ACTIVATION ENERGIES FOR HIGH TEMPERATURE
CREEP OF MAGNESIUM-CADMIUM
POLYCRYSTALLINE ALLOYS

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by

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Submitted in partial fulfillment
of the requirements
for the degree of
MASTER OF SCIENCE
IN
MECHANICAL ENGINEERING

United States Naval Postgraduate School
Monterey, California

1 9 5 5

This work is accepted as fulfilling
the thesis requirements for the degree of
MASTER OF SCIENCE
IN
MECHANICAL ENGINEERING

from the
United States Naval Postgraduate School

PREFACE

Current practice in the engineering world is to demand more and more from the materials of construction in order to gain new increments of efficiency, speed, or fuel rate. Specifically, the engineer wants a lighter and stronger material which will withstand the higher temperatures that are required.

The onus is placed upon the research engineer and metallurgist to provide the advances in metals which will allow the developments proposed for the end use.

In order to provide the answers for these converging requirements a comparatively new field of high temperature metallurgy has emerged. It has been imperative to develop limited basic knowledge, theories, and experimental results into usable data in an expedient fashion. A part of this larger study is the investigation of the mechanism of creep phenomena.

The purpose of this thesis is to contribute to the data and fundamental understanding of the mechanism of creep of a polycrystalline solid solution alloy. The data here developed supplements the already extensive study made by the Institute of Engineering Research at the University of California in Berkeley sponsored by the Office of Naval Research.

The writer wishes to express his appreciation to Professor Alfred Goldberg for his interest and capable guidance which helped considerably in the accomplishment of this work.

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LIST OF ILLUSTRATIONS

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TABLE OF SYMBOLS

(Listed in the order of their use in the text)

$\dot{\epsilon}$ - Creep rate (sec^{-1})

S - Structure parameter (sec^{-1})

ΔH - Activation energy (calories per mole)

R - Gas constant (cal. mol.$^{-1}$ $^{\circ}$K^{-1})

T - Absolute temperature ($^{\circ}$K)

β - Constant (Square inches per pound)

σ - Stress (psi)

A - Constant (sec^{-1})

CHAPTER I

INTRODUCTION

Currently there is considerable interest in the behavior of metals under stress at high temperatures. The aeronautical field is interested in magnesium and its alloys. (1)The nuclear energy field is interested in cadmium and its alloys. (2)Essentially all research and development engineers have out of necessity become involved in the field of high temperature creep.

The experimental data developed to date are not sufficient to formulate rational laws governing high temperature behavior of stressed metals. (3)It is believed that if more were known of the mechanism of creep, which may be defined as the time dependent deformation of solids under stress, such knowledge and understanding could ultimately remove the limitations of empiricism and allow formulation of useful equations from fundamental understanding. Such equations based on short time test results could be used to predict long time behavior of stressed metals at high temperatures. To this end numerous mathematical expressions have been presented.(3, 4) These are of either an empirical nature or have as a basis Erying's Reaction Rate Equation.(5, 6)

Dorn and co-workers in investigating the plastic properties of high purity aluminum and its alloys met with considerable success in formulating several creep laws which are related to Erying's Reaction Rate Equation which necessarily implies that creep is an activation process. They develop the basic equation.(7)

$$\dot{\epsilon} = S e^{\frac{-\Delta H}{RT}} e^{\beta\sigma} \qquad (1)$$

where

$\dot{\epsilon}$ = creep rate (sec^{-1})

S = a parameter which is a function of the creep stress or strain (structure parameter) (sec.$^{-1}$)

ΔH = activation energy (cal.Mol.$^{-1}$)

R = the gas constant (cal.Mol.$^{-1}$ ^{o}K^{-1})

T = the absolute temperature (^{o}K)

β = constant (square inches per pound)

σ = stress (psi)

and showed that for constant load tests the equation reduces to$^{(8)}$

$$\dot{\epsilon} = A e^{\frac{-\Delta H}{RT}} \qquad (2)$$

where

A = constant (sec.$^{-1}$)

ΔH = constant independent of temperature over wide ranges of temperature (above that where rapid recovery occurs which is about 0.45 the melting temperature). It is also independent of creep stress, creep strain, grain size, sub-structures developed during creep, small alloying additions, and cold work.

Thus, the activation energy, ΔH, for creep may be determined by evaluating two creep tests at different temperatures T_1 and T_2 using the relation

$$\Delta H = \frac{R \ln \frac{\dot{\epsilon}_1}{\dot{\epsilon}_2}}{\frac{1}{T_2} - \frac{1}{T_1}} \qquad (3)$$

Activation energies have been determined for several pure metals in-

cluding cadmium(9) and magnesium(10) and reported to be respectively 22,000 and 31,000 calories per mole. Essentially no work has been reported showing the effect of solid solution alloying on this constant.(3) This thesis involves the experimental re-determination of ΔH for pure cadmium and magnesium and for five alloys across the cadmium magnesium binary system.

The results show that there is no simple systematic variation of ΔH as a function of composition. There are significant positive and negative deviations from linearity.

CHAPTER II

MATERIALS AND PRELIMINARY WORK

Specimens were machined from extruded bars 0.75 inches x 0.10 inches obtained from Dow Chemical Company. The nominal atomic percents for the alloys are given in Table I. The chemical analyses are listed in Table IV of Appendix I.

Table I

Nominal Atomic Percents For Metals

Alloy	Percent Cd.	Percent Mg
69	0	100
70	100	0
71	91.4	8.6
72	74.8	25.2
73	47.8	52.2
74	26.8	73.2
75	11.6	88.4

Preliminary to the creep experimental work it was necessary to study the effect of the proposed high temperatures on the structure and surface of the metals. By means of the Smally X-rays technique it was established that the materials were as extruded (not annealed) and that a preferred orientation existed. A microscopic study of the specimens revealed that the grain count on the surface normal to the direction of extrusion increased across the binary system from 80 grains per inch for cadmium to 700 grains per inch for magnesium. Although it was later proved that the

activation energy was independent of structure (for alloy 69) it was considered necessary to minimize the effect of grain size by recrystallizing the metals to obtain the same nominal grain count. Inasmuch as cadmium recrystallizes at room temperature and also had the largest grain size, this metal was selected as the standard. However, it seemed advisable to anneal cadmium to a temperature above that of the creep test so that the structure would not be altered prior to loading. With these two thoughts in mind it was experimentally determined that the alloys should be annealed at temperatures ranging from 200 to 500°C to give a nominal grain size of 100 grains per inch. Temperatures used and the grain size obtained are listed in Table II.

Table II

Summary of Annealing Temperatures and Grain Counts

Alloy	Annealing Temperature (°C)	Annealing Time (Hours)	Number (Grains/inch)
69(a)	As extruded	0	700
69(b)	300	1	450
69(c)	500	1	130
70	200*	1	80
71	216	1	103
72	220	1	100
73	362	1	75
74	450	1	60
75	500	1	110

*All specimens were furnace cooled except alloy #70 which was air cooled.

Difficulty was encountered with alloys 69(c) and 75 due to oxidation of the surface at the selected annealing temperature of 500°C. A coating of silicone oil was found to prevent oxidation of alloy #75. However, it was necessary to anneal pure magnesium (alloy 69(c) in an inert atmosphere. To obtain this, helium was passed continuously through copper chips heated at 600°C, then through anhydrous magnesium perchlorate chips, and into a tube furnace where the specimens were annealed. The heated copper chips removed traces of oxygen and the perchlorate chips removed any moisture present.

The dimensions of the specimens are shown in Figure 1. *Tolerences of ± 0.001" along the reduced section were obtained in machining; however, after annealing a total of 0.015" was tolerated in the extreme case.

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*Similar specimens were employed in the creep studies performed at the University of California.(11)

