THE UNIVERSITY OF MICHIGAN

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THE DETERMINATION OF CREEP-INDUCED

STRUCTURAL CHANGES BY DIFFERENTIAL

DENSITY MEASUREMENTS

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ABSTRACT

Precise measurements have been made of the density changes accompanying stressed and unstressed exposure of two alloys at elevated temperatures. The two materials used in this study differed widely in their behavior during creep-rupture testing. One alloy, Chromel A, was subject to extensive crack formation and growth during stressed exposure and it exhibited large density decreases at high percentages of the anticipated rupture life. The second alloy, Rene' 41, exhibited very little evidence of cracking during creep-exposure. The density of this alloy changed only slightly during elevated temperature stressed exposure.

Measurements of density change during unstressed exposure showed Chromel A to exhibit significant density increases at 1000°, 1200° and 1400°F, the increases being greatest at the higher temperatures. This density increase may possibly be associated with an ordering reaction or carbide precipitation. Rene' 41 was shown to be highly sensitive to cooling rate from the exposure temperature, much more sensitive to this than to time of exposure at that temperature. This behavior probably resulted from the rapid γ' precipitation reaction active in the alloy.

The measured density changes were highly sensitive to the structural processes accompanying creep. The degree of sensitivity obtained was much greater than that associated with other techniques such as metallographic or x-ray examinations.

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

LIST OF FIGURES

LIST OF FIGURES (Continued)

INTRODUCTION

This report presents the results of an investigation initiated to determine the possibility of using very sensitive measurements of density change as a method of evaluating the processes which occur in alloys during creep exposure.

Engineers have long sought a practical method of determining the degree to which creep-rupture properties are "used up" during service at elevated temperatures. In addition there is a need for a test to estimate the influence of high temperature service on other mechanical properties. In both cases the deterioration of properties has been loosely referred to as "creep damage".

Creep damage can be considered to arise from three causes (Ref. 1):

1. The plastic flow of creep itself.
2. Thermally-induced microstructural changes.
3. Surface attack by the environment.

The damage associated with the plastic flow of creep has not been adequately determined. The most prevalent theory is that plastic flow is associated with the formation of voids and cracks in the grain boundaries. It is known, however, that creep life can be used up before voids or cracks have been detected. Preference is given to void and crack formation and growth as an explanation for permanent damage because these seem most consistent with the common finding that creep-rupture life cannot be restored by re-heat treatment. Voids and cracks would be expected to reduce density. There is a possibility that high dislocation densities which may precede crack formation might also reduce density. In any event, very sensitive measurements of changes in density offer the possibility of serving as a measure of damage resulting from the creep process.

Thermally-induced microstructural changes also can cause density changes. The present experiments were conducted so that unstressed specimens were exposed along with the stressed creep-rupture specimens. The difference in density between the two types of exposure was assumed to be due to the plastic flow of creep. It was recognized that stress (or strain) induced microstructural changes could occur in the creep specimens which would not occur in the unstressed specimens.

The difference in density between the original heat-treated materials and the specimens after unstressed exposure were also measured. This was done

with the expectation that such data might be interpreted in terms of the thermally-induced microstructural changes accompanying and influencing creep.

Surface attack was eliminated by machining off the surface of specimens after exposure. It was considered that environmental effects were too variable for consideration in this limited program.

For these exploratory experiments an alloy known to be subject to extensive void and internal crack formation during creep-rupture exposure was considered necessary. The 80Ni-20Cr alloy, Chromel A, was known to meet this requirement (Ref. 7) and was therefore used. It also was expected to be relatively free from density changes due to thermally-induced microstructural changes other than carbide precipitation.

For comparison the nickel-base superalloy Rene' 41 was also used. Like Chromel A it undergoes extensive elongation before rupture. It was known, however, (Ref. 10) that very little void or crack formation occurred. Moreover, it was expected that it would be subject to extensive microstructural changes from the effects of heating on the gamma prime (γ'), Ni_3 (Al, Ti), precipitate and the carbide phases.

BASIC ASSUMPTIONS AND TERMINOLOGY

The following table summarizes the concepts under which the study of density changes was undertaken:

	Type of Micro-structural Change	Primary Cause	Effect on Density
1.	cracking	creep	decrease
2.	voids	creep	decrease
3.	dislocations	stress	decrease
4.	type and amount of phase	temperature	increase or decrease
5.	type and amount of phase	stress	increase or decrease

A discussion of these follows.

1. Cracking. - An increase in the extent of cracking (whether macrocracking or microcracking) as creep proceeds will decrease the specimen density. It was assumed that cracking will not occur in the absence of stress and that unstressed exposure will not significantly affect the cracks if any, already present.

2

2. Voids. - The general term voids includes all the specific void types generated during creep, such as triple point voids (Zener cracking) and voids generated at boundary jogs, precipitates or inclusions. No attempt was made to differentiate between voids and microcracking. It has been assumed that an increase in the amount of any of the various void types will cause a decrease in the specimen density. It was further assumed that void formation is creep-induced and is not affected by temperature in the absence of stress.

3. Dislocations. - Since other work has indicated that the density changes during creep exposure caused by dislocations are insignificant (Ref. 2), it has been assumed that they are probably insignificant in Chromel A and Rene' 41. If density changes due to dislocations should be important in these alloys, available information indicates that dislocations would reduce density.

4. Types and amount of alloy phases. - The direction of the density change due to variations in the types and amount of phases present will be dependent upon the relative densities of the phases involved. A simple change in the distribution of a phase should not affect the density, however.

Several factors can affect the type and amount of phase present, the most important being (a) the disparity between the actual distribution of phases in an alloy and the equilibrium distribution (this disparity normally will tend to decrease with time at temperature) and (b) the influence of stress (or strain) on the equilibrium distribution of phases and on the kinetics of the move toward equilibrium.

It is convenient to simplify the above table further, assigning symbols to three major types of change:

M. - Voids, cracks and dislocations. - Items 1, 2 and 3 are all primarily stress- and/or strain-induced changes that decrease the specimen density. In the discussion that follows the symbol "M" has been used to represent specimen density changes due to the combined effect of these three stress-induced changes.

H. - Temperature-induced microstructural changes. - Item 4 refers to specimen density changes that are primarily temperature-induced and will henceforth be referred to by the symbol "H".

S. - Stress-induced microstructural changes. - Item 5 refers to specimen density changes that are primarily caused by the stress-induced precipitation of various phases. Also included are density changes resulting

3

from the influence of stress on the kinetics of temperature-induced microstructural changes. Specimen density changes of this type will henceforth be assigned the symbol "S".

It is important to note that the changes represented by M would be expected to decrease the specimen density, consequently M is always <0. It should be kept in mind that while the density should mainly reflect the above changes (and therefore reflect the extent of the creep damage) it does not necessarily reflect changes in morphology of the various phases in the microstructure which may have important effects on the extent of creep damage.

Using the above abbreviations, the density changes (relative to some reference density standard, defined as $\rho_{standard}$) are assumed to have a form $(\rho_{test} - \rho_{standard}) = f(M, H, S)$. Furthermore, for tensile creep it is assumed that:

$$(1) \quad (\rho_{tensile} - \rho_{standard}) = (M + H + S)$$

and for unstressed exposure:

$$(2) \quad (\rho_{exposure} - \rho_{standard}) = H$$

The difference between (1) and (2) representing stress-induced changes (i.e. precipitation, voids, cracking) can be measured directly or can be obtained by subtraction:

$$(3) \quad (\rho_{tensile} - \rho_{exposure}) = (M + S)*$$

Note that since M can only decrease the density of the specimen, if $(M + S) > 0$, then S must have been greater than the absolute value of M.

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* - Some further assumptions may be valid if compression creep testing data were considered, as noted in Appendix 2.

EXPERIMENTAL MATERIALS

Two materials were selected for study in this program. One was the binary alloy of 80 weight percent nickel and 20 weight percent chromium known as Chromel A. This alloy was chosen as one which should be relatively free from thermally-induced changes; any density change that occurred in Chromel A during creep exposure should primarily represent changes caused by the plastic deformation of creep alone. The second alloy, Rene' 41, was chosen to represent a more complicated alloy that undergoes both thermally-induced and deformation-induced changes. The Chromel A is also representative of the base upon which a large family of complex nickel-base alloys was established, including Rene' 41.

The Chromel A was received in the form of hot rolled $\frac{1}{2}$-inch diameter bar stock with the following reported chemical composition (weight percent):

Cr	Ni	C	Si	Mn	Fe
20.44	77.48	0.01	1.41	0.15	0.31

The bar stock was cut into 6-inch long blanks and heat treated in bundles of four for one hour at 2100°F, A.C. This treatment produced in the alloy an approximate ASTM grain size of No. 4.

The Rene' 41 alloy was received in the form of centerless ground 1 $\frac{1}{2}$-inch bar stock. The composition (weight percent) was reported to be:

Cr	Ni	Mo	C	Mn	Si
19.23	Balance	9.7	0.09	0.05	0.06

Co	Ti	Al	Fe	S	B
11.24	3.23	1.50	<0.03	0.007	0.0033

This alloy was rolled into $\frac{1}{2}$-inch square bars at the University of Michigan, cut into 6-inch long blanks and heat treated in bundles of four. Heat treatment consisted of a solution treatment of $\frac{1}{2}$-hour at 1950°F, A.C., plus a 16-hour age at 1400°F, A.C.

EXPERIMENTAL PROCEDURES

The experimental procedures used in this investigation were developed to facilitate the measurement of density change in specimens subjected to varying exposure conditions.

Creep Exposure Equipment and Procedures

Test specimens. - Details of the test specimens are shown in Figure 1A. This design was used for both the creep tests and the unstressed exposure tests. The specimens used for the density measurements were machined from the center of the gage length. Details of the density measurement specimens are shown in Figure 1B. Note that the outer (oxidized) surface of the exposure specimens was removed.

Creep testing equipment and procedure. - The creep exposure tests and the unstressed exposure tests were made in University of Michigan individual creep-rupture testing machines. The stress was applied through a third-class lever system, with a lever arm ratio of about 10:1. The specimens were gripped by threaded holders which fitted into a universal joint system to promote uniaxial loading. The specimens were heated by a wire-wound resistance furnace that fitted over the entire specimen holder assembly. Temperature measurements were made by three Chromel-Alumel thermo-couples located at the center and two ends of the gage length. All the thermo-couples were shielded from direct furnace radiation. The furnaces were brought up to within 50°F of the test temperature before the specimens were placed in the furnace and were then brought up to temperature and distribu-tion. The time necessary to reach temperature and distribution was typi-cally three hours. ASTM E159 Recommended Practices were followed in the control of the test temperature. The weight of the holders and neces-sary associated equipment produced a stress of 54.5 psi on the "unstressed" exposure specimens. At the completion of each test the furnace was turned off and the specimens furnace cooled under load.

Testing program. - As noted before, creep exposure as well as unstressed exposure tests were conducted. These tests were run at three temperatures and five stress levels; three stress levels at the intermediate temperature and one stress at each of the extremes of temperature. The tests were interrupted at various times, these times being chosen to be equal incre-ments of the expected rupture life on a logarithmic scale. For each com-bination of temperature and time used for the creep exposure tests, an

6

unstressed exposure was also made. The various temperatures, stresses and times used are tabulated below:

Stress (psi)	Temperature (°F)	Test interrupted at: (hours)				Expected Rupture Life (hours)
Chromel A						
39,000	1000		82,	240,	700	4000
11,000	1200	28,	82,	240,	700	2220
14,000	1200	28,	82,	240,	700	820
18,000	1200	10, 28,	82,	240		300
6,600	1400	28,	82,	240		300
Rene' 41						
105,000	1200		82,	240,	700	800
37,000	1400	28,	82,	240,	700	2200
46,000	1400	28,	82,	240,	700	750
59,000	1400	10, 28,	82,	220		238
24,000	1600	10,	82,	170		200

Density Difference Measurements

Two different sets of density difference measurements were made. One set measured the density changes (H) that occurred during unstressed exposure, relative to the as-heat treated specimen density. The second set measured the density changes (M + S) that occurred during creep exposure, relative to the corresponding unstressed exposure specimen density. Knowledge of M + S and H allowed the calculation of the total specimen density change during creep exposure by addition, M + S + H . It should be noted that the technique allows for the direct measurement of density change due to any two of the three variables, H, M + S and M + S + H. The total density change during creep exposure (M + S + H) could have been measured directly along with H which would have allowed calculation of M + S .

