS-STRAIN CURVES
CYCLIC HEAT TREATED AT 550° F.
TESTED AT ROOM TEMP.

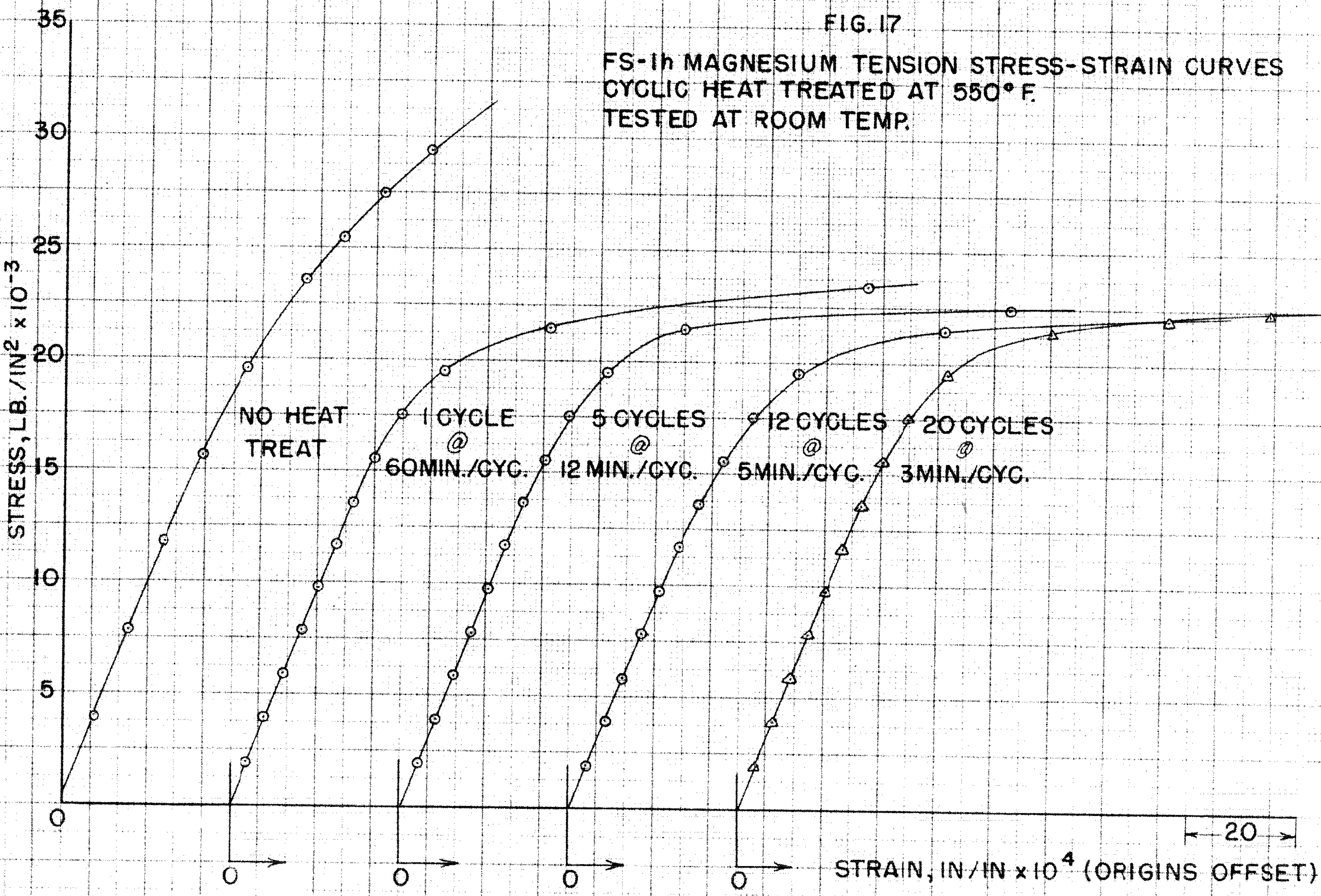


FIG. 18

FS-1h MAGNESIUM TENSION STRESS-STRAIN CURVES

CYCLIC HEAT TREATED AT 550° F.

TESTED AT 550° F.