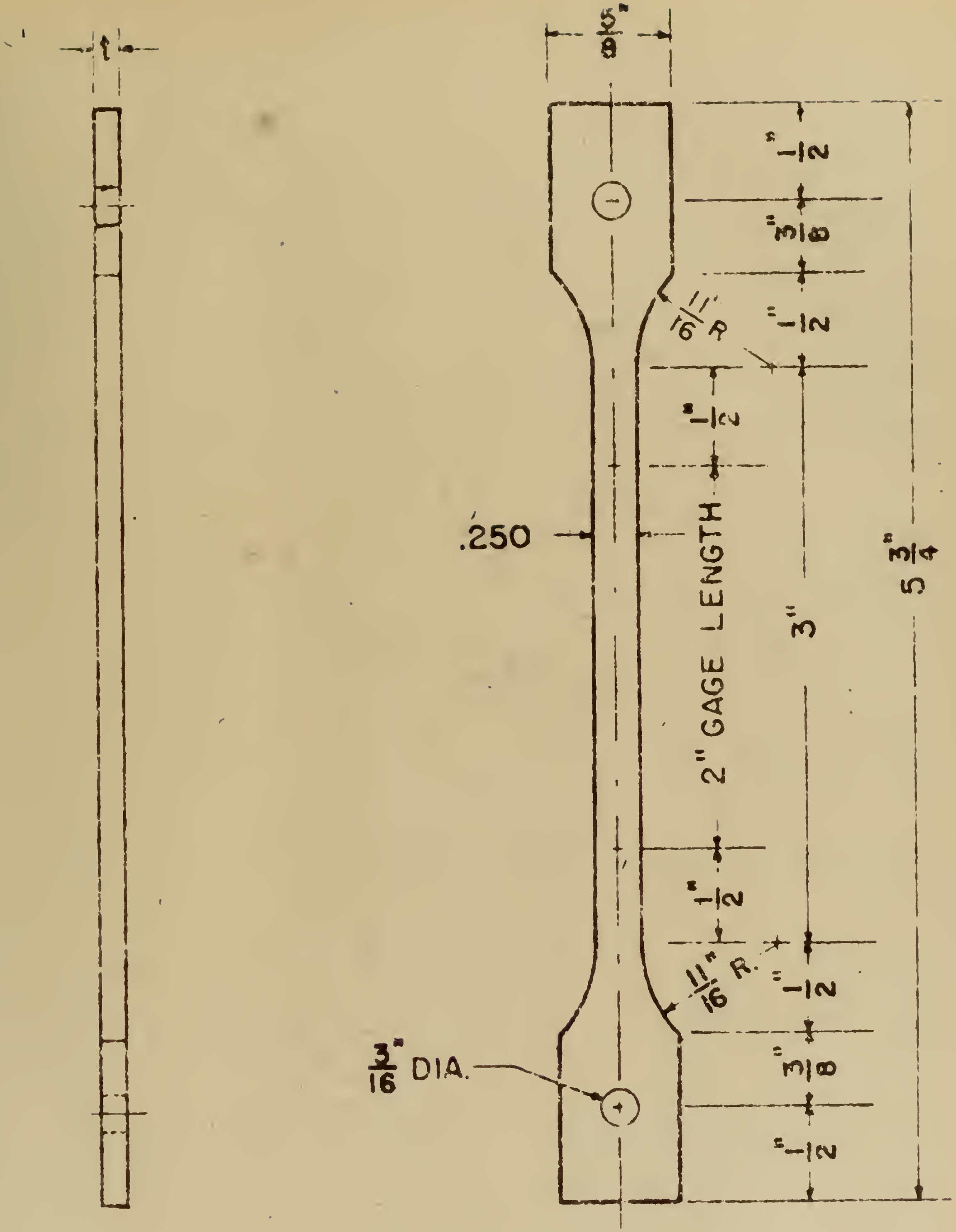
t
5/8"
1/2"
3/8"
11/16" R
1/2"
1/2"
.250
2" GAGE LENGTH
3"
5 3/4"
1/2"
11/16" R.
1/2"
3/8"
1/2"
3/16" DIA.

Fig. 1

CHAPTER III

EQUIPMENT

The test machine used for the creep experiments was a conventional single lever, constant load type manufactured by Baldwin-Lima-Hamilton Corporation. This machine was originally designed for testing heavier metals (6 ton capacity) and thus had to be modified to increase sensitivity for the lighter metals and to accomodate the extensometer. Figure 2 shows the experimental set-up. The alterations on the testing unit consist of the following:

(1) Addition of an adjustable counterweight to the lever arm in order to "zero" (balance the lever with the specimen installed and no applied stress) the machine with no load on the pan.

(2) Shortening the lever arm ratio to increase sensitivity.

(3) Addition of a gage to determine the angle of the lever during the test.

(4) Modification of the lower pulling tab connection to accomodate the reset turnbuckle.

The significant features of the machine are as follows:

(1) Loads are transmitted by means of 3 case hardened knife edges in the lever arm system.

(2) The lever arm assembly has a dowel pin at right angles to the direction of the knife edges thus giving the system three degrees of freedom.

(3) A balanced lever of this type if symmetrical compensates itself

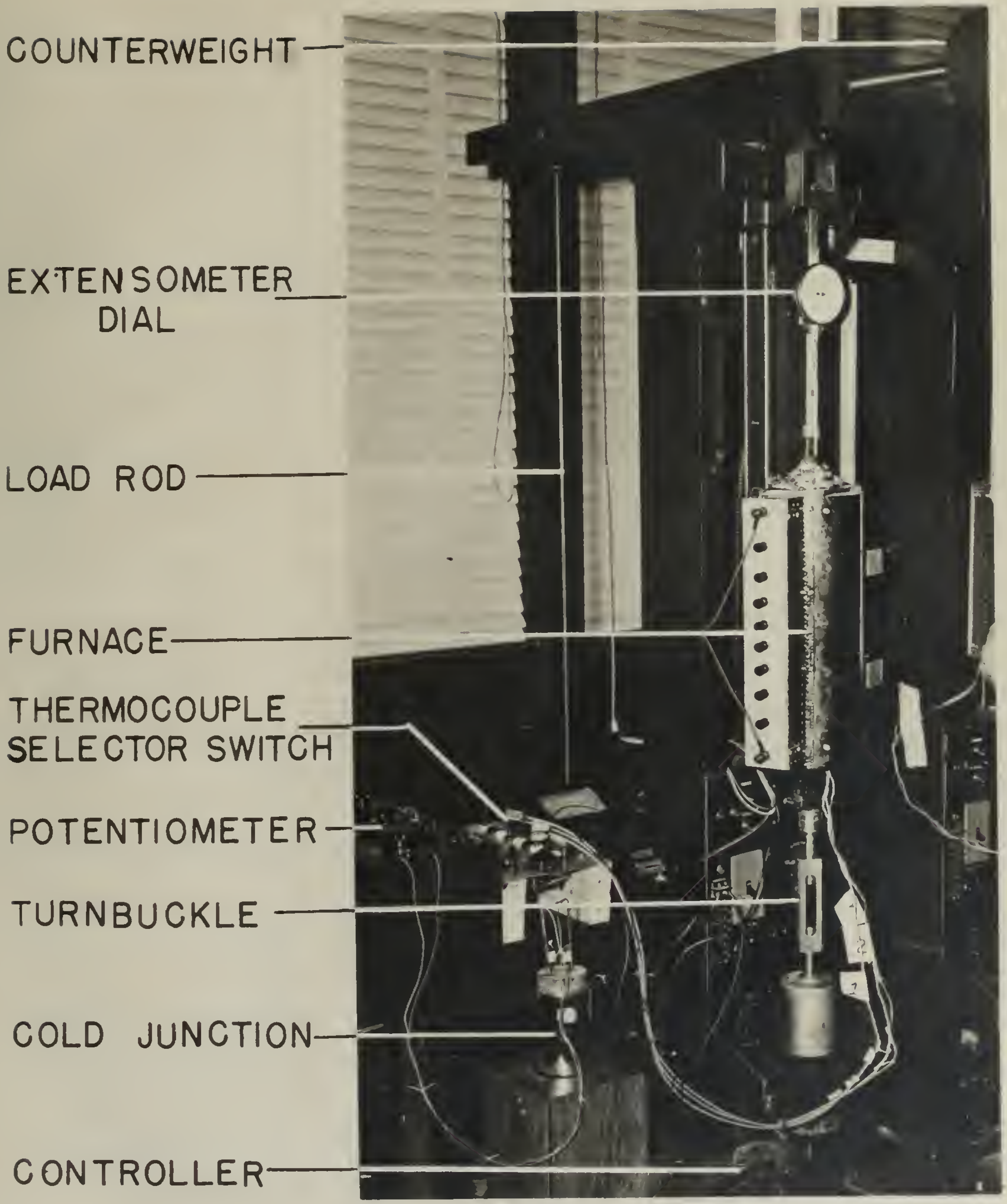

FIG. 2 THE EXPERIMENTAL SET UP

so that the lever ratio is the same regardless of the angle. Due to unsymmetrical cross section of the lever arm, the counterbalance which was in line with the knife edges could only be truly balanced in one position; however, the error is insignificant for small angles.

(4) The reset turnbuckle allows an initial adjustment of the lever before loading and reset after a specimen was elongated appreciably.

(5) The specimen is always uniaxially loaded which is an important prerequisite for accurate creep measurement.

The extensometer shown in Figures 3 and 4 was specifically designed for use with this creep machine and is suitable for the temperatures employed. The elements of the gage are shown in the figures while its features consist of the following:

(1) 2" gage length. This is accurate to +0.002", -0.000" by means of a detachable spacer.

(2) The gage blocks contain hardened steel points which penetrate the specimen 0.010" on one side and have a spring loaded clip on the other. This is necessary to accomodate for the reduced thickness of specimen as creep progresses.

(3) The upper gage block is rigidly connected to a tube which passes through a guide and finally attaches to the gage dial which is free to move vertically. The dial foundation has a guide slot for movement on the upper pull tab.

(4) The lower gage block is rigidly attached to a rod which moves through the above-mentioned tube and provides a resting platform for the dial gage stem which is spring loaded.