Theory of differential density measurement. - The density measurements used in this work were made by a differential hydrostatic weighing technique adapted from work done by R. V. Day (Ref. 2). This method allows the calculation of the density difference between two specimens from

$$\frac{\Delta\rho}{\rho_{ws}} = \frac{(\rho_{air}/\rho_{wts}-1)\{\rho_{ws}(m_{air}-m_{liq})+\rho_{air}m_{liq}-\rho_{liq}m_{air}\}}{m_{ws}(\rho_{liq}-\rho_{air})+\rho_{ws}(m_{liq}-m_{air})(1-\rho_{air}/\rho_{wts})}$$

where $\Delta\rho$ = density difference between the two specimens

ρ_{ws} = density of the specimen assigned as the weighing standard

m_{ws} = mass of the specimen assigned as the weighing standard

m_{air} = difference in mass of the two specimens in air

m_{liq} = difference in mass of the two specimens in the liquid

ρ_{liq} = density of the liquid

ρ_{air} = density of air

ρ_{wts} = density of the balance weights

The weighing standard was arbitrarily chosen to be the as-heat treated specimen when measuring H and the unstressed exposure specimen when measuring (M + S). The liquid used was tetrabromoethane, a very dense liquid with a variation in density with temperature given by

$$(\rho_{liq} = 3.00437 - 0.002141T)$$

where T is the temperature of the liquid in °C (Ref. 3).

Specimen preparation. - Great care was taken to ensure thorough and consistent cleaning of the specimens used for the density change measurements. The specimens were first washed in technical grade trichlorethylene. This was followed by a washing in a strong commercial detergent and water. The specimens were then washed in absolute alcohol (using an ultrasonic cleaner) to remove any traces of water and dried for half an hour at 200°F. This was followed by a final ultrasonic cleaning in electronic grade trichlorethylene and two hours drying at 200°F. After each step of the washing procedure the specimens were dried with cotton gauze to remove excess liquid that might evaporate and leave a deposit. The samples were kept in clean, covered glass beakers at all times and were stored in a dessicator when not being used for weighings. To avoid contamination the specimens were handled with stainless steel forceps.

Density measurement equipment. - The balance used for the density change measurements was a Sartorius Model 1800 microbalance having a capacity of 30 grams and capable of vernier readings to micrograms (0.000001 grams). This is an equal arm balance using air cylinders for damping to the rest point (Fig. 2). After adaptation for measuring density differences it had the capacity to weigh specimens differences as large as 0.099 grams. The weights were conveniently applied by actuating a graduated dial. The modifications necessary were:

1. Provision for holding the specimens.
2. Provision for suspending the specimens from the balance.
3. Provision for making the weighings in a liquid.

The requirements for the holder were rather stringent, the most important being that the volume of the specimen holders under the liquid be equal. Volume differences between the two holders were minimized by making the absolute volume of each holder as small as possible. This was done by using 0.0031-inch diameter Alumel wire. The holder was a short length of this wire with a loop at one end, the loop being the proper size to just slip over the specimen, as shown in Figure 3A. These holders were made on a jig to further ensure uniformity. This particular holder design had other advantages; it reduced the effects of surface tension by making the surface area penetrating the liquid small, and it minimized the problem of entrapped air bubbles. The specimen holders were fastened to "S" hooks that attached to a special holder connected to the knife edges.

The system used to contain and circulate the liquid (Fig. 3B) was designed to:

1. Permit circulation of the liquid.
2. Prevent contamination of the liquid.
3. Be removable, to allow weighings in air.

The system was made of glass to minimize contamination of the liquid. Stirring to avoid temperature gradients was accomplished by a small centrifugal pump using a teflon-covered stirring bar as an impeller. This stirring bar was turned by a rotating magnet outside the system. The liquid was stirred in a central reservoir outside the balance and was distributed equally between two beakers inside the balance from this central reservoir. The temperature of the liquid was measured at the central reservoir, using a thermometer readable to $\pm 0.05\,°C$. The balance was mounted on a 700-lb. marble table to minimize the influence of vibration and it was surrounded by a glass case with a sliding door in front. This case was covered with aluminum foil to reduce temperature changes due to external sources, such as heat from the operator's body.

<u>Density measurement technique.</u> - Careful attention was given to the technique of weighing. The quantity m_{ws} was first determined to ± 0.001 grams on an ordinary balance. Then m_{air} was determined to ± 0.001 grams in order to limit the degree of imbalance and thereby protect the microbalance.

The procedure used for measuring m_{air} on the microbalance was:

1. Determine zero point of the balance with the specimen holders in place.
2. Attach the specimens and measure m_{air}.
3. Recheck the zero point.

Before each weighing and zero point check the beam was released, arrested and released a second time before it was allowed to come to rest for the reading. One minute after the second release the reading was taken. The zero points before and after the measurement of m_{air} were averaged and used to correct m_{air}. Variations in the zero point were typically <0.000005 grams.

The procedure used for measuring m_{liq} was similar to that used for m_{air} except that in this case the zero point with the specimen holders in place can not be determined after the liquid weighing. This occurs because the holders are wet. Consequently the zero point change during weighing was approximated using the change in rest point with the holders off before and after the liquid weighing.

The liquid weighing procedure was as follows:

1. Determine the zero point of the balance with the specimen holders in place.
2. Measure the rest point with the specimen holders off.
3. Place the specimens in the specimen holders and suspend them from the beam into the liquid; stir the liquid for five minutes.
4. Release and arrest the beam three times. Release the beam a fourth time and allow one minute for the beam to come to rest, then measure m_{liq}.
5. Remove the specimens and their holders and measure the rest point a second time.

The zero point was adjusted an amount equal to half the change in rest point reading before and after weighing in liquid. This variation was also typically <0.000005 grams.

10

Analysis of errors. - The equation for $\Delta\rho/\rho_{ws}$ discussed previously can be simplified considerably by neglecting the buoyancy effect of air. This simplification causes an error in $\Delta\rho/\rho_{ws}$ of $<1 \times 10^{-8}$, negligible since the relative density changes measured were $>1 \times 10^{-4}$. This simplified equation is

$$\Delta\rho/\rho_{ws} = \frac{m_{air}\rho_{liq} - \rho_{ws}(m_{air} - m_{liq})}{m_{ws}\rho_{liq} - \rho_{ws}(m_{air} - m_{liq})}$$

Analysis of the effect of errors in the above variables on $\Delta\rho/\rho_{ws}$ yielded the following:

Variable	Maximum measurement error	Resulting error in $\Delta\rho/\rho$
m_{ws}	±0.001 gms	$\pm2 \times 10^{-9}$
ρ_{ws}	±0.01 gm/cm^3	$\pm4 \times 10^{-6}$
m_{air}, m_{liq}	±0.000025 gm	$\pm5 \times 10^{-6}$
ρ_{liq}	±0.0002 gms/cm^3	$\pm2 \times 10^{-7}$

ρ_{liq} was calculated using the equation given previously. To know ρ_{liq} to and accuracy of ±0.0002 gms/cm^3, one must known the temperature to an accuracy of $\pm0.1\,^\circ$C. This was easily achieved, as the thermometer used was readable to $\pm0.05\,^\circ$C.

A conservative upper bound on the measurement error of $\Delta\rho/\rho_{ws}$ (the sum of the above) is $<\pm1 \times 10^{-5}$, or an error in $\Delta\rho$ of $<\pm0.00008$ gms/cm^3. The maximum upper bound on the total error, as evidenced by a series of density comparisons on the as-heat treated material, was determined to be ±0.00025 gms/cm^3. This takes into account the variation in density of the as-heat treated material arising from such things as compositional variation along the bar and heat treatment variations, as well as measurement errors.

RESULTS AND DISCUSSION

Unstressed Exposure Tests of Chromel A

The density variations of the unstressed exposure specimens (H) of Chromel A, relative to the as-heat treated condition (one hour at 2100°F, A.C.) were determined at temperatures of 1000°F, 1200°F and 1400°F for times from ten to 700 hours. These curves are shown in Figure 4. The significant density change which occurred during unstressed exposure indicates that Chromel A was subject to thermally-induced changes. Therefore the original assumption that the density change in Chromel A would be due to stress-induced changes alone is not valid. It should be noted that positive values of H indicate that unstressed exposure increased density relative to that of the as-heat treated standard.

Consideration has been given to several aspects of the observed density variation with time and temperature:

1. The rapid density increase relative to the density of the standard which occurs during the first ten hours of unstressed exposure. If the three curves of Figure 4 are extrapolated to zero time, it can be seen that the initial rapid density increase is about the same at all three exposure temperatures - approximately +0. 00275 gms/cm^3.
2. The further increase in density of the unstressed exposure specimens from ten to 700 hours at all three temperatures.
3. The density increase during exposure from ten to 700 hours varied directly with the exposure temperature.

The cause of the rapid density change that took place during the first few hours cannot be established from available information. The following observations however, may be relevant:

1. It is known that 80Ni-20Cr alloy is subject to anomalous resistivity and lattice parameter changes in the vicinity of 915°F. The reason for these changes has never been established beyond doubt, although numerous investigations have been made (Ref. 4). The density changes could be related to the same phenomenon.
2. There could be an influence of cooling rate on density, since the as-heat treated standard was air cooled, while the unstressed exposure specimens were furnace cooled. A specimen air cooled after ten hours at 1400°F showed a density change of +0. 00245 gms/cm^3. This was somewhat less than noted in a sample furnace cooled after ten hours at 1400°F (+0. 00375 gms/cm^3), suggesting that cooling rate may affect the degree of change but not sufficiently to account for the total initial change.

12

One of the most prevalent explanations for the physical property anomalies of the 80Ni-20Cr alloys has been the development of an ordered structure. Lattice parameter decreases of 0.008 kx units have been reported in a 76Ni-24Cr alloy as the result of ordering (Ref. 5). A lattice parameter change of this magnitude would cause a density increase of 0.063 gms/cm^3. Such an increase is of an order of magnitude larger than the maximum density change measured. If ordering was responsible for the rapid density change then the density change being so much less than calculated could be due to incomplete ordering and/or compositional differences. It should be noted that the exposure temperatures used were higher than the temperature at which ordering was observed (915°F). Also the time during which the maximum rate of change of density occurred was relatively short compared to those normally required to produce an ordered structure. In spite of these differences the measured density change could be a result of lattice parameter changes accompanying incomplete ordering.

Study of the 80Ni-20Cr alloy system (Ref. 6) has shown two complex carbides, $(Cr, Fe)_7 C_3$ and $(Cr, Mn)_7 C_3$, to precipitate during ageing. The increase in density with time at temperature and the increase in density with exposure temperature at times from ten to 700 hours may be the result of carbide precipitation. Rough calculations (Appendix 1) indicate that the precipitation of $(Cr, Fe)_7 C_3$ and $(Cr, Mn)_7 C_3$ could cause an increase in density of about 0.0026 gms/cm^3. This compares with the maximum measured density increases in Chromel A (not including the initial rapid 0.00275 gms/cm^3 change) of 0.0024 gms/cm^3 after 240 hours at 1400°F, 0.002 gms/cm^3 after 700 hours at 1200°F and 0.00013 gms/cm^3 after 700 hours at 1000°F.

While vacancy diffusion and dislocation migration may be influencing the density during unstressed exposure, the effect is probably small. Reference 2 indicates that dislocations do not made a significant contribution to density variation in a 1% Cr-Mo steel, even under conditions of stress. Other than this, no data were found in the literature with which to estimate the degree to which vacancy diffusion and dislocation migration influence density during unstressed exposure.

Creep Exposure Tests of Chromel A

The following curves have been used to present the results obtained from the creep exposure tests:

1. The density difference between specimens which were subjected to creep exposure and those subjected to unstressed exposure for the same time and at the same temperature. The curves are labelled (M + S).
2. The density difference between specimens subjected to unstressed exposure and the as-heat treated standard (one hour at 2100°F, A.C.). These curves are labelled H and are the same as those of Figure 4.
3. The density difference between specimens which underwent creep exposure and the as-heat treated standard. These differences were calculated by adding the measurements described under 1. and 2. above and are labelled (M + S + H).

Of particular interest is the curve (M + S) versus time. Because the densities of the stressed exposure specimens were compared to those of the unstressed exposure specimens in the determination of (M + S), the uncertainties involved in the variation of H with time were avoided since they were cancelled out. The most striking feature of the curves of (M + S) versus time (Figs. 5 to 9) was the marked decrease in density for those tests which were carried out to near rupture. As might be expected, this marked decrease occurred in shorter times the higher the stress at 1200°F (compare Figs. 5, 6 and 7). The one test conducted at 1400°F indicated only a slightly faster loss of density than occurred in a 1200°F test with a similar expected rupture time (compare Figs. 7 and 8). The test conducted at 1000°F (Fig. 9) was not run to a large enough fraction of the rupture life to ascertain whether or not the alloy behaved in the same manner as observed at the higher temperatures.