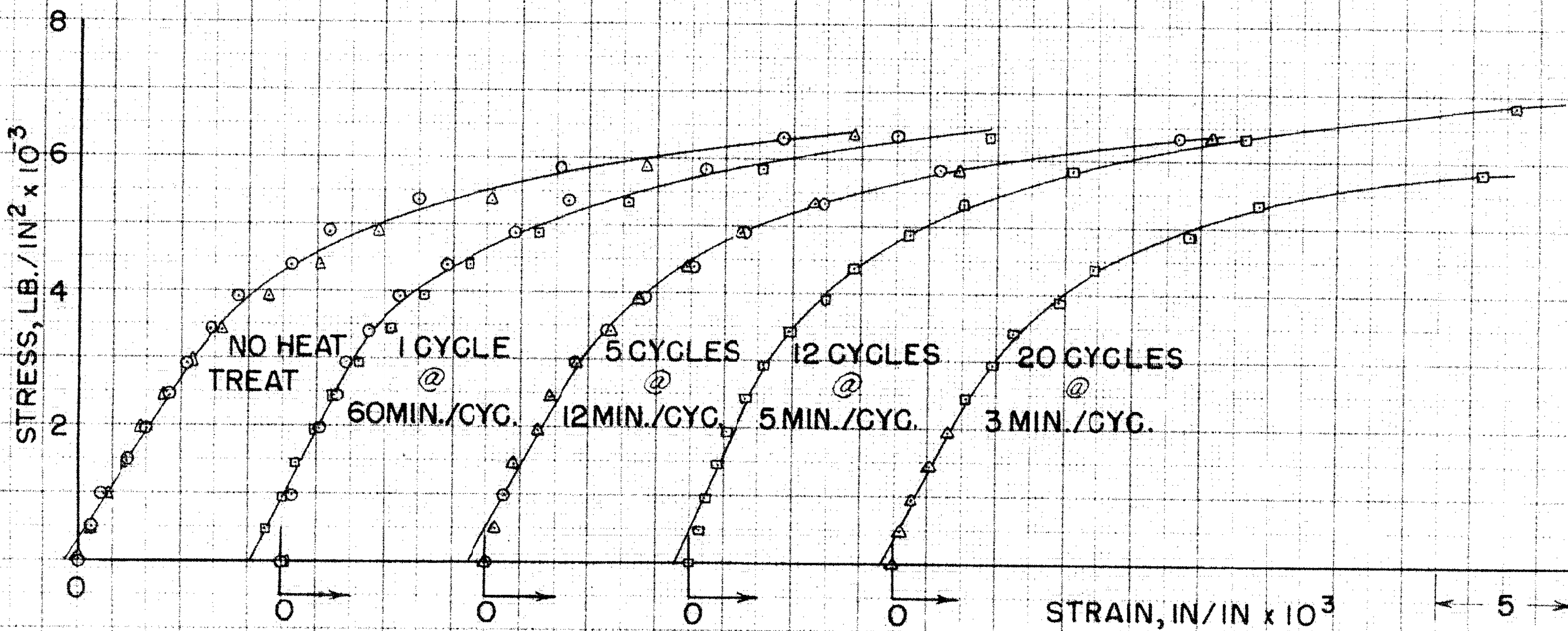


FIG. 19

FS-1h MAGNESIUM "SHORT TIME" TENSION CREEP CURVES
CYCLIC HEAT TREATED AT 250°F.
TESTED AT ROOM TEMPERATURE
STRESS = 39,600 PSI