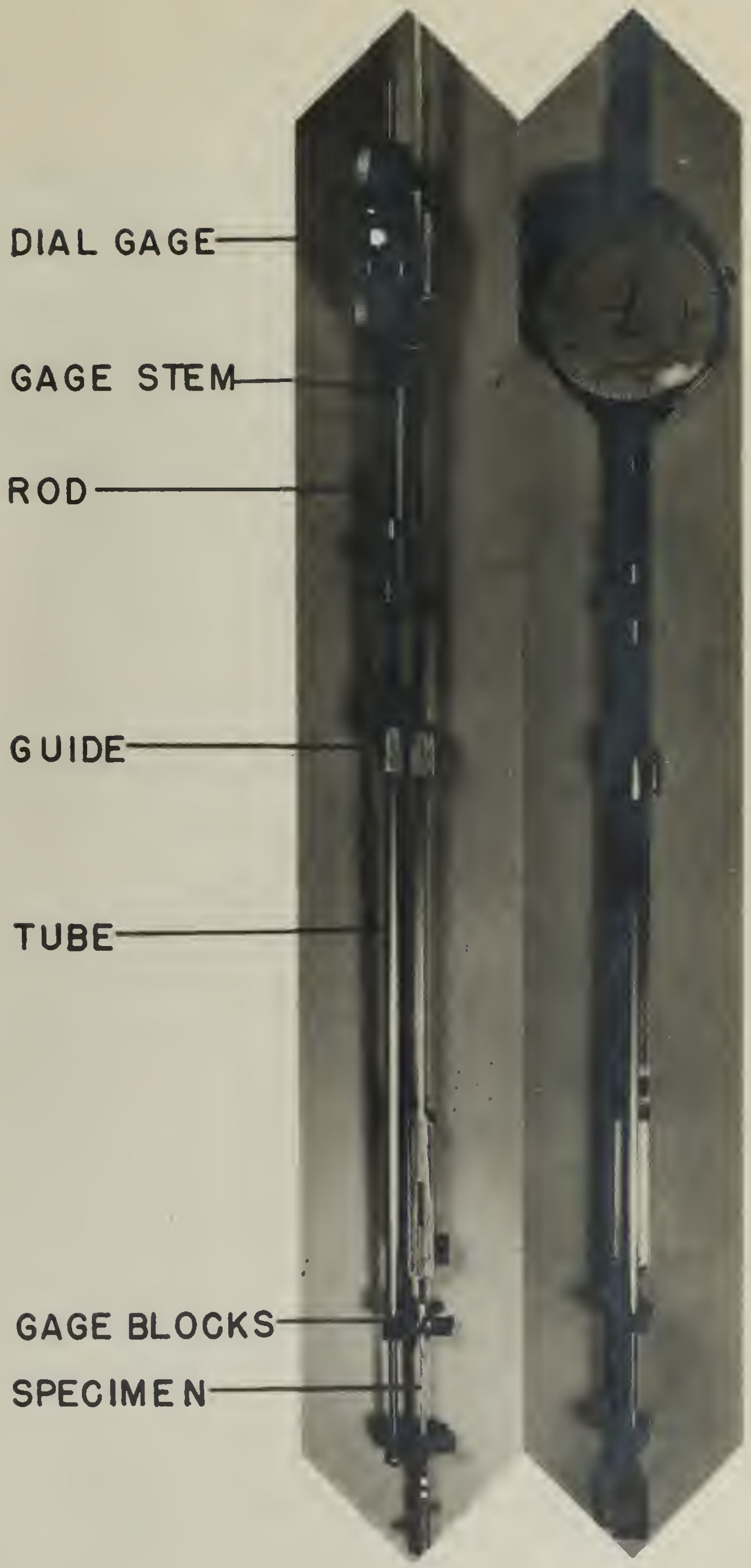

FIG. 3 THE EXTENSOMETER

(5) All high temperature components of the gage were made from stainless steel.

An L. H. Marshall 115V nichrome-wound cylindrical type furnace 16" long, 7" O.D. and 2" I.D. was used. Eight taps for shunting were provided for temperature control. However, it was not necessary to use these shunts because of the satisfactory temperature gradient obtained across the gage length of the specimen by careful insulation of the top and bottom. The desired temperature was obtained and kept constant by a variac with the aid of an ammeter.

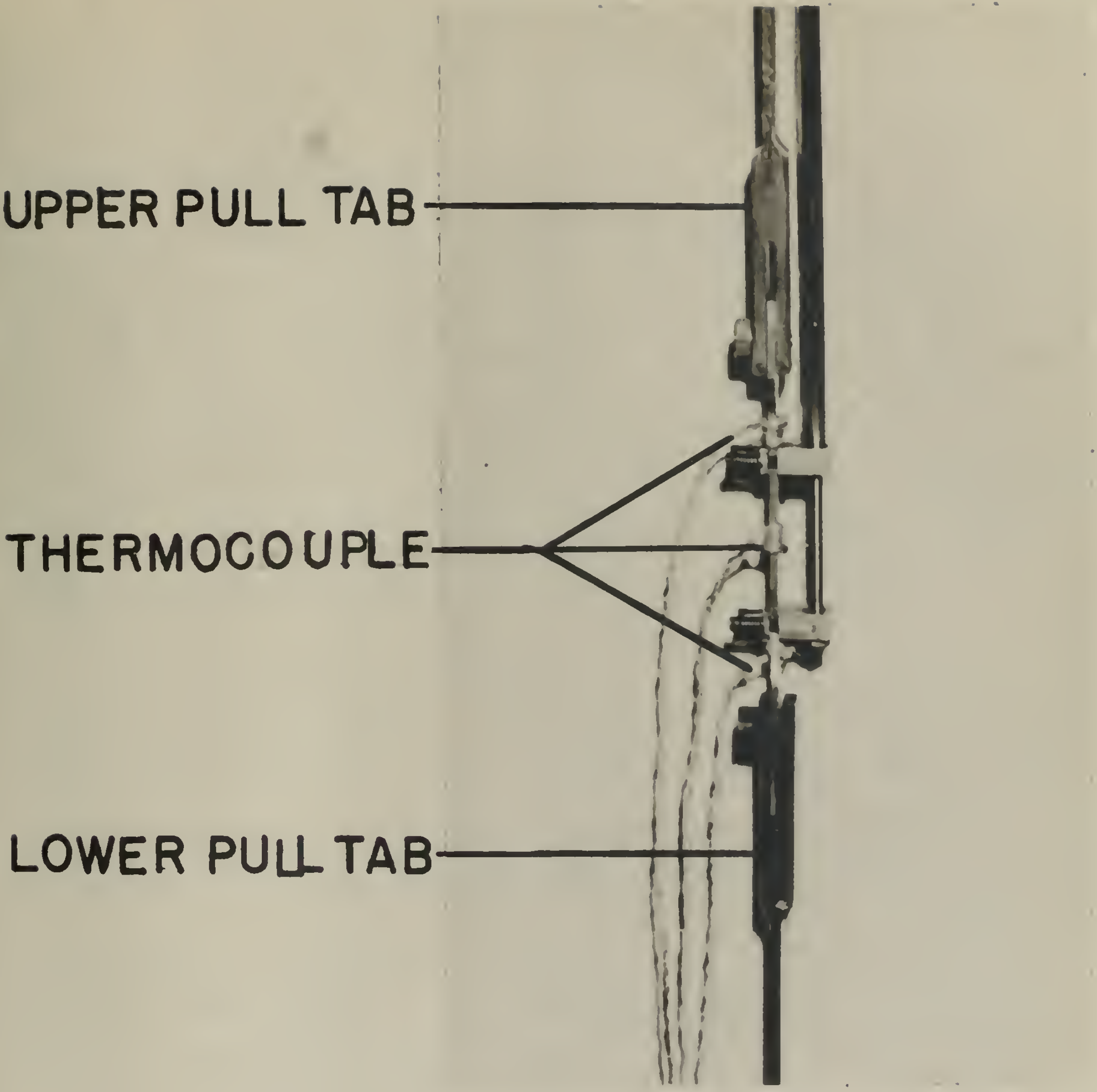

FIG. 4 THERMOCOUPLE-SPECIMEN ARRANGEMENT

CHAPTER IV

EXPERIMENTAL TECHNIQUE

A. Preliminary:

In high temperature creep work of this nature there are four main factors to consider. They are stress, strain, temperature, and material.

Inasmuch as these experiments are of the constant load type only the initial stress is of concern. In uniaxial loading the measured area is multiplied by the desired initial stress to determine the loading. This load when divided by the mechanical advantage of the lever establishes what weight shall be added to the loading platform.

Strain rate varies exponentially with applied stress (Equation 1) and must be carefully controlled. The test machine lever arm ratio was calibrated with a Baldwin SR-4 Type U (0-2000 lbs) standard load cell which has an inaccuracy of ± 1/4% of full range at a point. The lever arm ratio was found to be 14.75:1. With this instrument, it was demonstrated that the lever gave no detectable loading error within the range of ± 7.5 degrees from the horizontal. Because all the experimental work for this thesis was done on the same test machine and was of the constant load nature in which tests of identical loads were compared, errors that might have arisen by the machine would be of a systematic nature and automatically cancelling. Thus, the errors introduced in controlling stress are reduced to the errors of determining a representative cross sectional area of the specimen and measuring the applied weights. The representative sectional area was determined by multiplying an average of three widths by an average

of three thicknesses. Weights were measured on a balance accurate to 2×10^{-3} lbs. Combining these extreme errors a stress variation of less than 10 psi would result.