The short-time behavior of the (M + S) curves is less striking. The density variations, however, are much larger than the sensitivity of the technique and are therefore significant. The following points should be noted in the consideration of the behavior of the (M + S) curves:

1. M can only decrease specimen density.
2. A deviation of (M + S) from zero indicates that the density is being changed by either M or S or both.
3. When (M + S) >0, stress-induced precipitation must have occurred such that S is greater than the absolute value of M.

The calculations discussed in Appendix 1 show that carbide precipitation in Chromel A should result in a density increase. Since stress should cause

precipitation of the same phases that precipitate during unstressed exposure, stress-induced precipitation (S) should increase specimen density. If this reasoning is valid, whenever (M + S)<0 then M (void and crack formation) must have occurred. Furthermore, a negative slope to the (M + S) curve indicates that the rate of density change due to M is greater than the rate of density change due to S.

The (M + S) curves at 1200°F (Figs. 5, 6 and 7) initially exhibited a positive slope to some maximum value followed by a negative slope. The peaks of the curves occurred at shorter times the higher the stress. This indicates that the time of initiation of voids and cracks (M) varied inversely with stress. The initial positive slope of the curves indicates that either stress-induced precipitation or ordering or both occurred during the creep process. The resultant density increase, however, was soon masked by the marked density decrease accompanying void and crack formation.

Figure 8 shows the density variation for tests run at 1400°F and 6,600 psi. Comparison of the (M + S) curves of Figures 7 and 8, where in both cases the expected rupture lives were 300 hours, showed that M increased relative to S as the temperature was increased.

Detection of Creep Damage without Regard to Prior History

An important case for detecting and measuring creep damage is in applications where the prior history is unknown. A technique which would permit the determination of the degree of damage accumulated in an alloy during long-time service would have immediate application. In the present study unknown prior history has been assumed. In this case the only information available would be the total change in density (M + S + H) of Figures 5 to 9 as a function of percent rupture life (Fig. 10). For the tests conducted there was a marked decrease in specimen density after 30 to 38 percent of the rupture life, indicating the onset of relatively large amounts of M (voids and cracks). Because the density increased during the first few percent of the rupture life to a significant extent and then did not change markedly for at least 30% of the rupture life, the data suggest that in the absence of detailed information, more than 30% of the rupture life would be used up before damage would be evident from density change measurements.

Previous work on Chromel A (Ref. 7) supports the conclusions drawn from Figure 10. Data presented there indicated extensive cracking started between 34% and 72% of the rupture life. Unfortunately metallographic examination of specimens exposed for life fractions between these limits was not carried out.

Detection of Creep Damage with Detailed Prior History Available

The primary measurement of this investigation was the difference in density between specimens that had undergone unstressed exposure and specimens which had undergone varying degrees of creep. These curves of (M + S) are summarized in Figure 11 as a function of percent rupture life. The overall tendency is similar to the total density changes of Figure 10, except that the uncertainties involved in H (temperature-induced density changes) have been removed. Again the dramatic change in slope occurred at 30 to 38 percent of the rupture life, indicating the start of extensive cracking. The maximum of these curves (as discussed above) could be used to indicate the initiation of density changes due to M. If density changes due to M can be considered to be creep damage, this implies that the present method may be capable of detecting creep damage as early as 4% of the rupture life at 1200°F in Chromel A.

When Figures 5 to 9 are compared to Figures 10 and 11, it is evident that the overall density changes in Chromel A correlate better with percent rupture life than times of exposure. Attempts to correlate the density changes (under the various exposure conditions) with creep strain were unsuccessful.

Crack Detection in Chromel A

A few tests were conducted on specimens of the alloy using a dye penetrant. No cracks were detected in the Chromel A specimens that had shown the largest decreases in density after creep. Tests on rupture samples (on the as-tested surface) showed cracks slong the entire gage length. Previous studies (Ref. 7) on Chromel A indicated that extensive cracking occurred throughout the cross-sections of the specimen, not just on the surface. Apparently the machining of the density specimens "smeared" the surfaces and prevented the dye from penetrating the cracks.

The results of the dye penetrant examination of the rupture specimens and a few specimens that had been discontinued before rupture (also on the as-tested surface) gave a qualitative support to the evidence cited previously that the extent of M (cracking and voids) in Chromel A increased as the time or temperature increased.

16

Rene' 41 Alloy

Rene' 41 undergoes marked hardening and strengthening through pre-cipitation of γ' phase, Ni_3 (Al, Ti). In addition it contains various carbide phases, as well as inert Ti (C, N). In theory it is a considerably more complicated alloy than Chromel A, although this may be an oversimplifi-cation if the suggested ordering reaction actually occurs in the Chromel A alloy. While it was possible to estimate the density changes caused by carbide precipitation in Chromel A, this could not be accomplished for Rene' 41.

From the viewpoint of voids and microcracking from creep, there is a marked difference between Chromel A and Rene' 41. Chromel A under-goes extensive internal cracking during the third stage of creep prior to rupture. Rene' 41 also has high elongation, but the degree of internal cracking is slight compared to that of Chromel A. Density decreases due to the formation of cracks and voids would therefore be expected to be sig-nificantly less in Rene' 41 than in Chromel A.

Prior research has shown that the γ' particles (in other γ' precipitat-ing alloys) agglomerate quite rapidly during exposure at 1600°F. It has also been shown that there is an accompanying decrease in the volume percent of γ' (Ref. 8). The γ' agglomeration also occurs at 1400°F, but at a much slower rate. Reliable data showing the changes in the state of the γ' during exposure at 1200°F were not available and are difficult to obtain due to the small size and slow growth rate of the γ' particles devel-oped at this temperature. There are also no data known to the authors which show the effects of γ' instability on the density.

Unstressed Exposure Tests of Rene' 41

The changes in density of specimens in the heat treated condition ($\frac{1}{2}$-hour at 1950°F, A.C., plus 16 hours at 1400°F, A.C.) when exposed with-out stress at 1600°, 1400° and 1200°F and furnace cooled were unexpected (Fig. 2). Two features of the H versus time curves require explanation:

1. The absolute magnitude of H at 1400°F is too large, as the 1400°F exposure in effect represents a continuation of the original ageing treatment. Small changes in density in short-time unstressed expo-sure at 1400°F would be expected, not the large changes measured.
2. The relative positions of the three curves are reversed from those of the Chromel A.

The marked change in density after ten hours of unstressed exposure at 1400°F is probably due to variations in the cooling rate between the as-heat treated standard and the exposed specimens. The standard specimen was air cooled after the 16-hour age at 1400°F while the unstressed exposure specimens were furnace cooled. In order to determine whether cooling rate affected density, a sample was aged the usual 16 hours at 1400°F, but was then furnace cooled instead of air cooled. The density of this specimen was practically the same as the density of the specimen in the aged condition which had been exposed for ten hours hours at 1400°F and furnace cooled. The major change in density at 1400°F was therefore due to the difference in cooling rate from 1400°F and not to unstressed exposure at this temperature.

The $\gamma \rightarrow \gamma + \gamma'$ reaction is suggested as a possible cause of this behavior. Previous work (Ref. 9) has shown that the rate of the $\gamma \rightarrow \gamma + \gamma'$ reaction is sufficiently rapid to cause difference amounts of γ' to be present after furnace or air cooling.

Another possible cause might be an ordering reaction, as was suggested for the Chromel A. While there is no evidence known to the authors that would suggest that an ordering reaction occurs in Rene' 41, the fact that one has been identified in Chromel A implies that the possibility of such a reaction should be investigated.

It is possible that the reversal of positions of the three curves for Rene' 41 (compared to Chromel A) might also be due to the $\gamma \rightarrow \gamma + \gamma'$ reaction. Since the equilibrium amount of γ' increases as the temperature decreases, more γ' precipitation would be expected on furnace cooling than on air cooling. This suggests based on the furnace versus air cooling data at 1400°F, that the density of Rene' 41 increases as the amount of γ' increases. While the precipitation of γ' is rapid, it most likely is not rapid enough to permit true equilibrium during the furnace cooling, especially since in this case the furnace cooling was relatively rapid; the specimen cooled from 1600° to 1200°F in about 35 minutes. The amount of γ' and hence the density would therefore be expected to increase as the temperature of the unstressed exposure decreased, even when the specimens are all furnace cooled. Thus the relative position of the density curves shown in Figure 12 might be explained in terms of the amount of γ' formed in the alloy. It is important to note that positions of the curves of Figure 12 and the measured density changes between air and furnace cooling are consistent.

It should be noted that this analysis would predict that a specimen that had been exposed without stress at 1600°F and air cooled should have a lower density than that of the as-heat treated standard. This test was

18

carried out and the results support the analysis; the resulting specimen (as noted on Fig. 12) was 0.00495 gms/cm^3 less dense than the as-heat treated standard specimen.

Creep Exposure Tests of Rene' 41

The density variation data measured in Rene' 41 after exposure under stress are presented in Figures 13 to 17. The curves of (M + S) versus time (since they compare the density of two specimens that have both undergone furnace cooling) should not reflect the cooling effects discussed above.

The data indicate that creep had very little effect on specimen density, especially in view of the large variations due to the cooling rate alone. The fact that (M + S) >0 indicates the occurrence of stress-induced precipitation in all but the tests run at 1600°F. While there may be reason to suspect that M had occurred when the slope of (M + S) versus time changed from positive to negative, the trend was not at all definite and the magni-- tude of the density change was much smaller than in the Chromel A.

The absence of large decreases in density indicates that cracking is a minor factor in Rene' 41. An example of this can be seen in Figure 14. Only very slight decreases in density due to M are evident even though the tests were run to 92.5% of the rupture life.

Crack Detection in Rene' 41

A dye penetrant was used on a few Rene' 41 creep specimens that had not been machined after testing. These included some rupture specimens as well as two tests that were discontinued at 70% of the rupture life at 1600°F and at 95% of the rupture life at 1400°F. No cracking was evident on either of these tests, a result supported by previous metallographic work (Ref. 10). While the ruptured specimens showed evidence of a few large cracks immediately adjacent to the rupture surface the rest of the gage length was found to be crack-free. These results support the conclusion made previously that cracking is of minor importance in Rene' 41.

Dye penetrant tests were also made on a few of the density specimens of Rene' 41. Even though the specimens tested had the largest decrease in density, no evidence of cracking was found.

Interpretation of Density Changes in Rene' 41

The most striking result was the unexpected effect of cooling rate on the density after unstressed exposure. This finding warrants further investigation for several reasons. Presumably it is indicative of dimensional instabilities which have not been well known. Likewise the structural effects causing the density change could be associated with mechanical property variations, particularly where heating times are very short before loads are applied. The apparent stability of mechanical properties at ambient temperatures after creep exposure found in a previous investigation (Ref. 11) may have been influenced by this factor to some extent. Perhaps the most important reason could be the finding that there are unrecognized structural features which may have an important influence on metallurgical characteristics. For this reason the subject should be studied further. Moreover it should be established to what degree the cooling rate effect is controlled by solution treatment conditions, time of heating at service temperatures and by the presence of stress. It would also be interesting to know if the effect is reversible. Although experimentally rather difficult to carry out, it should be both desirable and possible to control the cooling rate effect so as to be more certain of the density changes associated with creep.

One puzzling aspect of the data was the lack of density changes which would correlate with the apparent decrease in volume fraction of γ' in similar alloys and the increase in γ' particle size found by Rowe (Ref. 8). It would seem that this might have been expected to be fairly prominent if density changes were sensitive to the state of the γ'. Rowe's data were based on exposure at 1500° and 1600°F.

The data at 1600°F in the present investigation is very erratic, as shown in Figure 12. This behavior is probably a result of fluctuations in the cooling rate among samples. These fluctuations could have masked any trend that would either confirm or contradict results found by Rowe.

Rene' 41 is an alloy which accumulates elongation by creep with very little microscopic evidence of internal void formation and microcracks. Gluck (Ref. 10) showed that room temperature mechanical properties were not changed appreciably by creep exposure when the specimen surfaces were remachined after creep. Apparently the major cause of rupture failure was the initiation and growth of a few intergranular cracks from the surface. The results of the present investigation tend to support this concept. This mechanism of failure is in contrast to that noted in Chromel A, which undergoes extensive overall elongation through the formation of numerous internal voids and cracks.

20

GENERAL DISCUSSION

Density change measurements appear to offer considerable promise as a means of detecting structural changes with a significantly higher degree of sensitivity than afforded by other methods such as microscopic examination, x-ray diffraction, etc. The ability to detect changes which had not heretofore been recognized with a sensitive and simple technique is unusual. There is a high probability that a review of the density changes could clear up many of the ill-understood effects of composition and heat-treatments on creep-rupture properties in many alloy systems.

Density changes are not always easy to interpret. Their main value will be to point out unknown or unexpected structural changes which can then be correlated with the known properties of alloys.