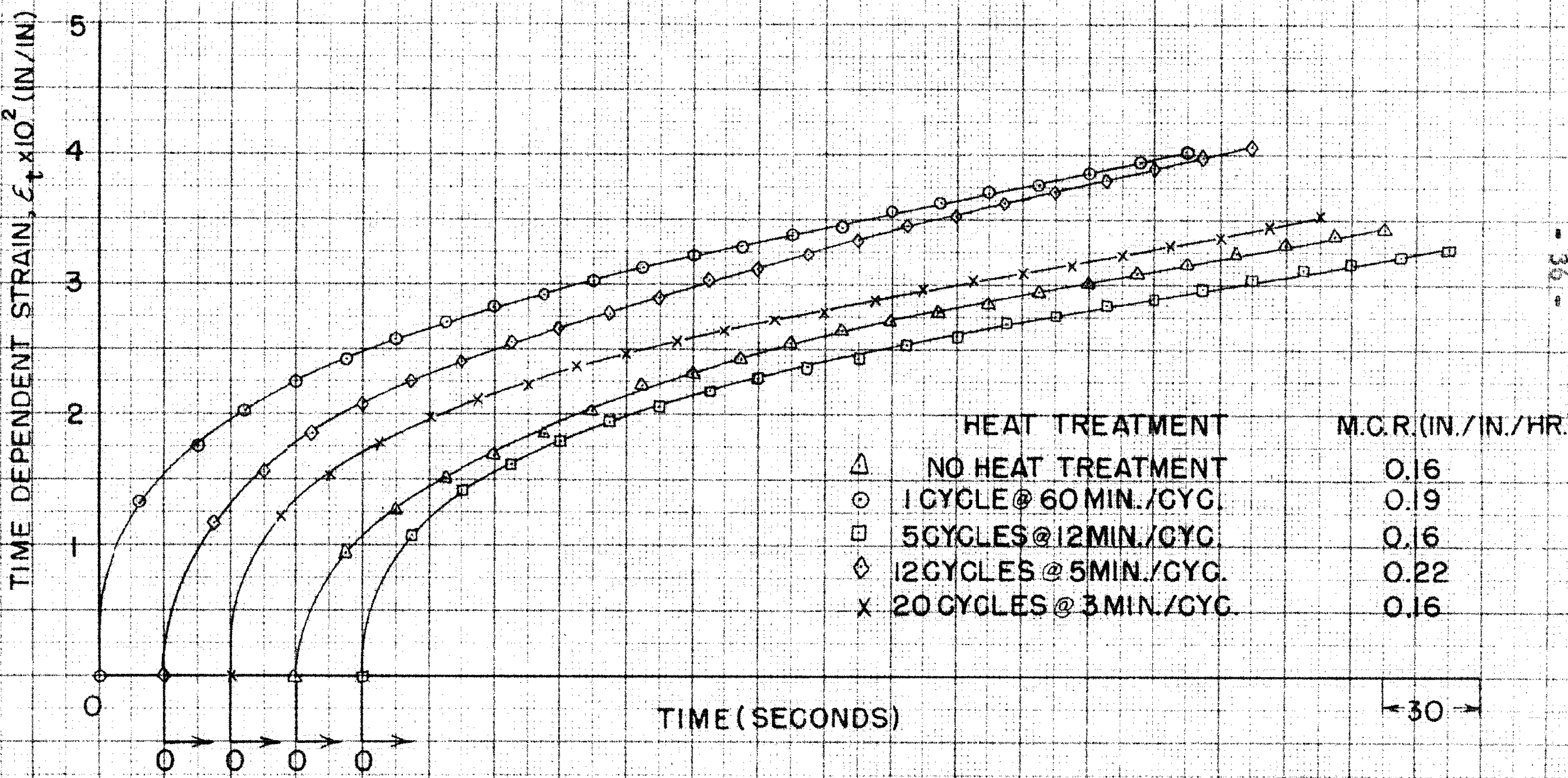
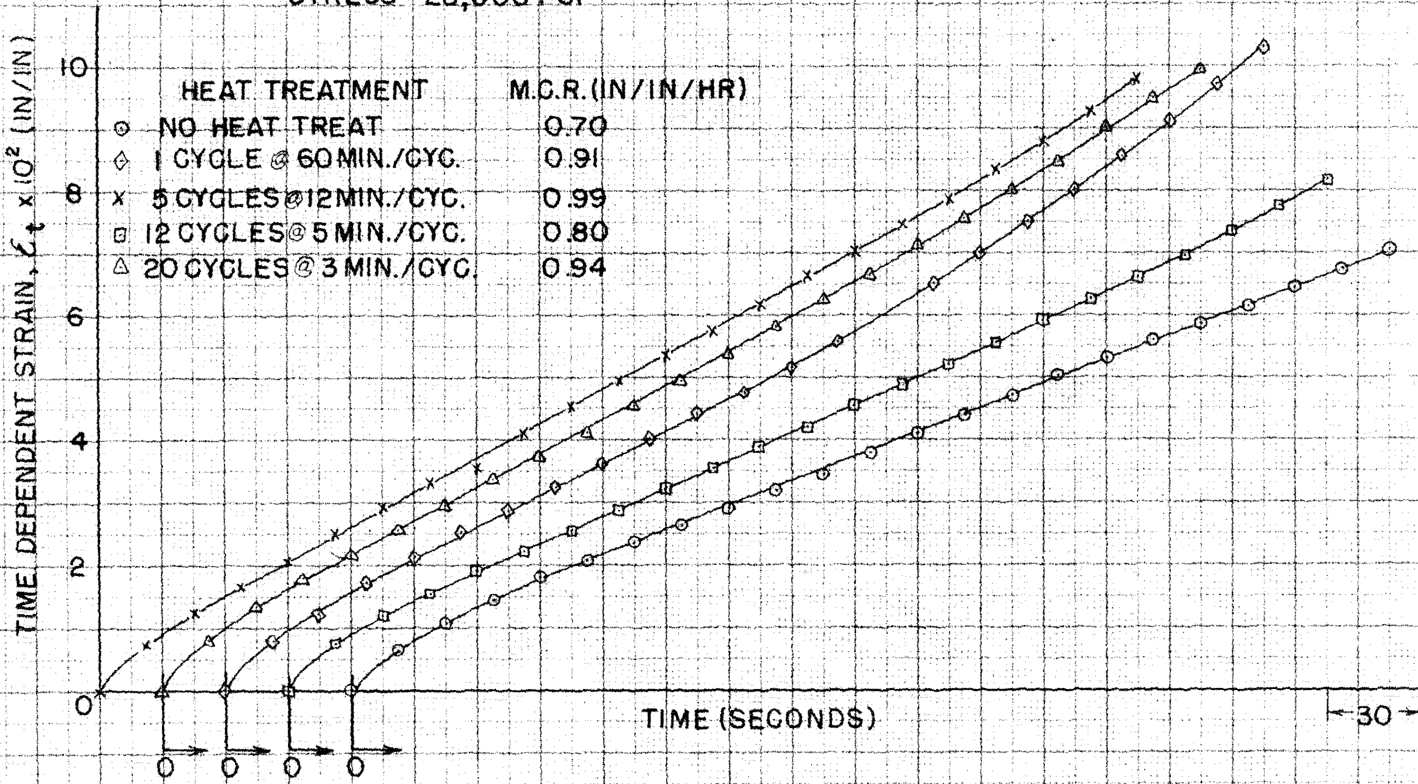