The extensometer was calibrated by mounting in a tension-testing machine with a calibration specimen with SR-4 gages between the gage points on both sides of the specimen. The extensometer was found to be accurate to its sensitivity of 2×10^{-4}.

A substantial part of the preliminary work on this thesis was devoted toward getting satisfactory temperature control in the furnace and a satisfactory gradient across the specimen. For constant stress, the creep rate varies exponentially to $^1/T$. It is mandatory to minimize the temperature gradient across the gage length in order to get accurate data. Because of the nature of the ΔH equation (Equation 3) it is essential to determine the temperature accurately. This was done by means of three duplex fiberglass insulated chromel-alumel thermocouples attached with glass thread to the specimen at the center and just outside of the gage blocks as shown in Figure 4. The thermocouple loads were led through a cold junction (ice water) to a selector switch connected to a potentiometer. This thermocouple arrangement was calibrated at $0^\circ C$ and gave readings of $\pm$ $0.75^\circ C$ for each thermocouple.

The furnace temperature could be controlled automatically by putting the center thermocouple to the controller input; however, cyclic variations of $5^\circ C$ were obtained. This could be improved by the proper amount of resistance placed in parallel with the control relay. However, the voltage variation over a 24 hour period was of the order of 10 percent which would

again broaden the temperature variation. Thus, better control was exercised by establishing the required constant current to maintain the desired temperature and then noting the ammeter and constantly changing the variac to maintain a constant ammeter reading. Utilizing this technique the gradient across the specimen was reduced to $\pm$ 2°C and the average temperature variation during the critical part of the run was less than 0.5°C. It is estimated that the variation and uncertainty in temperature introduced an error of less than 3 percent in the determination of ΔH .

B. Test Procedure:

The determination of an activation energy using Equation 3 requires that two creep tests be run at two different temperatures under the same initial load. The problem then was to determine the load and temperatures such that sufficient experimental points could be obtained at the high and low temperatures within about five minutes and twenty hours respectively. This often required frequent preliminary runs as each new alloy was tested.

The recrystallized specimen was mounted in the extensometer and thermocouples attached (see Figure 4). The furnace was brought into position and insulated at the top and bottom with aluminum foil and felt. The furnace was then turned on and allowed to come up to temperature. The test was started after the desired equilibrium conditions were established. This took from three to five hours after the power was turned on. An automatic timer was used for the rapid test.

The low temperature test (50 - 100°C less) was generally discontinued after reaching a strain of about 6 percent, the important part of the test being in the region of 3 to 6 percent extension where frequent readings

were taken and fine temperature control maintained.

The data from the two runs were reduced and plotted on strain-time coordinates. The creep rates ($\dot{\epsilon}$) were obtained by drawing tangents to the curve at a given strain. The exact temperatures at the time corresponding to this strain were used. Thus the four unknowns of Equation (3) are available for evaluation.

In the experimental work on this thesis satisfactory results were obtained when two high temperature runs gave essentially the same result with one low temperature run.

ΔH was determined for the seven metals shown in Table III. Three separate ΔH values were determined for alloy #69 to demonstrate that ΔH is essentially independent of structure.

C. Sample Calculation -

(1) To calculate ΔH it is necessary to substitute a set of experimental values into Equation (3),

$$\Delta H = \frac{R \ln \left(\frac{\dot{\epsilon}_1}{\dot{\epsilon}_2} \right)}{\frac{1}{T_2} - \frac{1}{T_1}} = \frac{T_1 T_2 R \ln \left(\frac{\dot{\epsilon}_1}{\dot{\epsilon}_2} \right)}{T_1 - T_2}$$

Consider the isothermal strain-time curves of Figure 6 where,

$T_1 = 574^\circ K$

$T_2 = 473^\circ K$

By evaluating the slope at some particular strain say,

$\epsilon = 40 \times 10^{-3}$

the creep rates are found to be -

$$\dot{\epsilon}_1 = 177 \times 10^{-6}/\text{Sec}$$

$$\dot{\epsilon}_2 = 0.486 \times 10^{-6}/\text{Sec}$$

$$\Delta H = \frac{(574)(473)R \ln\left(\frac{177}{0.486}\right)}{574 - 473}$$

$$= 30{,}500 \text{ cal/mole}$$

(2) To calculate $\dot{\epsilon}$ standardized to 200°C at 2000 psi consider Equation 3 rearranged,

$$\ln\left(\frac{\dot{\epsilon}_1}{\dot{\epsilon}_2}\right) = \frac{\Delta H (T_1 - T_2)}{R\ T_1\ T_2}$$

Now consider the data of Figure 8 where

$$T_1 = 559^\circ K$$

$$\dot{\epsilon}_1 = 97.3 \times 10^{-6}/\text{Sec}$$

Let the standardized temperature = 473°K and ΔH = 30,000 cal/mole then;

$$\ln \frac{97.3 \times 10^{-6}}{\dot{\epsilon}_2} = \frac{(30{,}000)(86)}{(2)\ (473)\ (559)} = 4.89$$

$$\dot{\epsilon}_2 = \frac{97.3 \times 10^{-6}}{133} = 0.732 \times 10^{-6}\text{Sec}^{-1}$$

CHAPTER V

EXPERIMENTAL RESULTS

The activation energies for the metals tested are reported in Table III and are shown graphically in Figure 5. Typical strain vs time curves for each alloy used for determining ΔH are given in Appendix II, Figures 6-16.

Table III

Experimentally Determined Activation Energies For the Cd-Mg Binary System

Alloy No.	Magnesium (Atomic %)	Cadmium (Atomic %)	Temperature Annealed (°C)	Activation Energy (cal/mole)
69	100	0	As Extruded	32,400
69	100	0	As Extruded	30,500 (Fig 6)
69	100	0	300	30,300 (Fig 7)
69	100	0	300	30,300
69	100	0	500	30,300 (Fig 8)
69	100	0	500	29,800
75	88.4	11.6	500	38,500 (Fig 9)
75	88.4	11.6	500	39,200
74	73.2	26.8	475	26,100 (Fig 10)
74	73.2	26.8	475	25,900
74	73.2	26.8	450	26,400 (Fig 11)
73	52.2	47.8	350	33,600
73	52.2	47.8	350	33,900
73	52.2	47.8	380	71,700 (Fig 12)
73	52.2	47.8	380	57,600 (Fig 13)
72	25.2	74.8	220	18,500
72	25.2	74.8	220	18,400 (Fig 14)
71	8.6	91.4	216	23,300 (Fig 15)
71	8.6	91.4	216	23,300
70	0	100	200	22,100 (Fig 16)

CHAPTER VI

DISCUSSION OF RESULTS

The results show an absence of any systematic variation of ΔH across the Mg-Cd system. However, the good agreement of the results on both pure Cd and Mg with those reported in the literature (9, 10) gives confidence to the other determinations in this thesis. It is significant that the activation energies for Cd and Mg are insensitive to minor differences in metal preparation, purity, and structure. The duplication of ΔH for Mg in the "as extruded", annealed to 300°C and 500°C (grain sizes in Table II) implies that for this pure metal ΔH is essentially independent of grain size and small amounts of strain.

An attempt was made to obtain identical grain sizes (recrystallized) for all alloys. The small variations in the final grain sizes used should not affect the ΔH values. It is thus felt that the results shown in Figure 5 are real. In what follows is an attempt to delve into some of the possibilities that might account for these values.

Jette (12) in analyzing Vegard's law of additivity states that negative and positive deviations from linearity exist due to interactions which are both chemical and physical in nature. These deviations might well be expected after considering the extraordinary differences in the component metals of this solid solution. The major differences of this system follow;

(1) significant difference (8%) in atomic radii thus resulting in distortional strain energies.

(2) high (2 volts) difference in electrochemical negativity giving strong interaction associated with tendency toward compound formation.

(3) high exothermic heat of formation (4.6 K cal./g. atom) compared to other solid solutions (the order of ± 1.0 K cal./g. atom) which is due to strong interaction and can be related to negative deviations of the atomic radii based on the law of additivity.

The interaction should give a larger ΔH and thus a positive deviation from the straight line joining the values for the two pure metals. The greatest effect of interaction per atomic percent added would be felt on the first addition of the solute to the host metal; namely at 10% on either side of the Cd-Mg system (the lowest alloy compositions studied). This was true for the 10% Cd alloy only.

The results of the 25% Cd, and 75% Cd are unexpected and difficult to explain; however, the drop is not large. On the other hand variable results were obtained with the 50% Cd alloy.

An examination of the phase diagram shows the existence of an ordered structure, γ , below 250°C for the 50% composition. (This also corresponds to the maximum temperature).