The research has shown that density change measurements can be used to evaluate the degree of creep damage within certain limits. One aspect of creep damage is particularly important. There has been a considerable backlog of not well documented experience which indicates that there can be damage which is permanent, i. e. when creep life cannot be restored by heat treatment alone. This concept is usually further extended to suggest that only the loss in life associated with thermally-induced structural changes can be restored by heat treatment. The literature suggests that the "permanent" loss in life is associated with the formation and growth of voids and cracks. It would seem that an investigation in which "permanent" density changes were measured should be useful in clarifying the mechanism of creep damage. In such an investigation the measurement of the density changes reversible during reheat treatment after varying amounts of creep would be studied.

The investigation of density change as a function of exposure conditions avoided surface attack and cracking as factors which might influence the results. Their inclusion in the program would have resulted in a very complicated problem associated with the infinite variety of environmental effects which would have influenced surface condition.

Combinations of differential density measurements and microstructural studies offer promise of identifying factors affecting properties which have heretofore not been suspected. For example, it should be noted that decreases in volume are rather common in the early stages of creep tests on some materials. Usually this so-called "negative creep" is masked by the super-imposed elongation of first-stage creep. It is possible, however, that volume decrease is an important factor in determining the creep characteristics of

an alloy. The results of this investigation therefore suggest that research on the reactions occurring during the early stages of exposure to temperature and stress as well as cooling rate effects on structure could be productive in clarifying some of the causes for variability in creep-rupture strengths.

CONCLUSIONS

Highly sensitive differential density measurements were carried out on two alloys to investigate their applicability to study of thermally-induced and creep-induced structural processes. The following conclusions were reached:

1. Microstructural changes were detected which normally could not be detected by other techniques. When heated at 1000°, 1200° and 1400°F an 80Ni-20Cr alloy (Chromel A) was found to be subject to rapid density changes, the degree of change being primarily dependent on temperature of heating and secondarily on cooling rate after solution treatment. The density of a precipitation-strengthened nickel-base alloy (Rene' 41) was shown to be highly sensitive to cooling rate from the temperature of exposure. These features of the behavior of the alloys had not previously been known.
2. Creep-induced structural processes including strain-induced precipitation and crack initiation and growth were found at stages much earlier than could be detected by other techniques.
3. Coupled with theoretical analysis, density change measurements can be used not only to detect creep-induced processes but also to limit them to possible types for identification and correlation with properties.
4. With adequate knowledge of microstructural changes and their effect on density, the technique could be used as a sensitive measure of creep damage in materials subject to moderate or extensive cracking during the creep process. Since the demand for a sensitive measure of creep damage is rising, this potentiality may prove to be of prime importance.

The conclusion of the research considered to be of primary importance at this time, however, was the determination that differential density measurements are capable of detecting structural changes in metals with a higher degree of sensitivity than obtainable with other methods. As evidence of this, unsuspected structural changes were found in two widely used alloys which have been the objects of extensive research in the past.

APPENDIX 1

Density Change due to Carbide Formation in Chromel A

In calculating the density change in Chromel A resulting from carbide precipitation the following assumptions have been made:

1. The carbide phase is Cr_7C_3. Previous work (Ref. 6) has shown the major carbide phases to be $(Cr, Fe)_7C_3$ and $(Cr, Mn)_7C_3$. Since the molecular weights of iron, chromium and manganese are not very different this assumption seems reasonable.
2. The alloy contains only chromium, nickel and carbon and that their relative amounts are the same as those in the actual alloy. This is indicated in the following table:

	weight % (original)	molecular weight	weight % (assumed)	moles	mole %
chromium	20.44	52	20.872	0.401	.23
nickel	77.48	58.7	79.118	1.348	.769
carbon	0.01	12	0.010	0.00085	.00048
	97.93		100.000	1.74985	

3. The dissolved carbon is present interstitially.

When Cr_7C_3 is formed five changes occur that will affect the overall density of the specimen. These are:

A. The molecular weight of a unit cell in the matrix increases as chromium leaves the solid solution to form a carbide. As chromium atoms are "taken" from the matrix to form Cr_7C_3 the matrix solid solution has a higher percentage of the heavier element nickel.
B. The molecular weight of the unit cell decreases as the interstitial carbon is removed.
C. The matrix lattice parameter a_0 changes as the percent nickel is changed.
D. The matrix lattice parameter a_0 decreases as carbon is removed from the interstices.
E. The overall density changes because the new phase Cr_7C_3 has a density (6.95 gms/cm^3) different from that of the original matrix (8.275 gms/cm^3).

Analysis of these five changes follows:

1. The change in molecular weight (ΔM) due to loss of chromium from solution if

$$n_i = \text{number of moles of element } i$$

and $\qquad T = n_i = \text{total moles present} \quad (1.75)$

then the "average" weight of the atoms in the matrix can be determined from:

$$\frac{(52 \times n_{cr} + 58.7 \times n_{ni})}{T} = M_{av} = 57.02$$

When n_c moles of carbon combine with $(7/3 \times n_c)$ moles of chromium from the matrix to form $Cr_7 C_3$, the new average molecular weight $M_{av}{}'$ is

$$M_{av}{}' = \frac{52 \times (n_{cr} - 7/3 n_c) + 58.7 n_{ni}}{(T - 7/3 n_c)}$$

Subtracting and combining terms yields

$$M_{av}{}' - M_{av} = \Delta M = + \frac{11.7 n_c}{(T - 7/3 n_c)}$$

2. ΔM due to the loss of interstitial carbon.
Similar analysis of the effect of carbon leaving the interstitial positions to form carbide yields

$$\Delta M = - \frac{45.02 n_c}{(T - n_c)}$$

3. The change in lattice parameter (Δa_0) due to a loss of chromium from the matrix.
The change in a_0 with composition of the solid solution of nickel and chromium has been tabulated (Ref. 12). The lattice parameter decreased nearly linearly as the atomic percent chromium decreases; a change of 1.0 atomic percent chromium causes a lattice parameter change $\Delta a_0 = 0.00145$ kx units. Assuming that a_0 does vary linearly with the atomic

percent chromium, the change in the lattice parameter of the matrix with a change in the amount of chromium is

$$\Delta a_0 = -0.083 n_{cr} = -0.193 n_c$$

4. Δa_0 due to the removal of interstitial carbon.

Assuming a hard sphere model and defining the atomic radius to be the distance of closest approach, Δa_0 can be found. The radius of an interstitial hole in a face-centered cubic structure (R_I) can be shown to be (Ref. 13)

$$R_I = \frac{0.41 \times 2 a_0}{4} = 0.51 \text{ kx}$$

Using the Hume-Rothery value for the radius of a carbon atom, 0.75 kx, $\Delta a_0 = -0.28$ kx per unit cell that loses a carbon atom. Averaging this change over all the cells

$$\Delta a_0 = -0.64 n_c \quad .$$

Combining the above results, using $n_c = 0.00085$

$$\Delta M = -0.0162$$

$$\Delta a_0 = 0.00071$$

yielding

$$M_{av}' = 57.0038$$

$$a_0' = 3.5403 \text{ kx units}$$

where

$$M_{av} = 57.02$$

$$a_0 = 3.541$$

It can further be shown that the total density change $\Delta \rho$ is

$$\Delta \rho = \left\{ \frac{4 \times M_{av}}{a_0^3} - \frac{M_{av}'(T - 1/3 n_c) + M_{carbide} \times 1/3 n_c}{\tfrac{1}{4} a_0'^3 + 1/n \, V_{carbide} \, 1/3 n_c} \right\}$$

26

where

$M_{carbide}$ = average molecular weight of Cr_7C_3 atom

$V_{carbide}$ = volume of a unit cell of Cr_7C_3

Substituting, the density change due to carbide precipitation

$$\Delta\rho = -0.0026 \text{ gms/cm}^3$$

APPENDIX 2

Testing done in this program included only tension and non-stressed exposure tests. For the sake of completeness, it is noted that work has been done that indicates no cracking or void formation occurs during compression creep (Ref. 2). If this is true, it is possible to write further:

$$(\rho_{compression} - \rho_{standard}) = H + S$$

$$(\rho_{compression} - \rho_{exposure}) = S$$

$$(\rho_{compression} - \rho_{tension}) = M$$

The above requires that $S_{tension} = S_{compression}$. This should be reasonable as a first approximation.