FIG. 20

FS-1h MAGNESIUM "SHORT TIME" TENSION CREEP CURVES
CYCLIC HEAT TREATED AT 250 °F.
TESTED AT 250 °F.
STRESS = 25,000 PSI



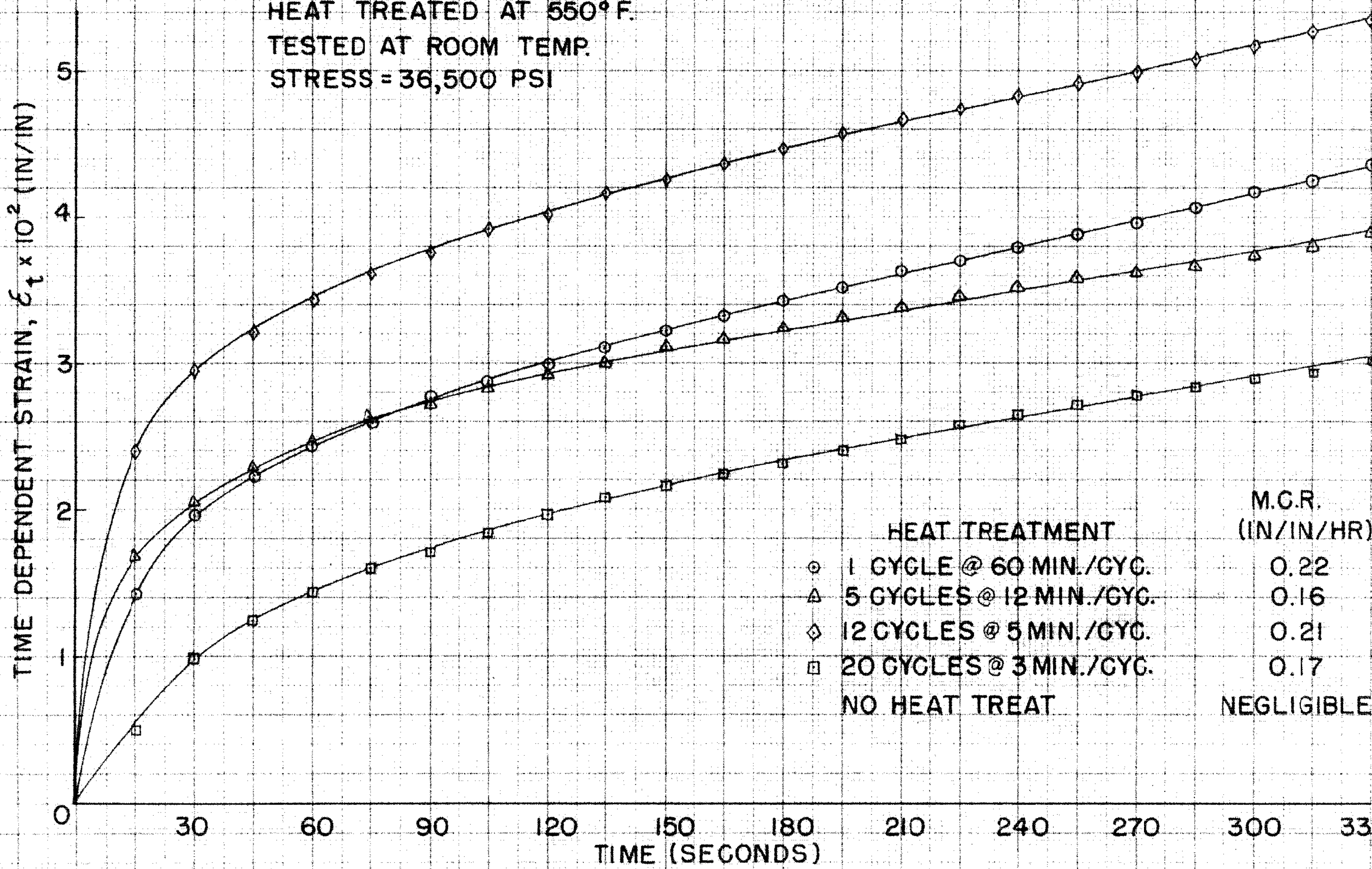