The lowest ΔH value reported for this composition was obtained from tests run in this temperature range. Additional tests were run at temperatures above 250°C (where the random structure, α , exists.) Two sets of tests gave surprisingly high and different ΔH values. The reliability of the data on the one hand and the great spread of values for the 50% Cd alloy on the other hand suggests that the creep mechanism is in some manner affected by the disorder-order reaction taking place (slowly)

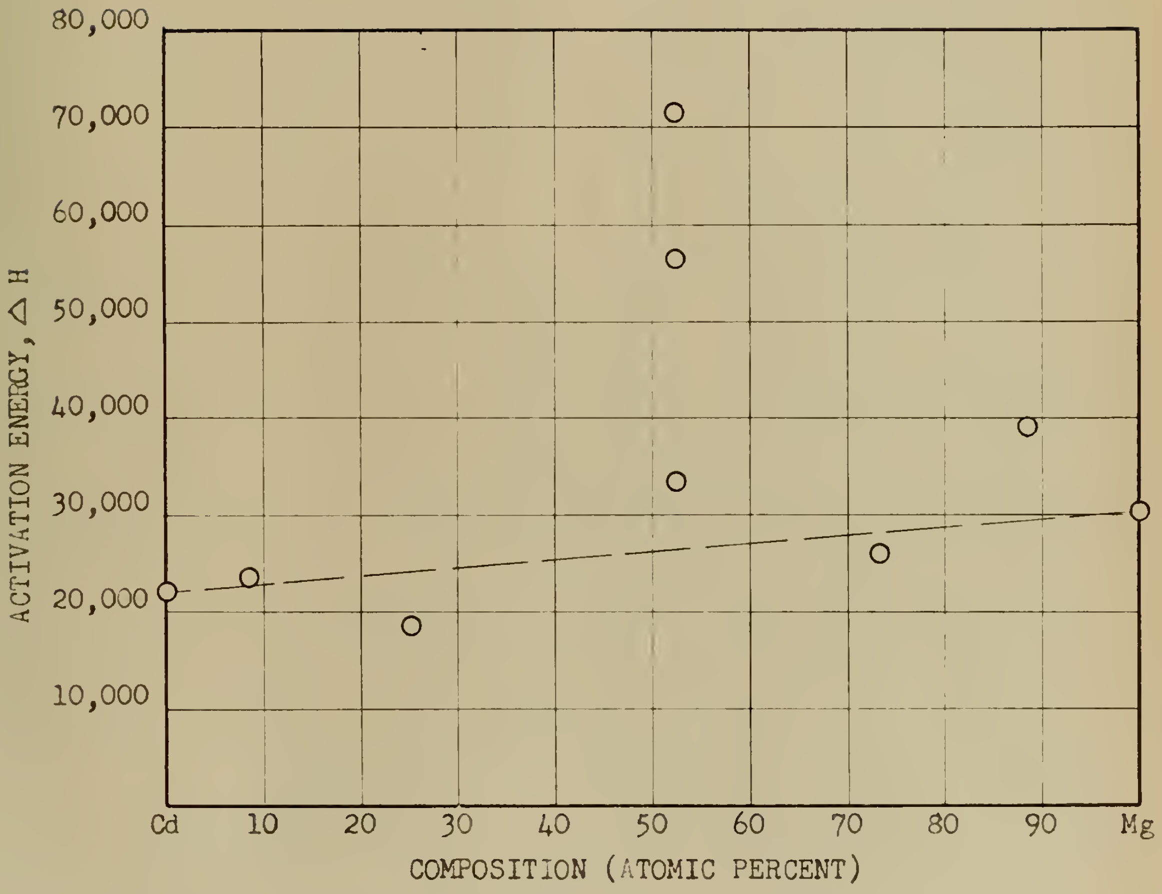

FIG. 5 ACTIVATION ENERGY vs COMPOSITION FOR THE Cd - Mg SYSTEM

in this composition. It is felt that additional study of this composition would be of benefit in furthering our knowledge not only of creep but also on the order-disorder reaction and its influence on creep.

It might be of interest to note that the 50-50 alloy showed the greatest creep resistance. The creep rate for all compositions stressed at 2000 psi were standardized to 200°C. A plot of ln $\dot{\epsilon}$ vs alloying is shown in Figure 5A. As a reference, a straight line joins the creep rate of the pure metals (this supposes that the increase (or decrease) in rate is proportional to a composition change). A comparison between Figures 5 and 5A shows that negative deviations from $\dot{\epsilon}$ (increased strength) and positive deviations from ΔH (increased bonding interaction) are consistent, thus giving further evidence to the validity of the data obtained. Again, the abnormal behavior of the 50% Cd alloy is exhibited in that a significant deviation (increased creep resistance) as might have been expected was not obtained.

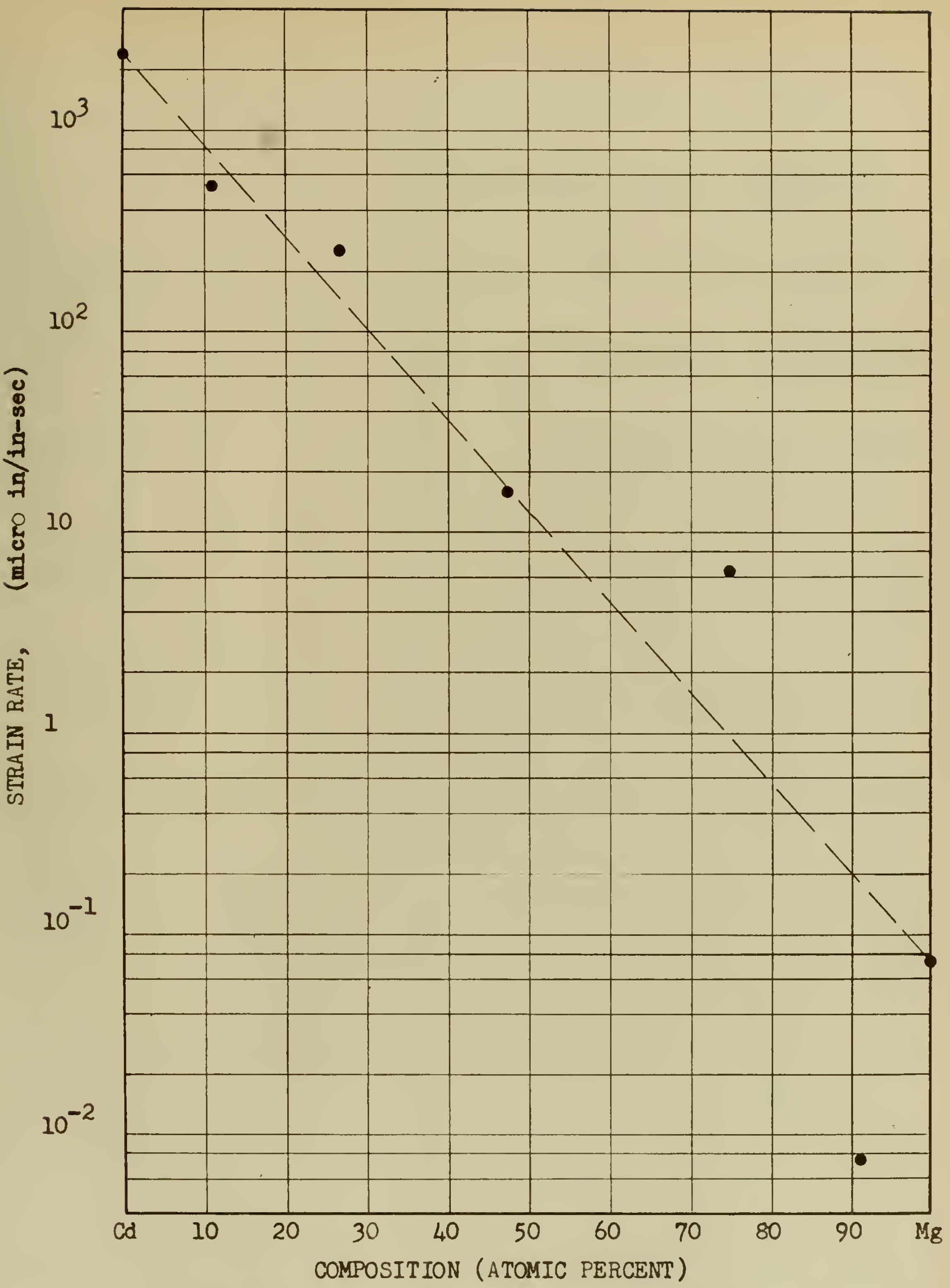

FIG. 5A STRAIN RATE vs COMPOSITION FOR THE Cd - Mg SYSTEM
Standardized to 200°C at 2000 psi

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APPENDIX I

Table IV

Chemical Analysis of Metals in Percent by Weight

Alloy No.	Percent Mg (analyzed)	Percent Cd (by subtraction)
70	0	100*
71	1.98	98.02
72	6.79	93.21
73	19.15	80.85
74	37.08	62.92
75	62.35	37.65
69	100*	0

*The cadmium magnesium alloys were prepared from 99.9 pure cadmium. The impurity analysis of the magnesium follows:

Impurity	Percent
Al	0.012
Ca	< 0.01
Cu	< 0.001
Fe	< 0.001
Mn	0.001
Ni	< 0.001
Pb	0.005
Si	0.022
Sn	< 0.01
Zn	0.052

APPENDIX II

Typical Strain vs Time Curves
for each ΔH Determination (Figs. 6 - 16)