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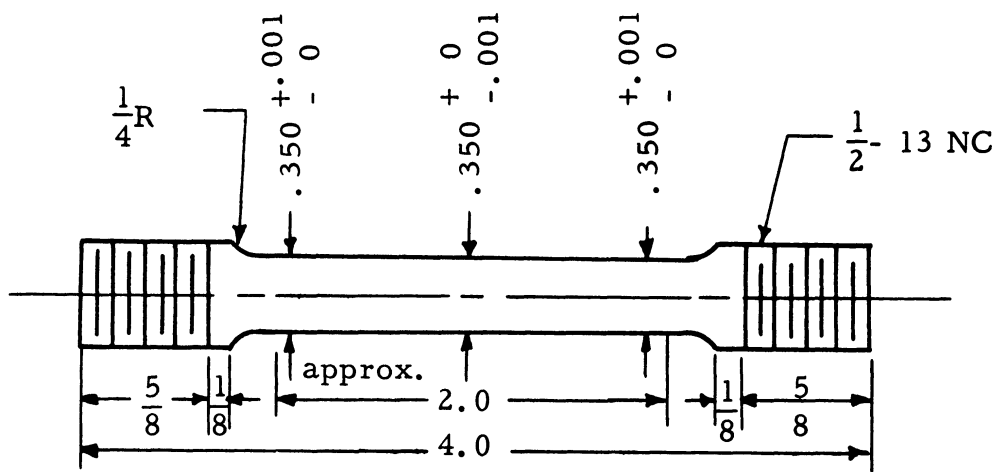
Figure 1A. Creep and Unstressed Exposure Specimen
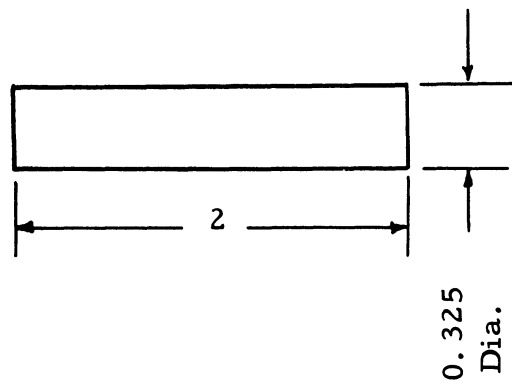
Figure 1B.   Specimen for Density Measurements
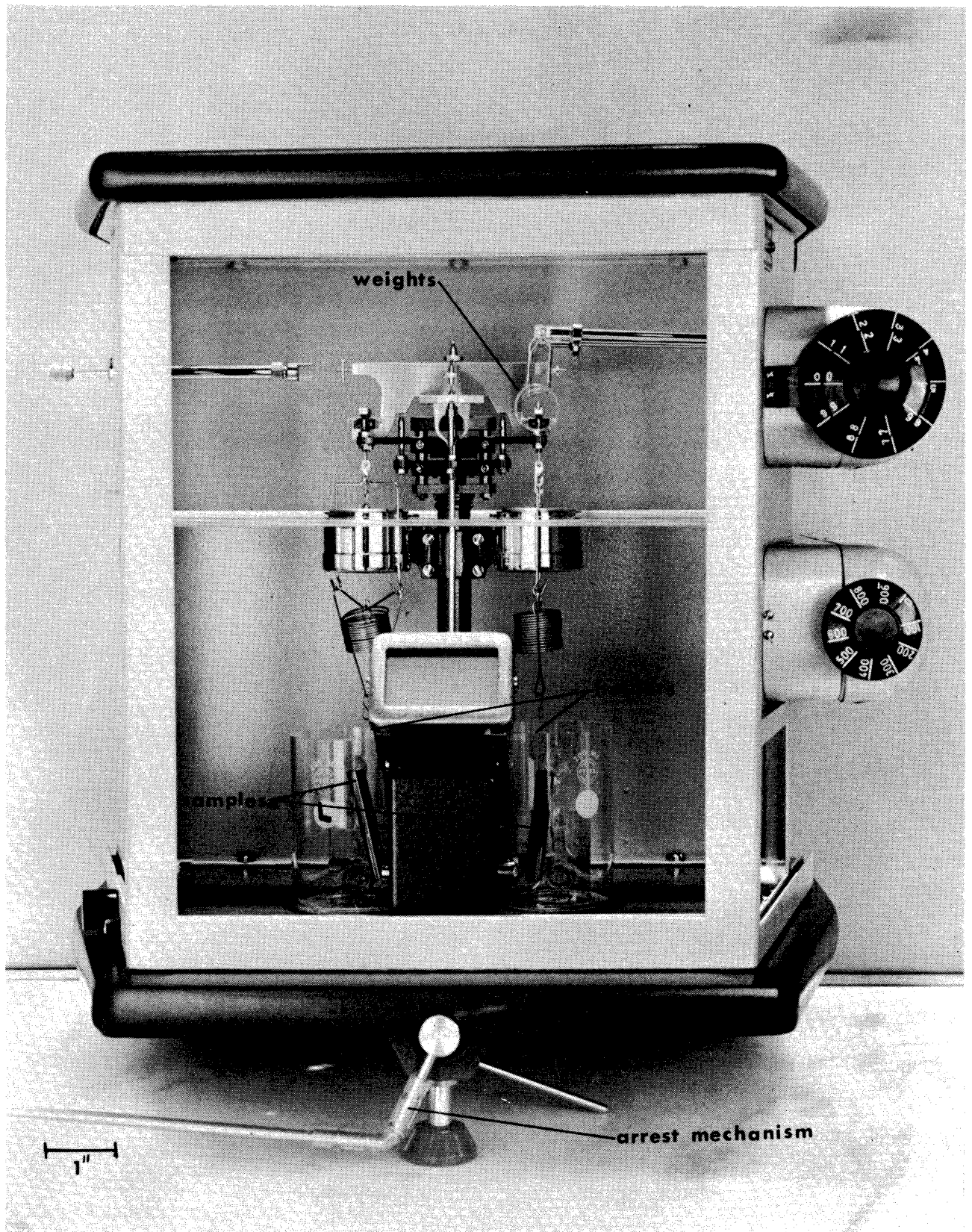
Figure 2. Microbalance as Adapted for Density Measurements.
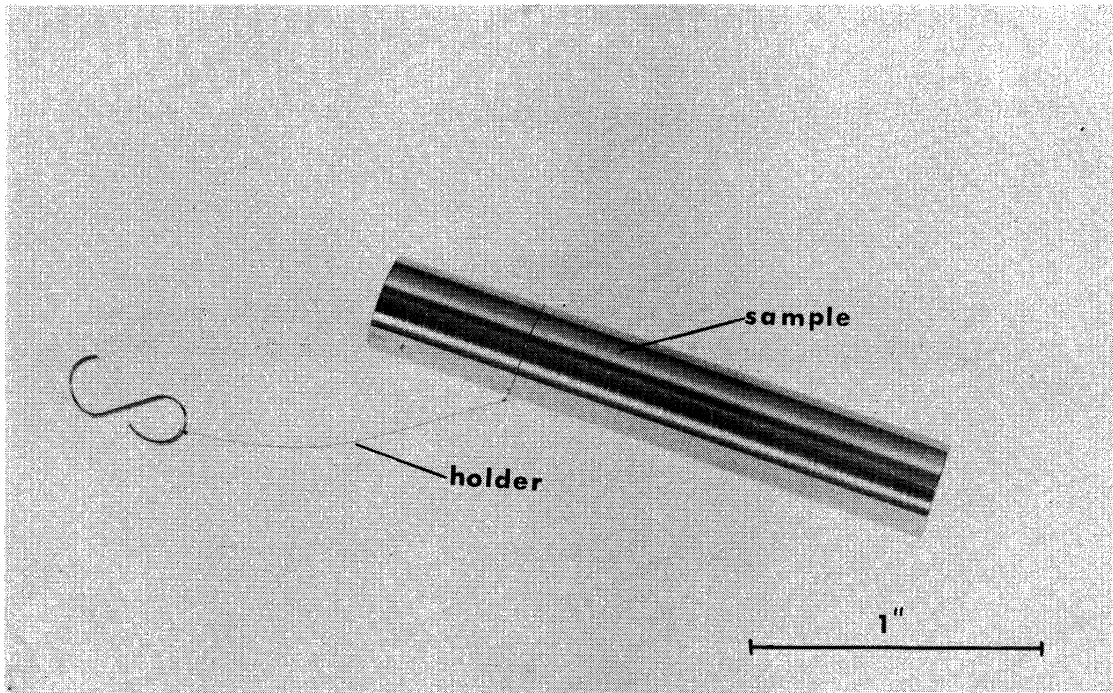
Figure 3A. Density Measurement Specimen and Specimen Holder.