FIG. 21

FS-1h MAGNESIUM ALLOY "SHORT TIME" TENSION CREEP CURVES
HEAT TREATED AT 550° F.
TESTED AT ROOM TEMP.
STRESS = 36,500 PSI

FIG. 22

FS-1h MAGNESIUM "SHORT TIME" TENSION CREEP CURVES
CYCLIC HEAT TREATED AT 550 °F.
TESTED AT 550 °F.
STRESS = 4,300 PSI

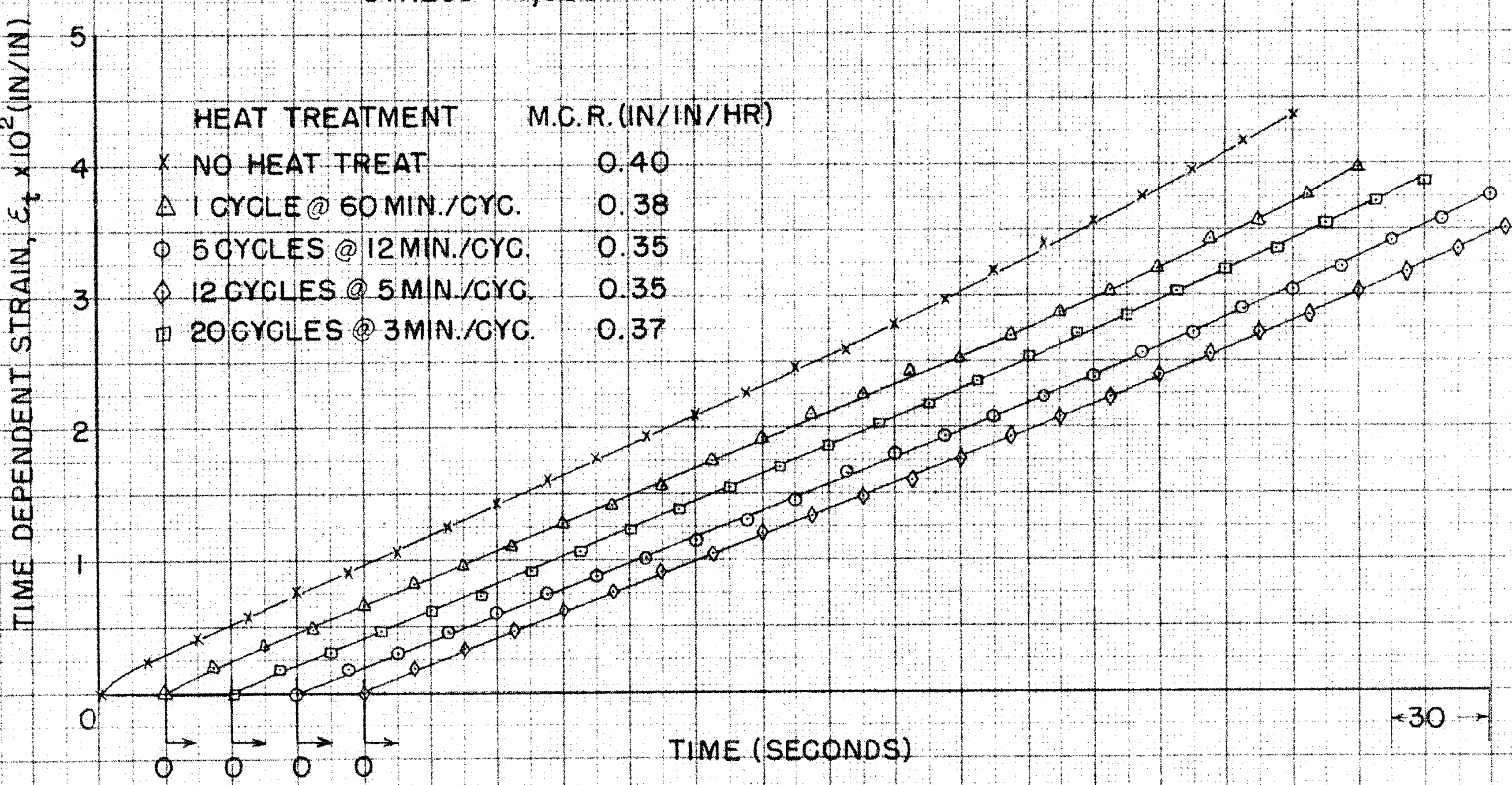
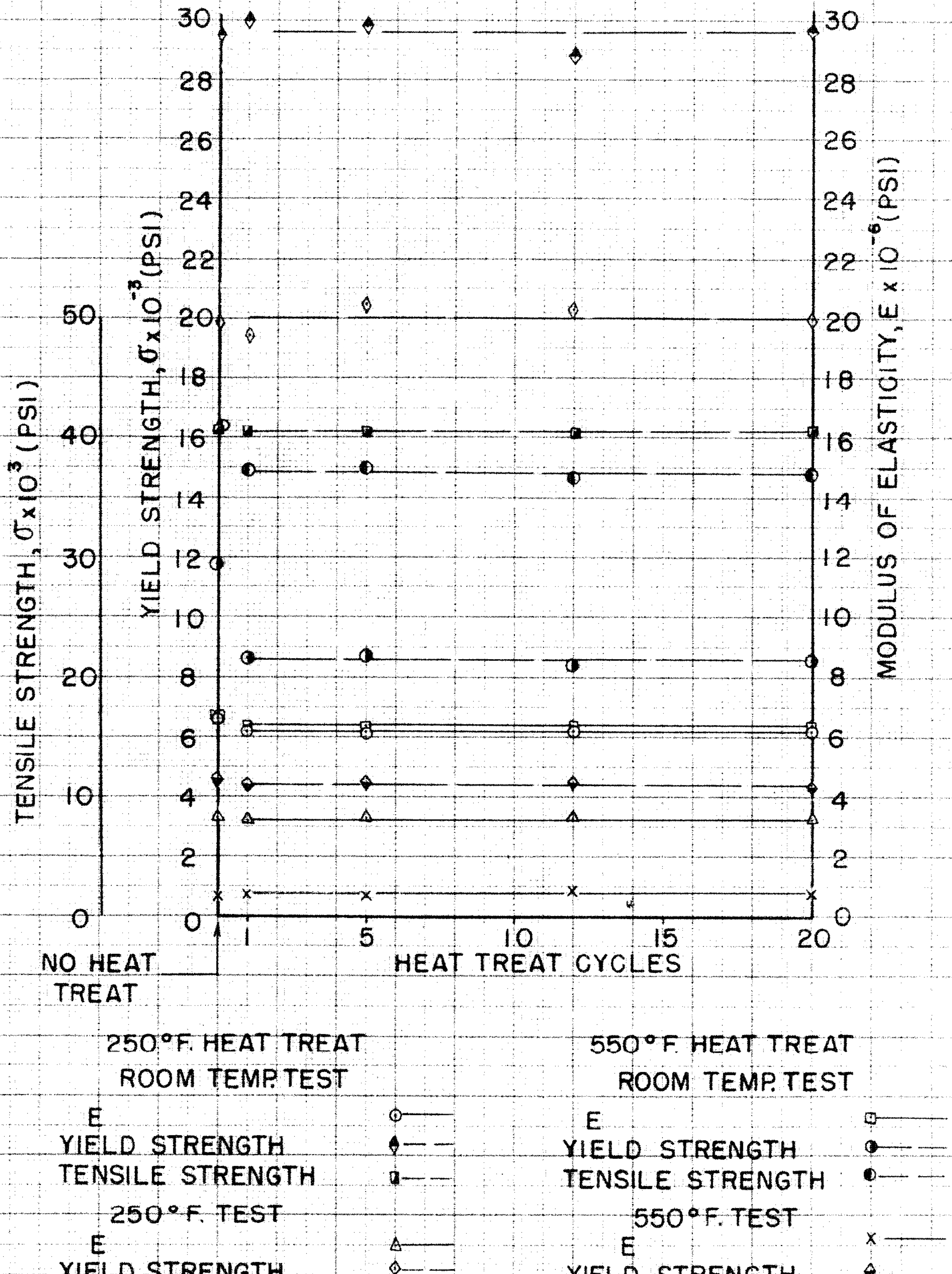