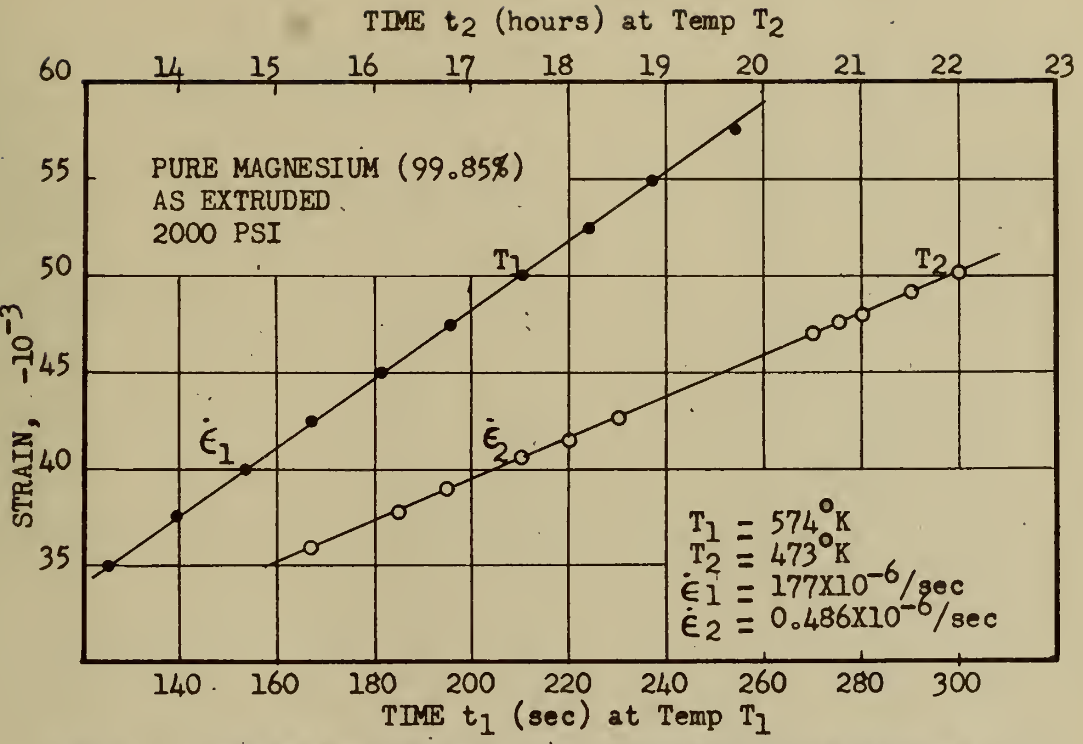

FIG. 6 STRAIN vs TIME FOR 99.85 PERCENT PURE MAGNESIUM (AS EXTRUDED)

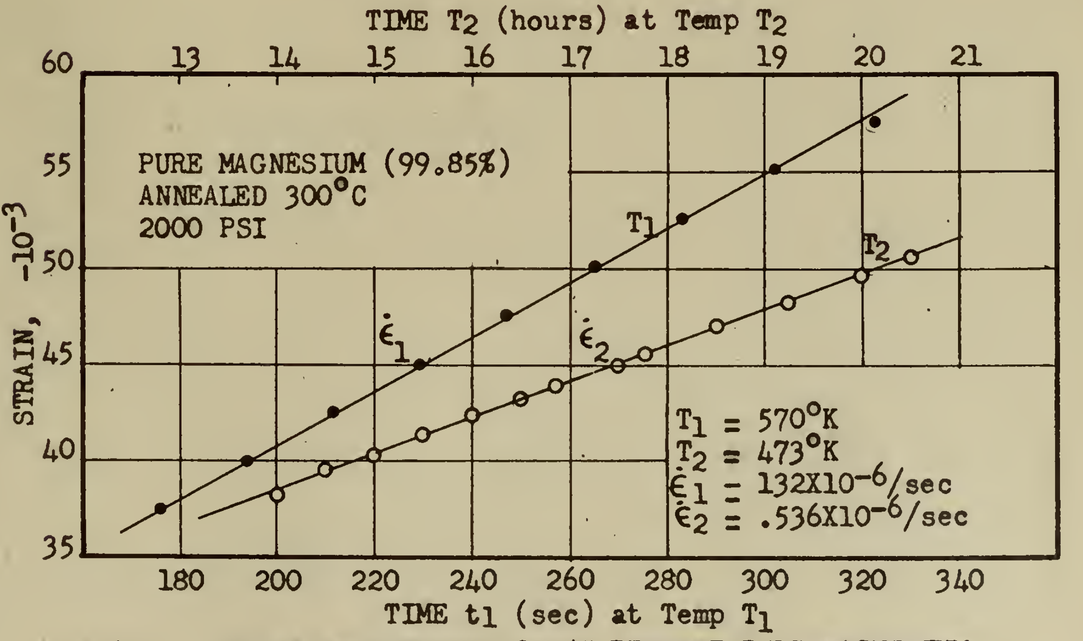

FIG. 7 STRAIN vs TIME FOR 99.85 PERCENT PURE MAGNESIUM (ANNEALED 300°C)

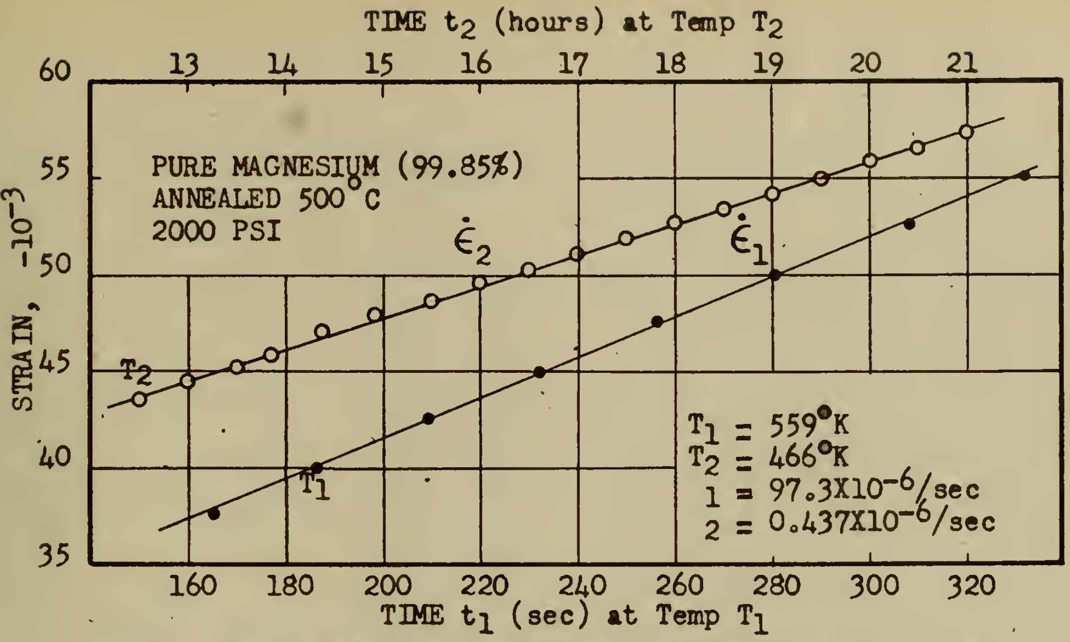

FIG. 8 STRAIN vs TIME FOR 99.85 PERCENT PURE MAGNESIUM (ANNEALED 500°C)