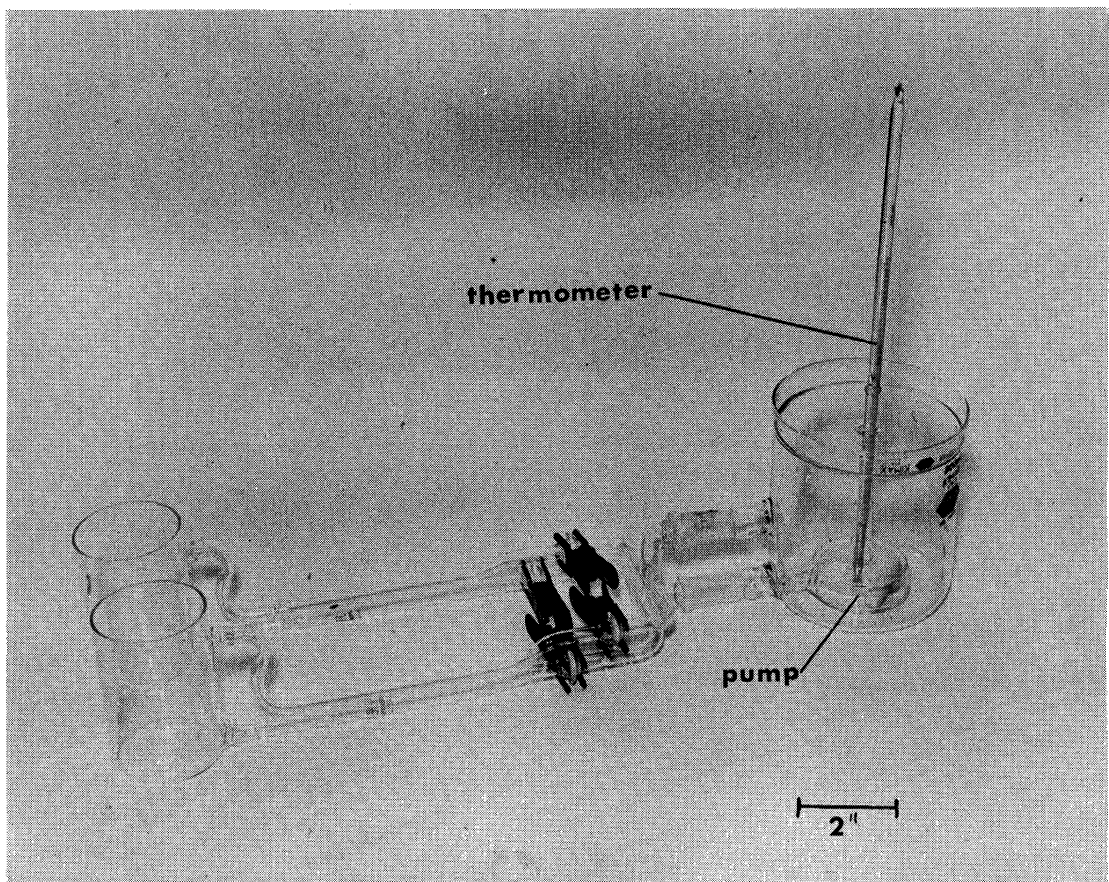
Figure 3B. System to Contain and Circulate the Liquid used for
the Density Measurement Weighings.
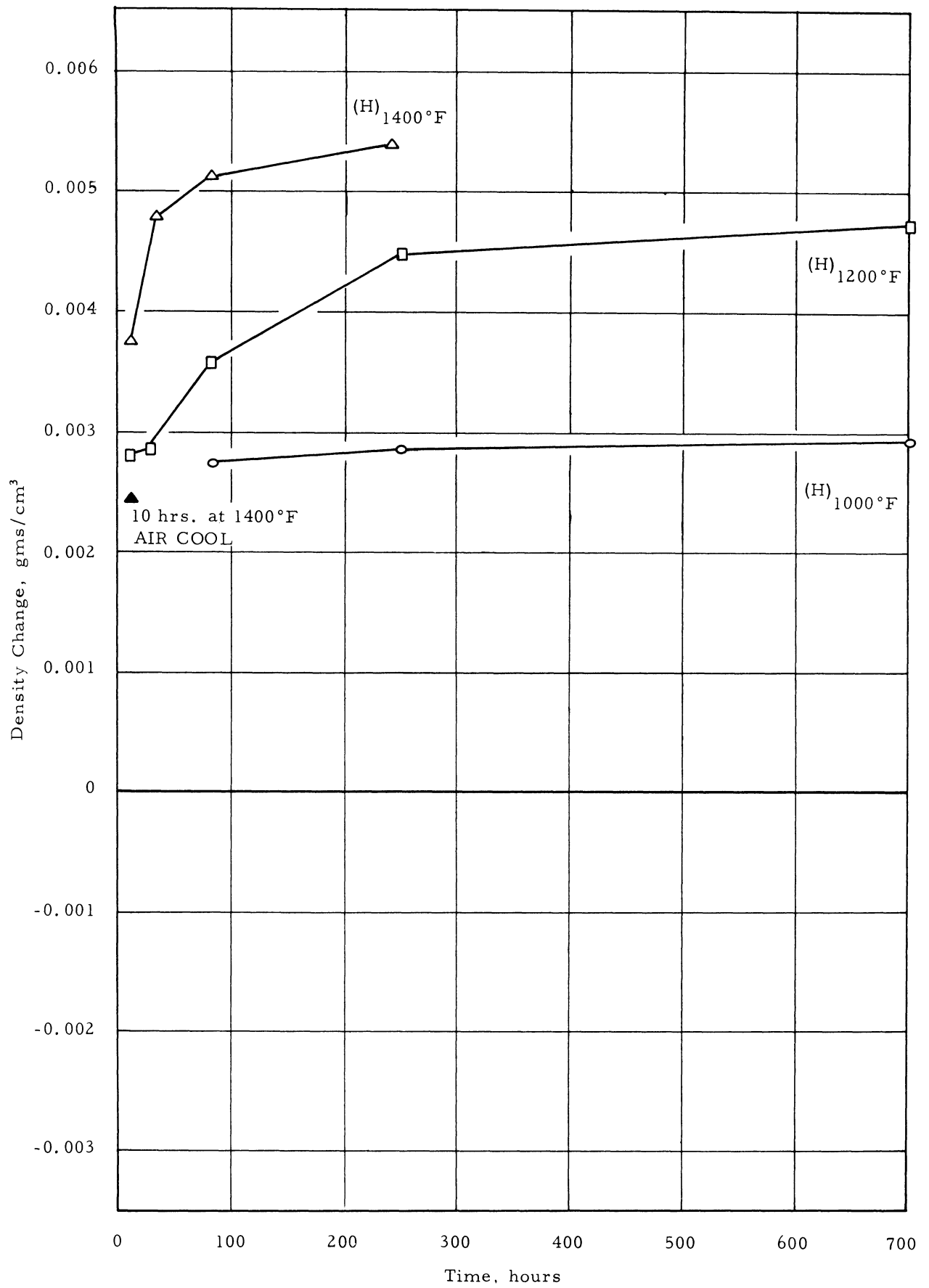
Figure 4. Unstressed Exposure Density Change versus Time for Chromel A
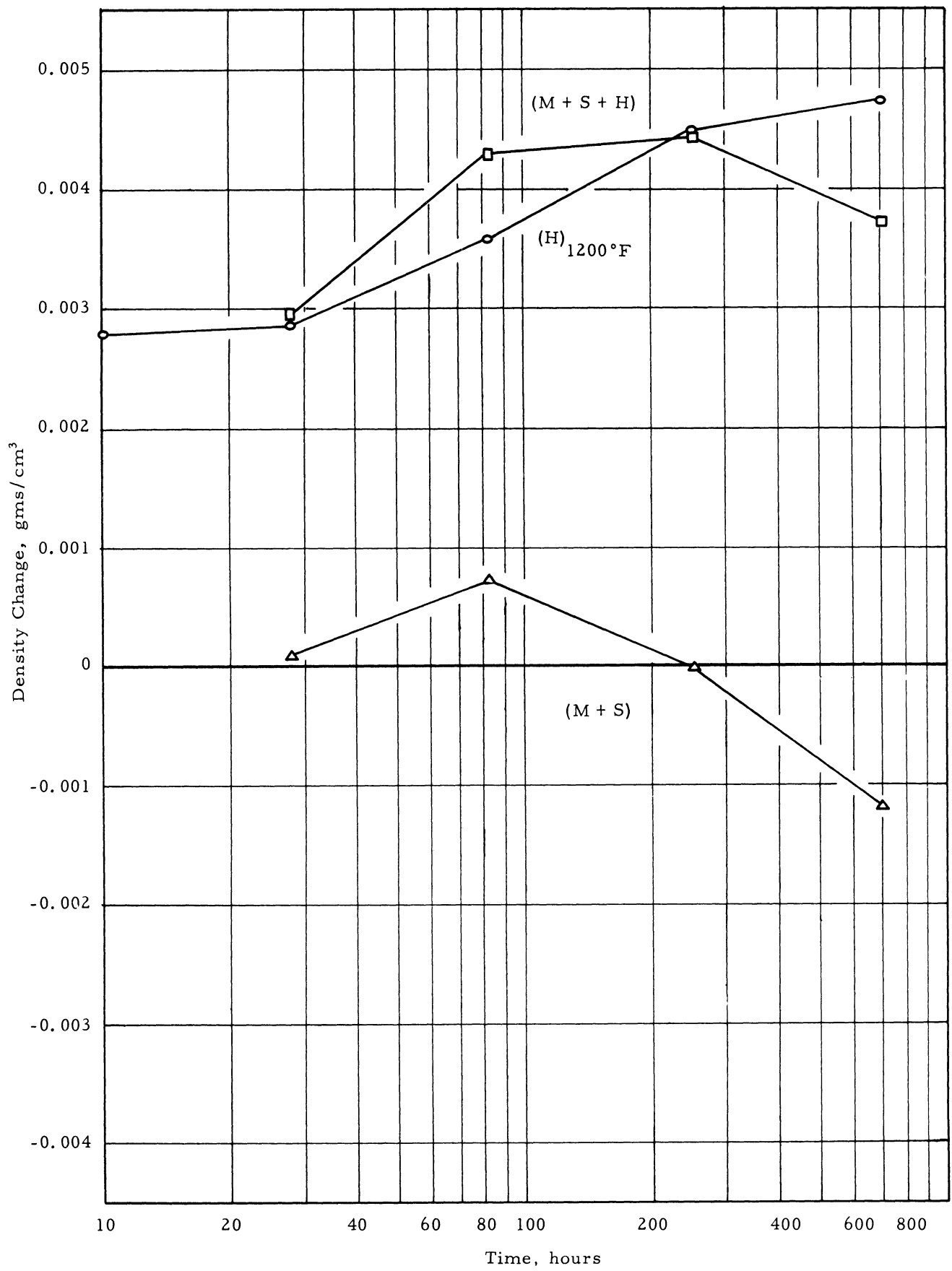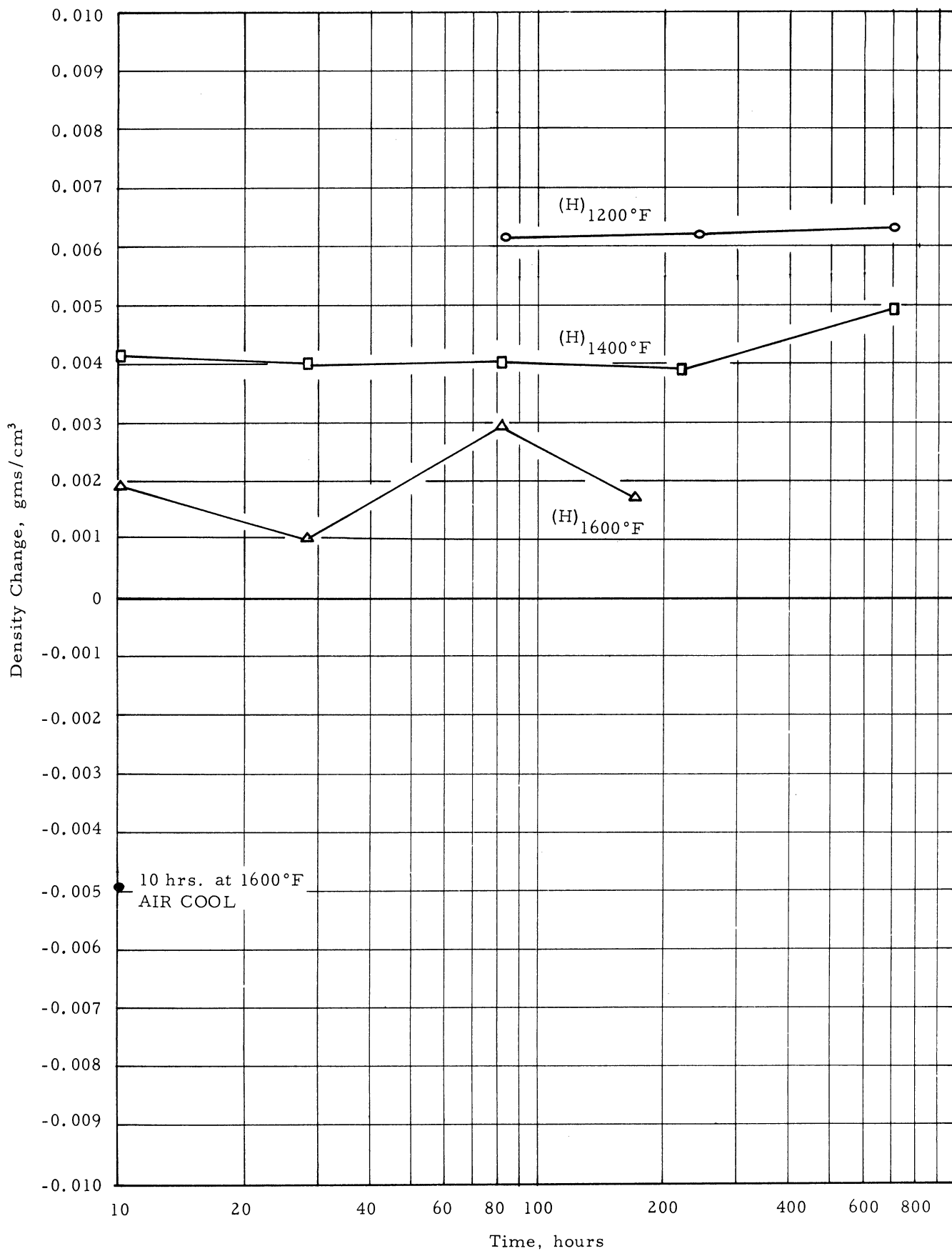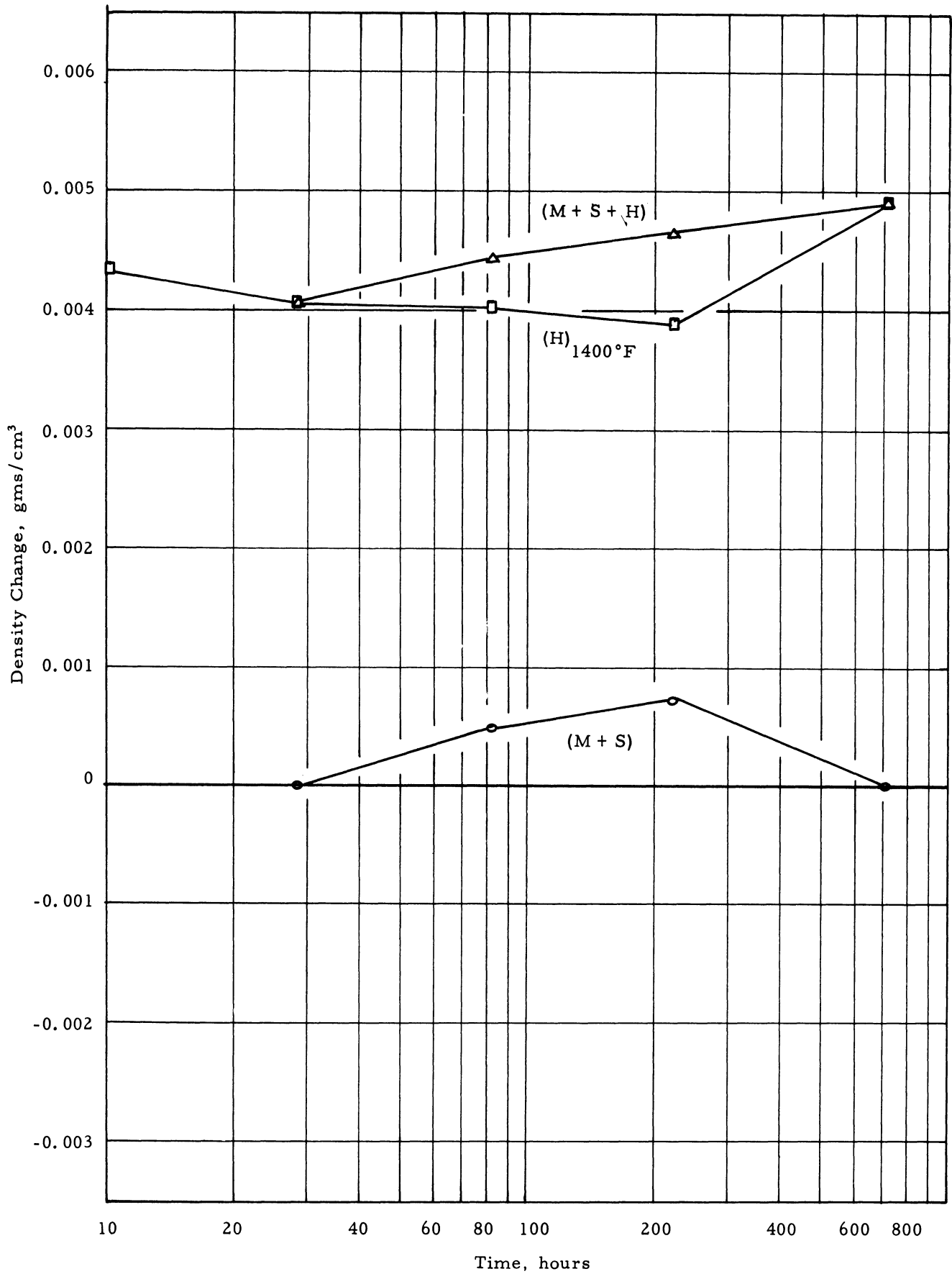at 1000°, 1200° and 1400°F.