FIG. 23

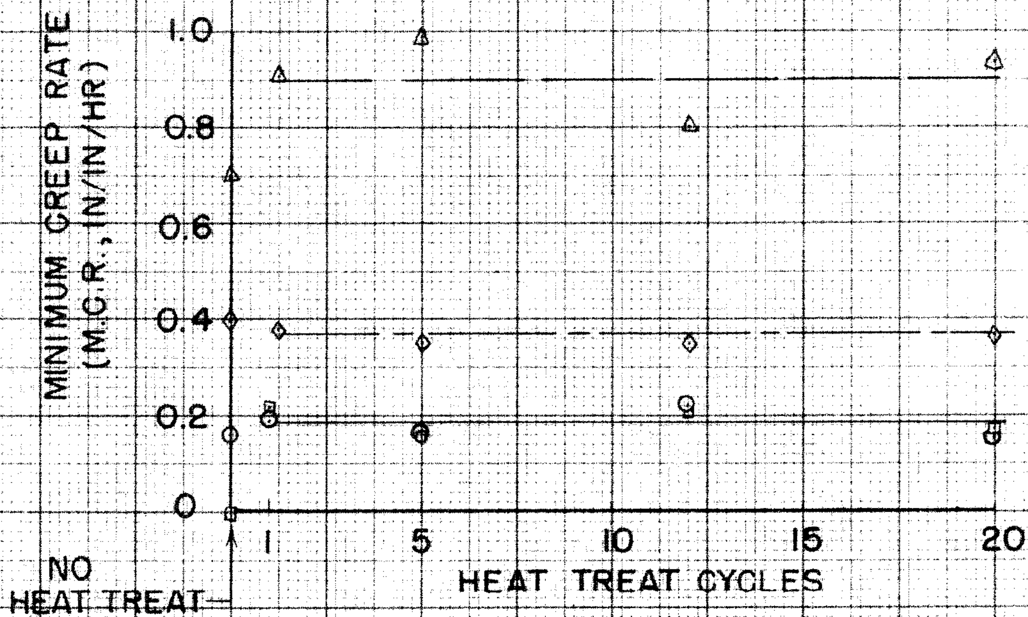
FS-1h MAGNESIUM TENSILE PROPERTIES vs HEATING-COOLING CYCLE



(SPECIMENS 60 MIN. TOTAL TIME AT HEAT TEMP.)

FIG. 24

FS-1h MAGNESIUM "SHORT TIME" MINIMUM CREEP RATES vs HEATING-COOLING CYCLES



250°F. HEAT TREAT

ROOM TEMP. CREEP σ = 39,600 PSI

250° F. CREEP σ = 25,000 PSI

550° F. HEAT TREAT

ROOM TEMP. CREEP σ = 36,500 PSI

550° F. CREEP σ = 4,300 PSI

(SPECIMENS 60 MIN. TOTAL TIME AT HEAT TREAT TEMP.)