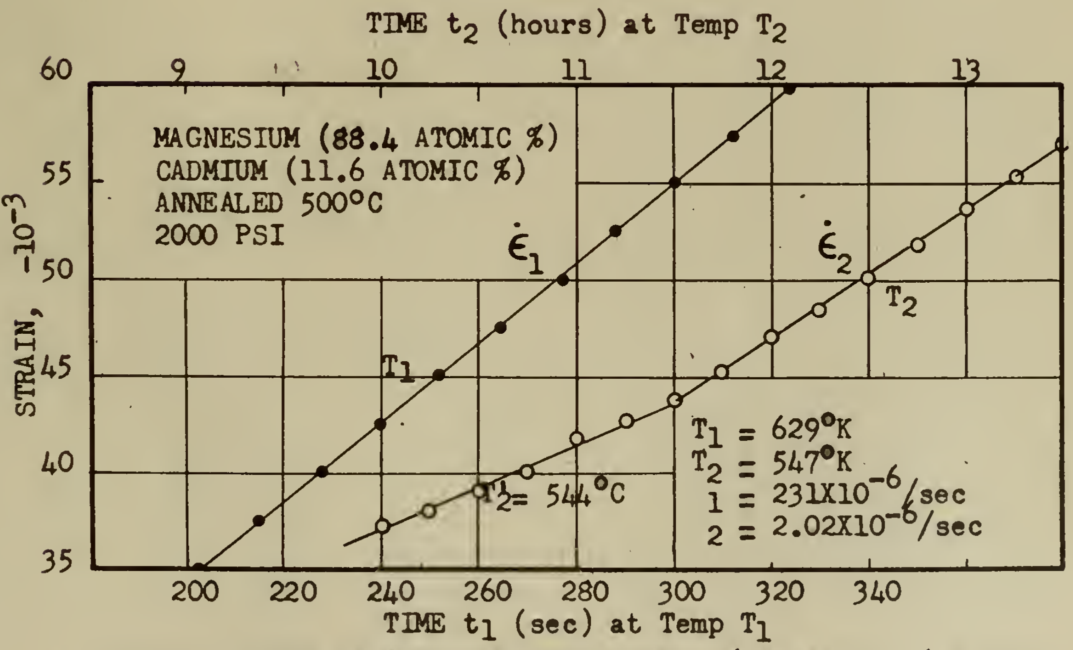

FIG. 9 STRAIN vs TIME FOR 88.4 ATOMIC % Mg and 11.6 ATOMIC % Cd ANNEALED 500°C

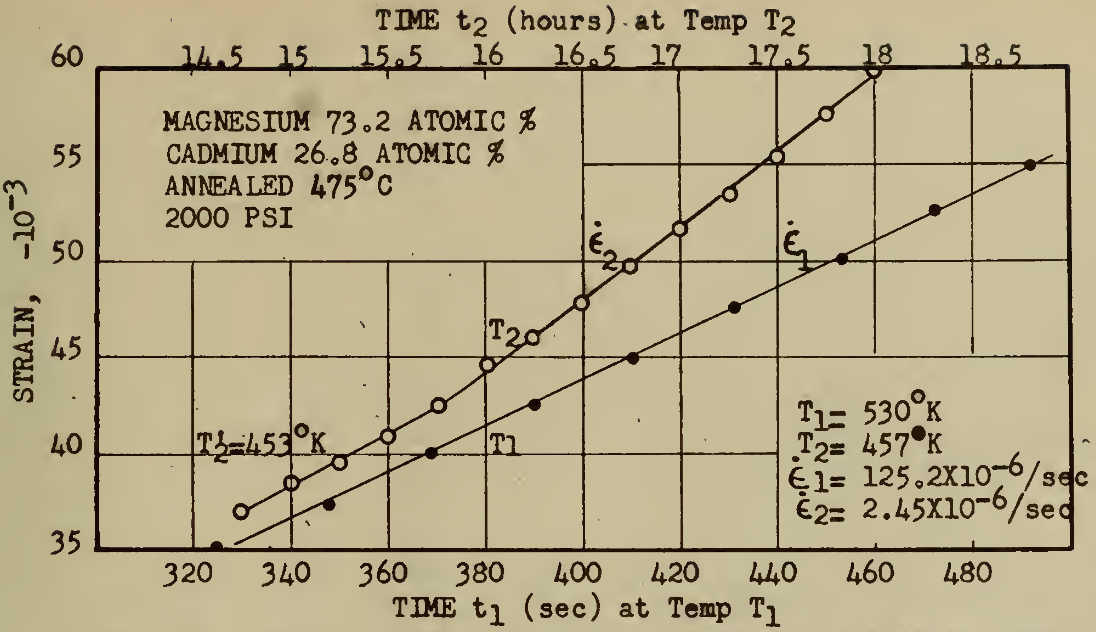

FIG. 10 STRAIN vs TIME FOR 73.2 ATOMIC % Mg and 26.8 ATOMIC % Cd ANNEALED 475°C

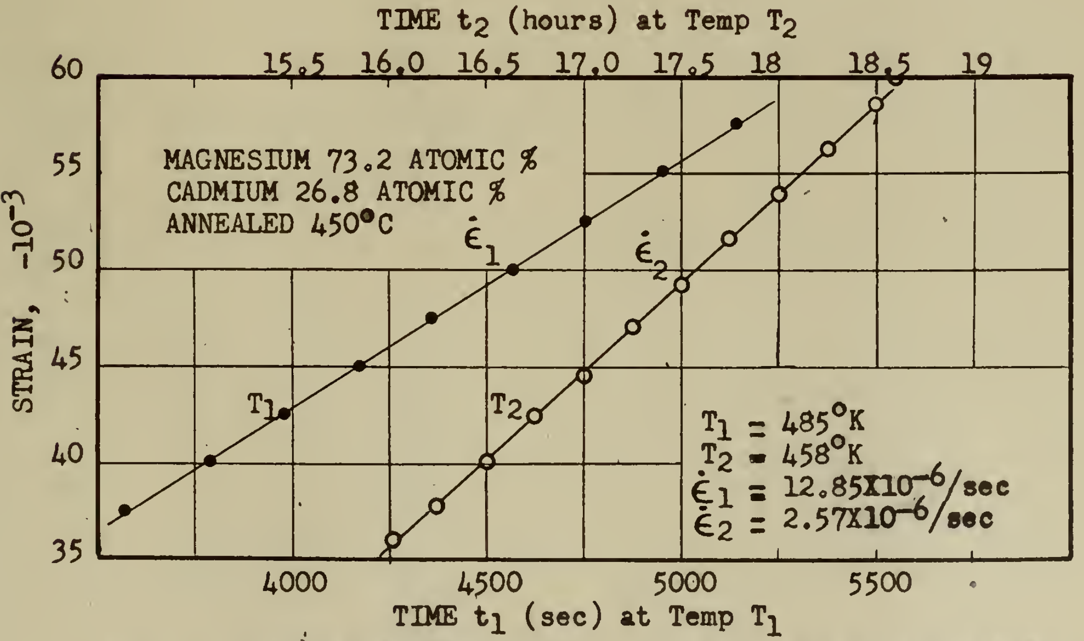

FIG. 11 STRAIN vs TIME FOR 73.2 ATOMIC % Mg and 26.8 ATOMIC % Cd ANNEALED 450°C

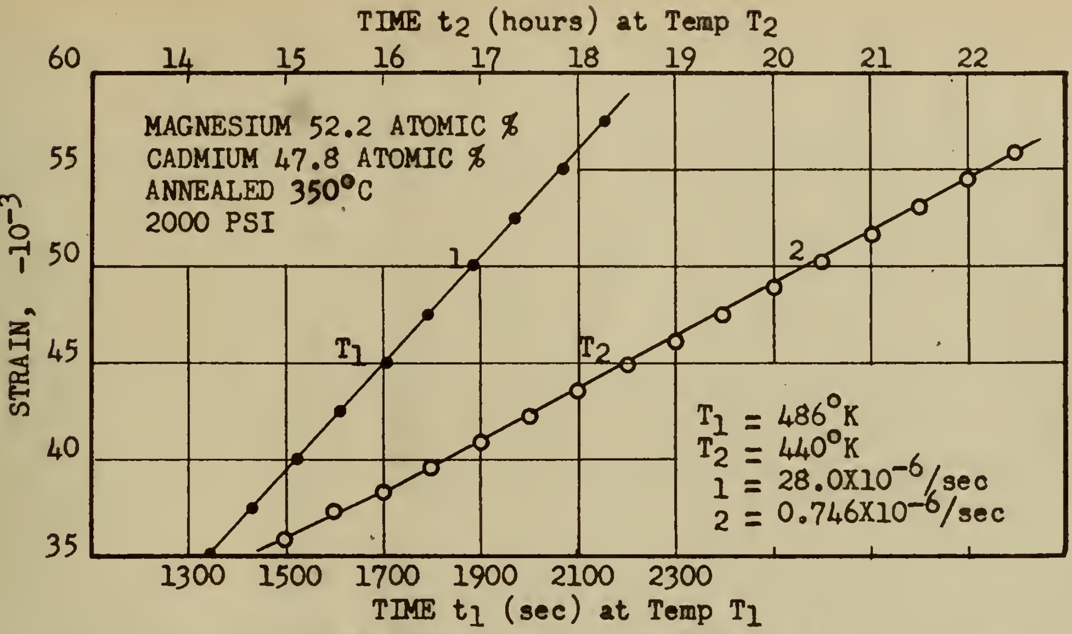

FIG. 12 STRAIN vs TIME FOR 52.2 ATOMIC % Mg and 47.8 ATOMIC % Cd. ANNEALED 350°C