33

Figure 5.   Creep Exposure Density Change versus Time for Chromel A at 1200°F ,
11,000 psi.   Expected Rupture Life 2,220 Hours.
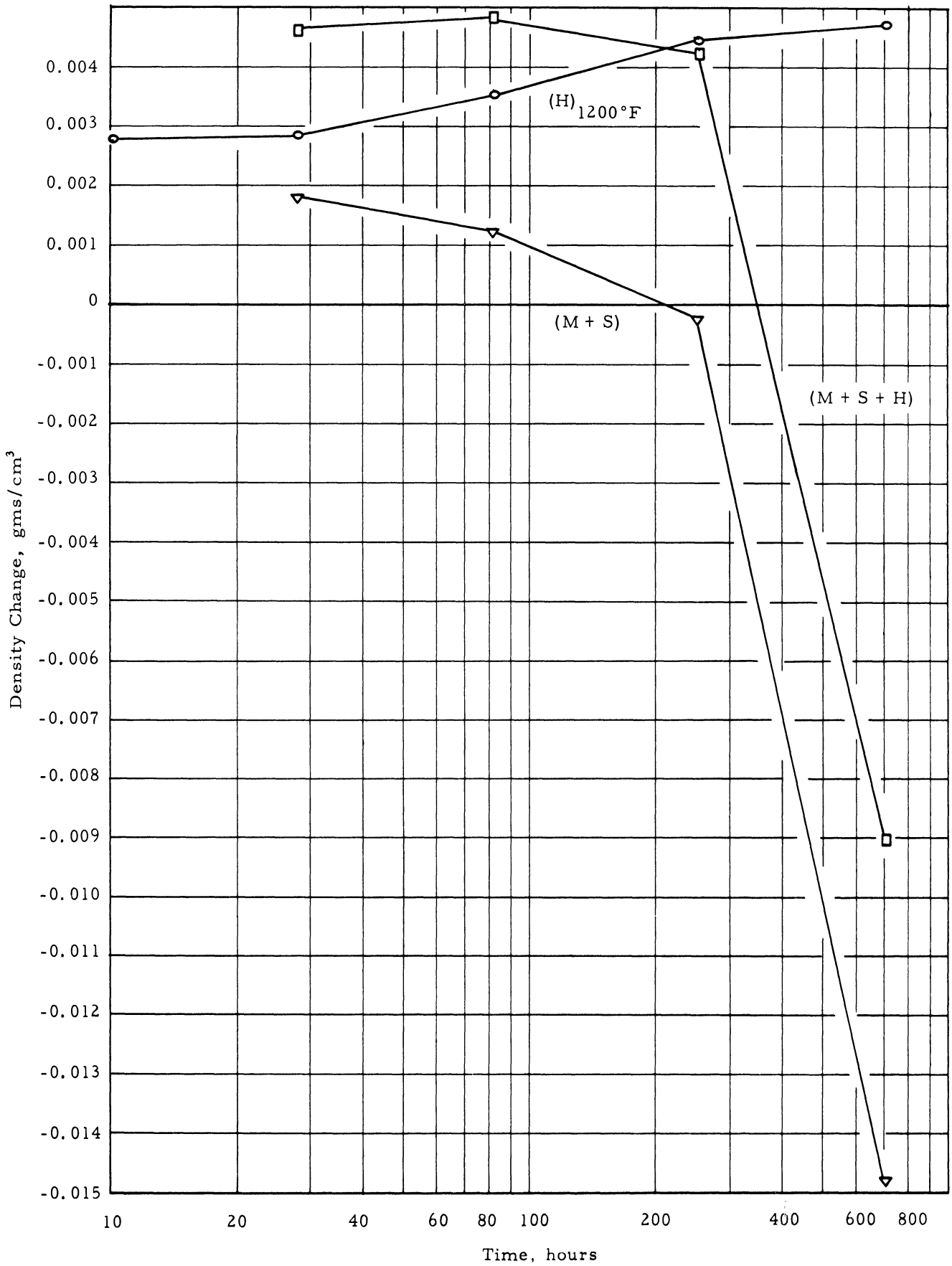
Figure 6. Creep Exposure Density Change versus Time for Chromel A at 1200°F.
14,000 psi. Expected Rupture Life 820 Hours.
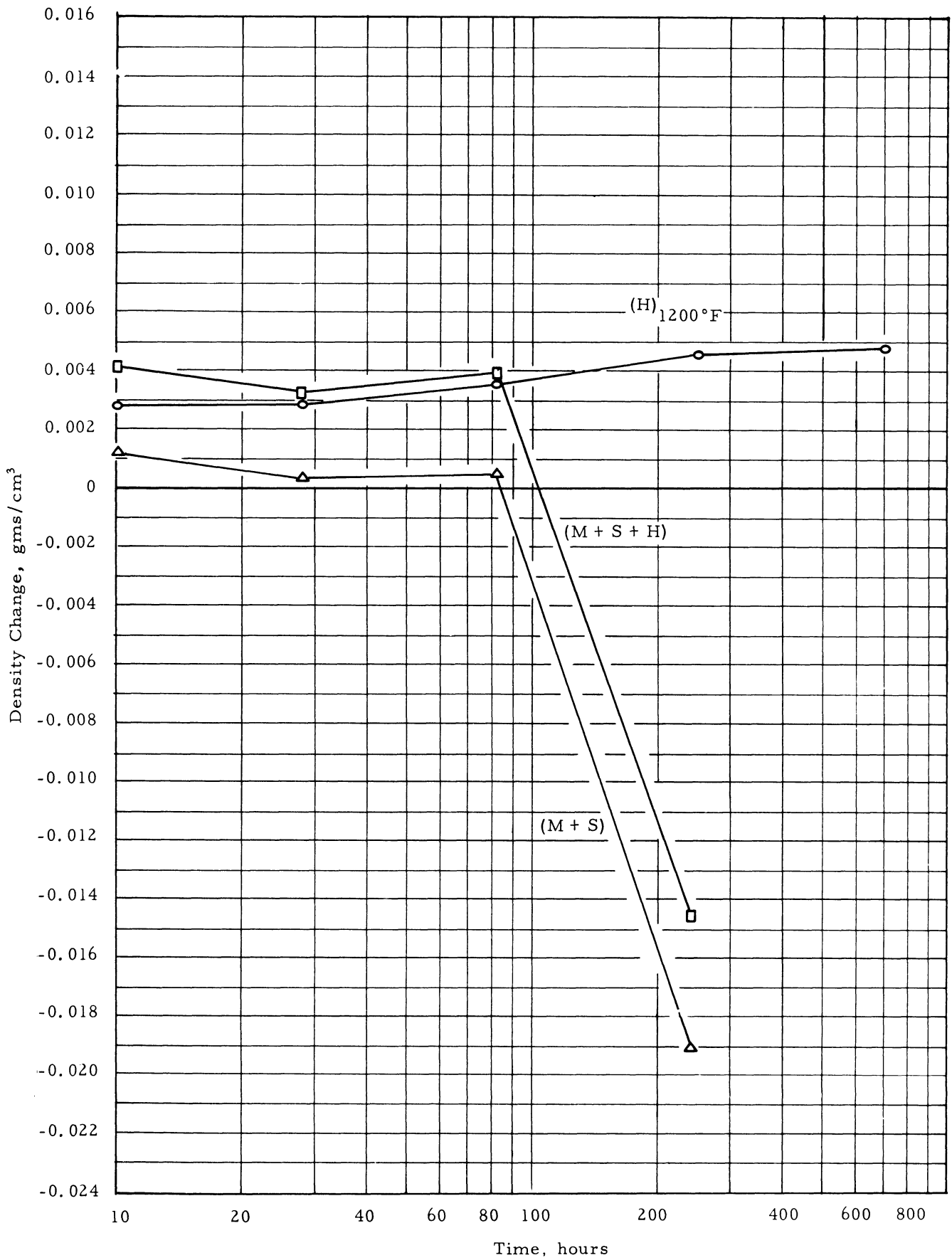
Figure 7.   Creep Exposure Density Changes versus Time for Chromel A at 1200°F,
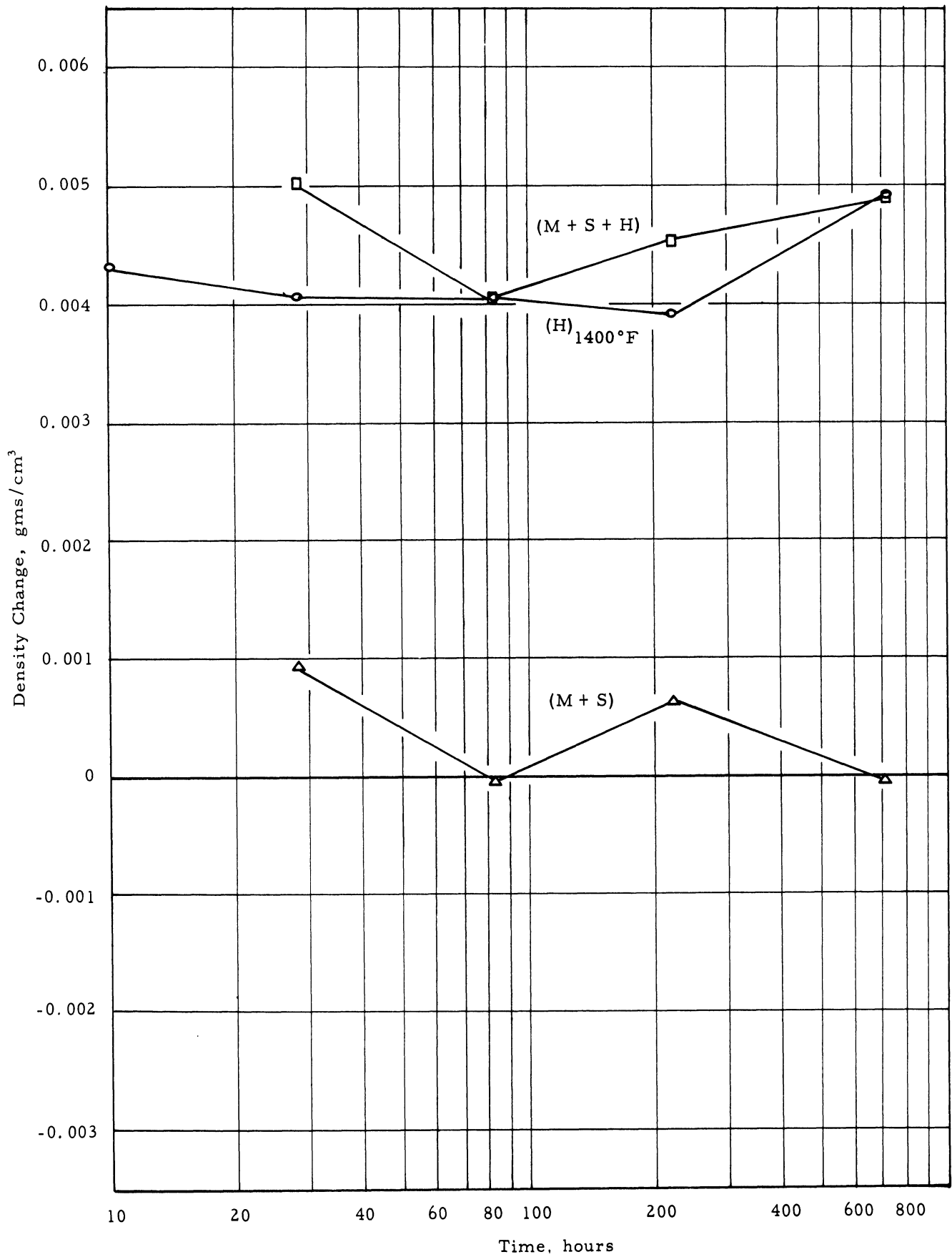18,000 psi.   Expected Rupture Life 300 Hours.