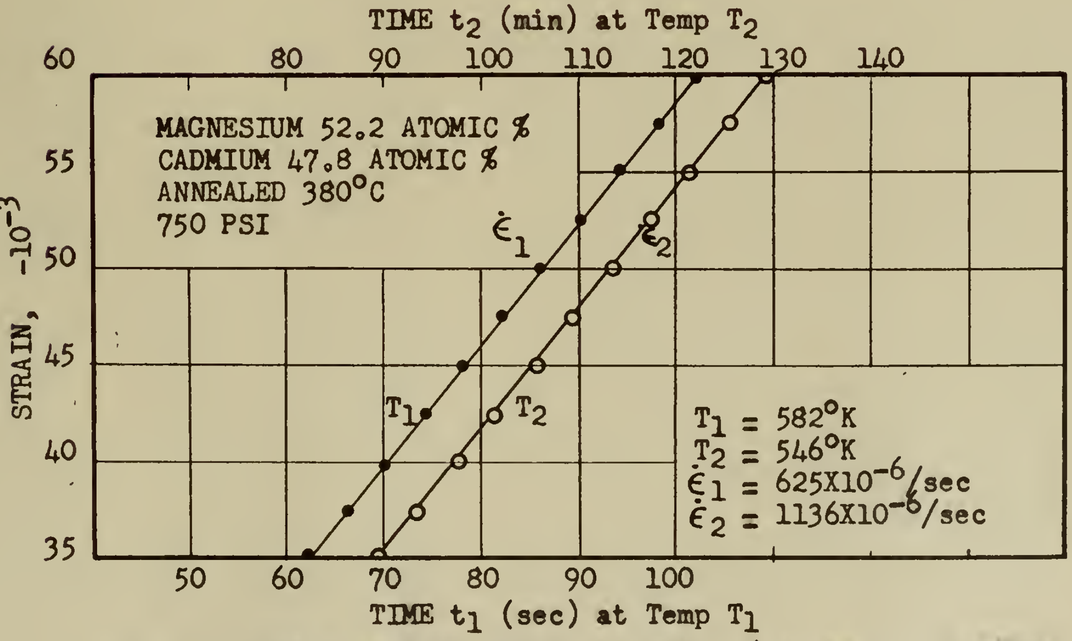

FIG. 13 STRAIN vs TIME FOR 52.2 ATOMIC % Mg and 47.8 ATOMIC % Cd ANNEALED 380°C

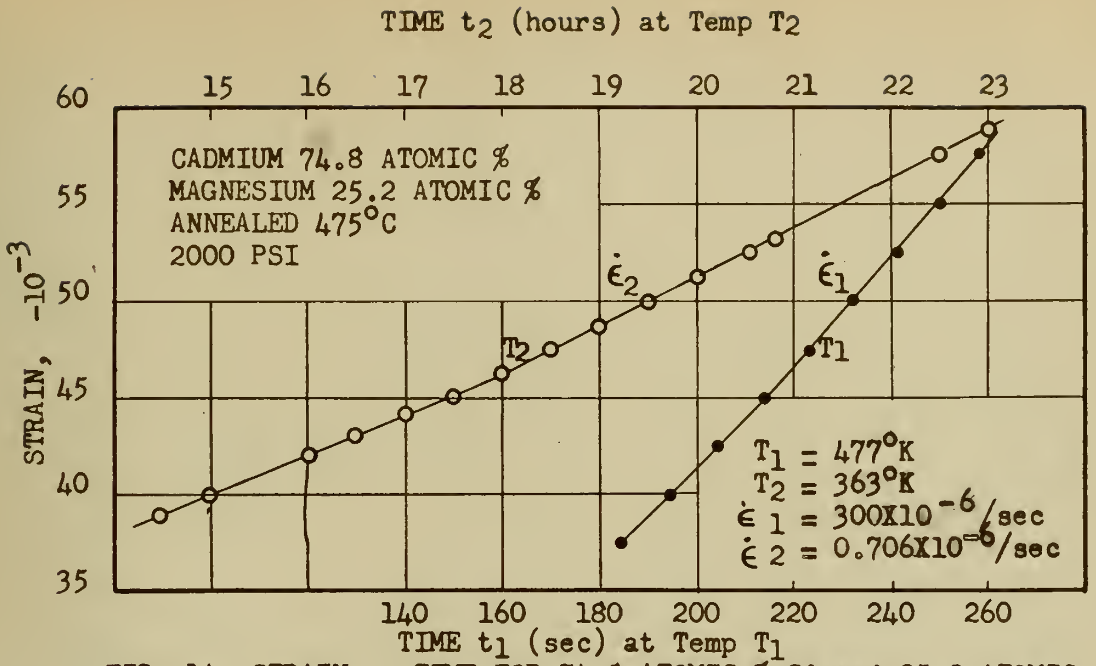

FIG. 14 STRAIN vs TIME FOR 74.8 ATOMIC % Cd and 25.2 ATOMIC % Mg

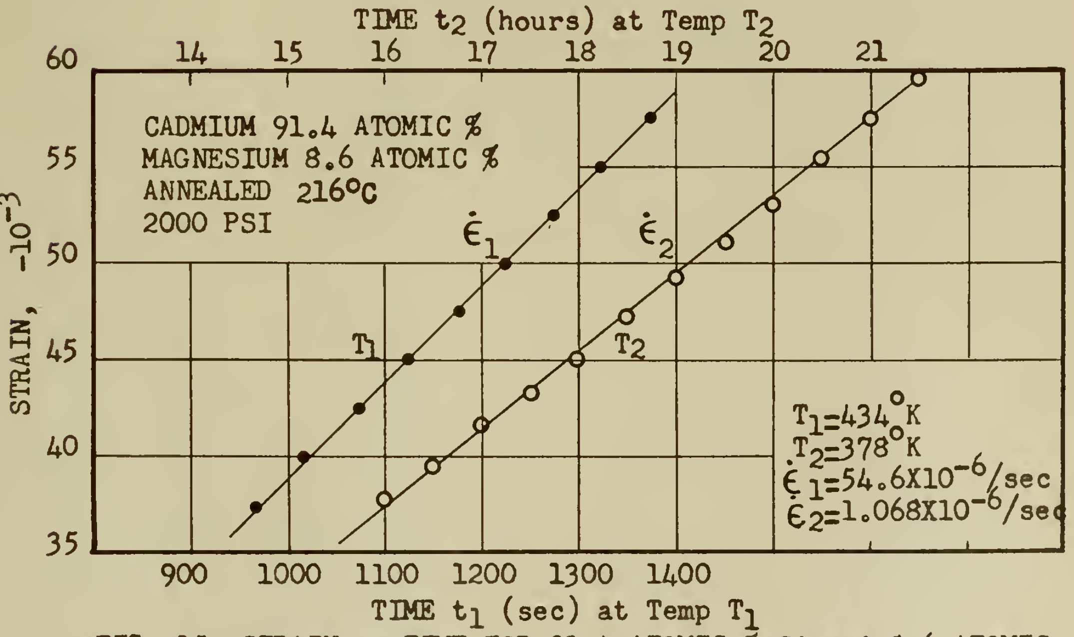

FIG. 15 STRAIN vs TIME FOR 91.4 ATOMIC % Cd and 8.6 ATOMIC % Mg

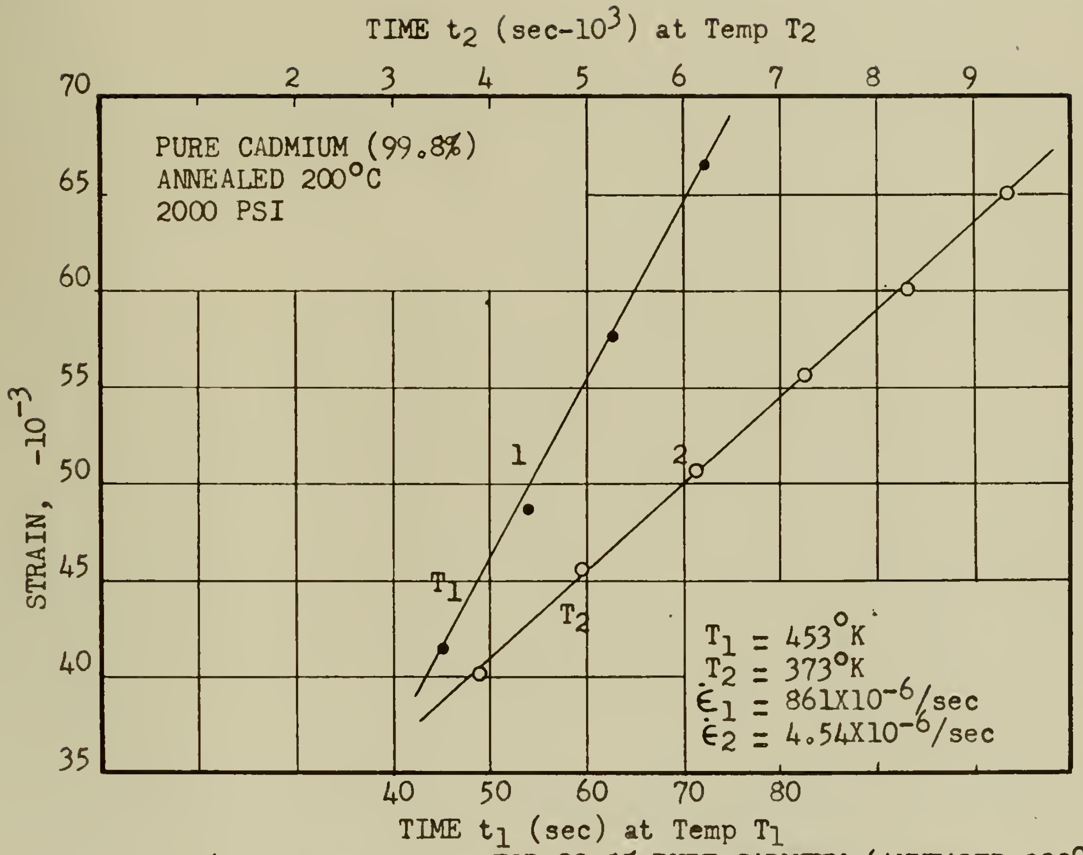

FIG. 16 STRAIN vs TIME FOR 99.8% PURE CADMIUM (ANNEALED 200°C)

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