36
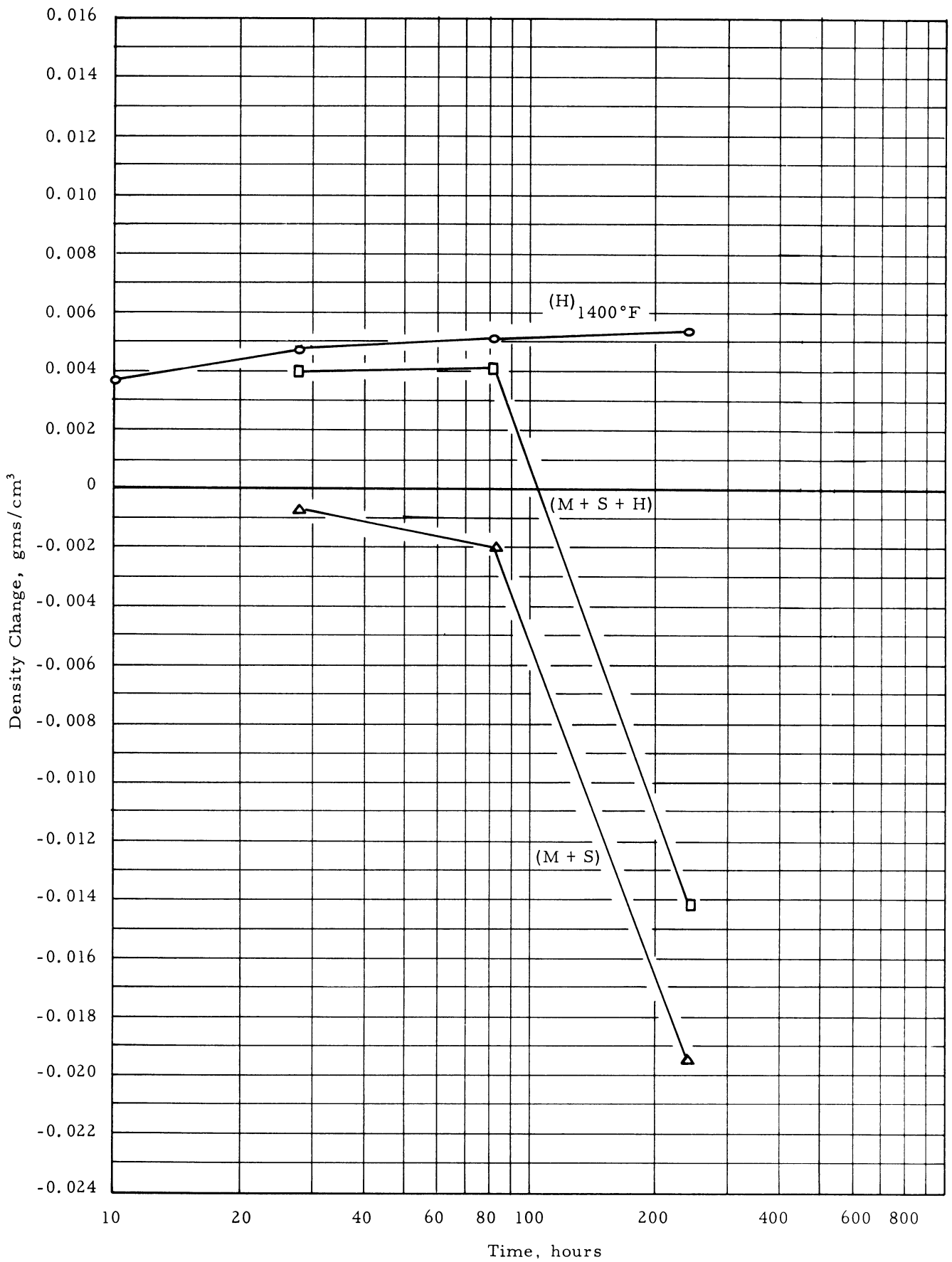
Figure 8.   Creep Exposure Density Change versus Time for Chromel A at 1400°F,
6,600 psi.   Expected Rupture Life 300 Hours.
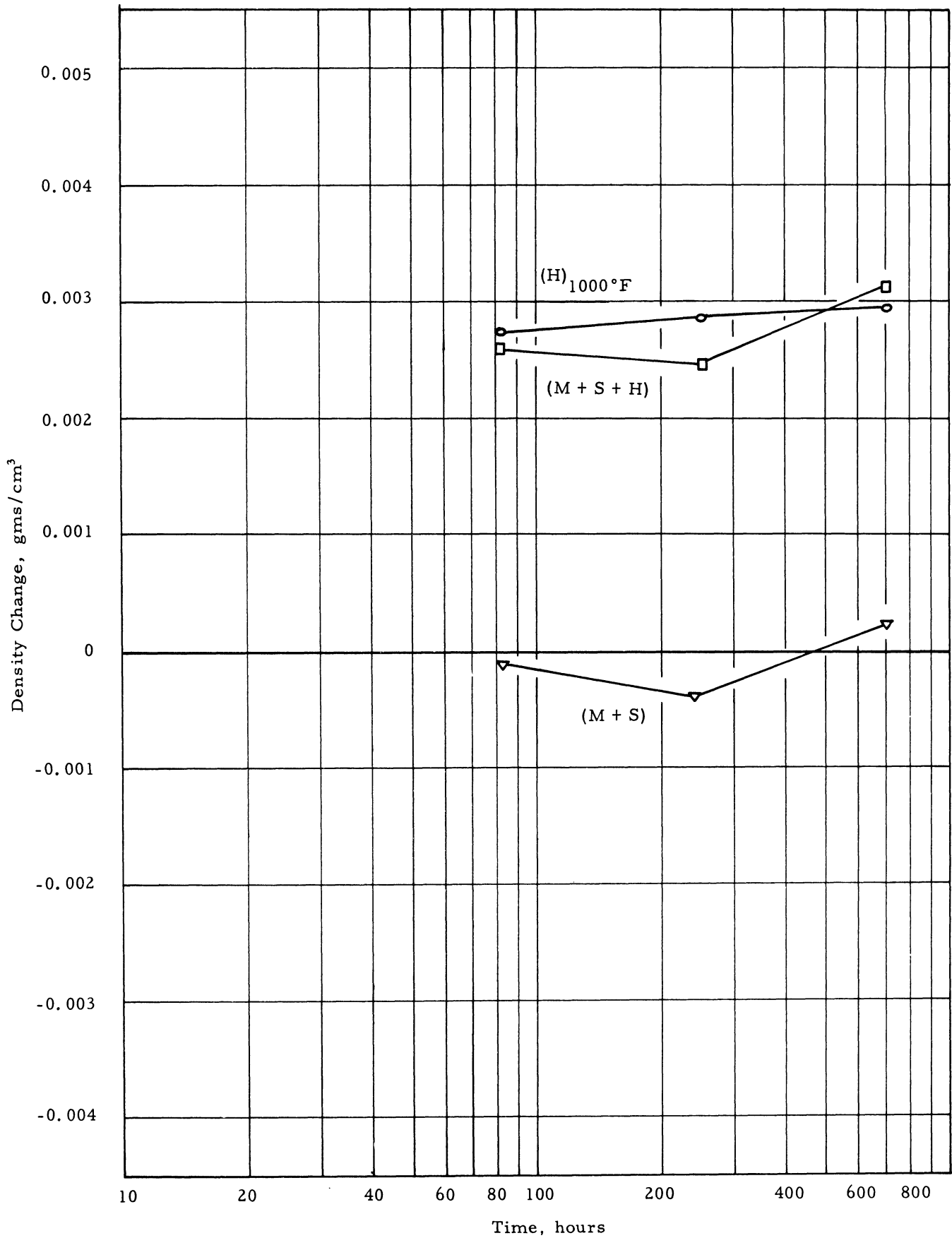
Figure 9. Creep Exposure Density Change versus Time for Chromel A at 1000°F, 39,500 psi. Expected Rupture Life 4000 Hours.
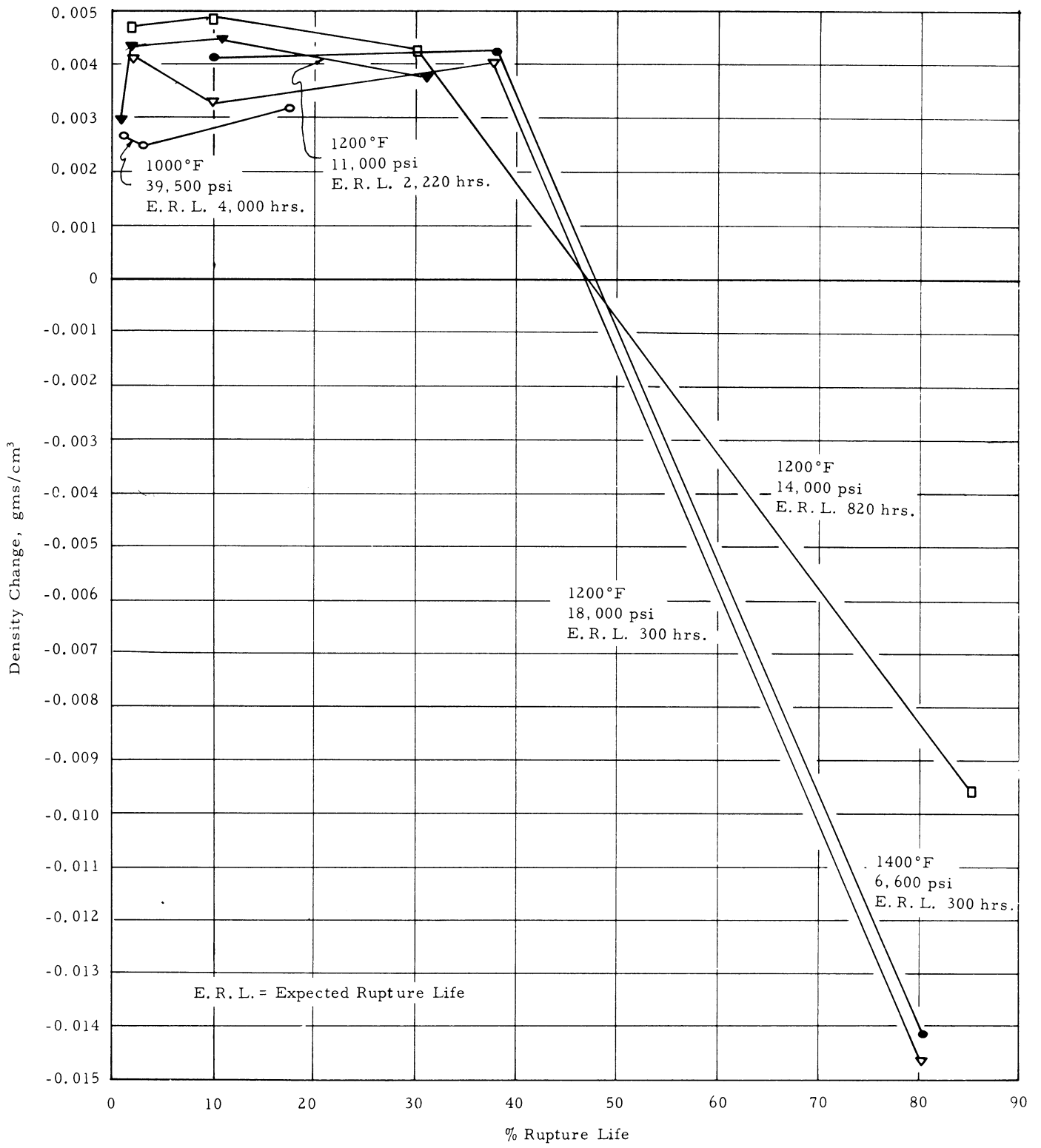
Figure 10. Total Density Change During Creep Exposure versus Percent Rupture Life for Chromel A.
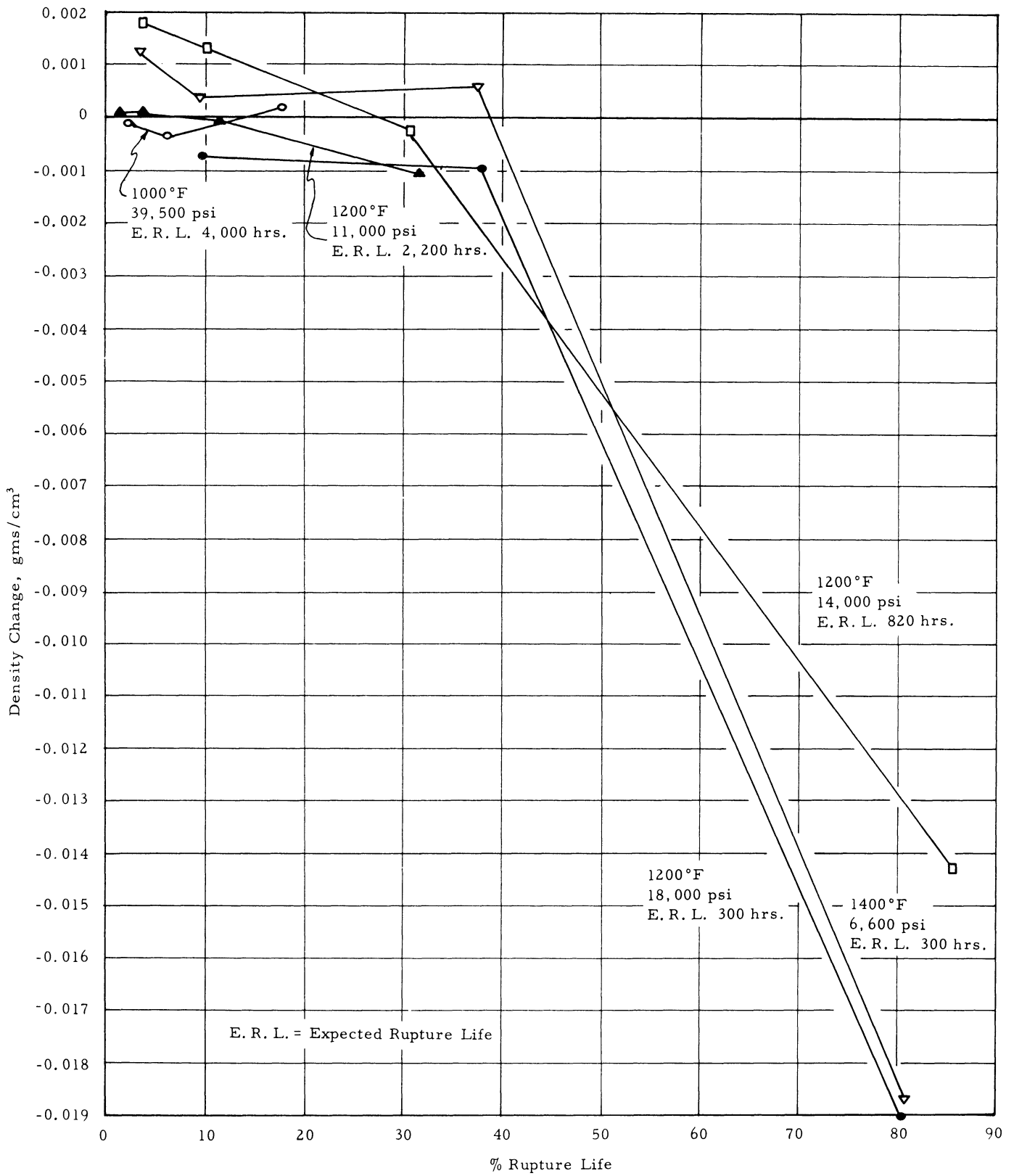
Figure 11. Stress-Induced Density Change (M+S) versus Percent Rupture Life for Chromel A.

Figure 12. Unstressed Exposure Density Change versus Time for Rene' 41 at 1200°, 1400° and 1600°F.

Figure 13. Creep Exposure Density Change versus Time for Rene' 41 at 1400°F,
37,000 psi. Expected Rupture Life 2,200 Hours.

Figure 14. Creep Exposure Density Change versus Time for Rene' 41 at 1400°F,
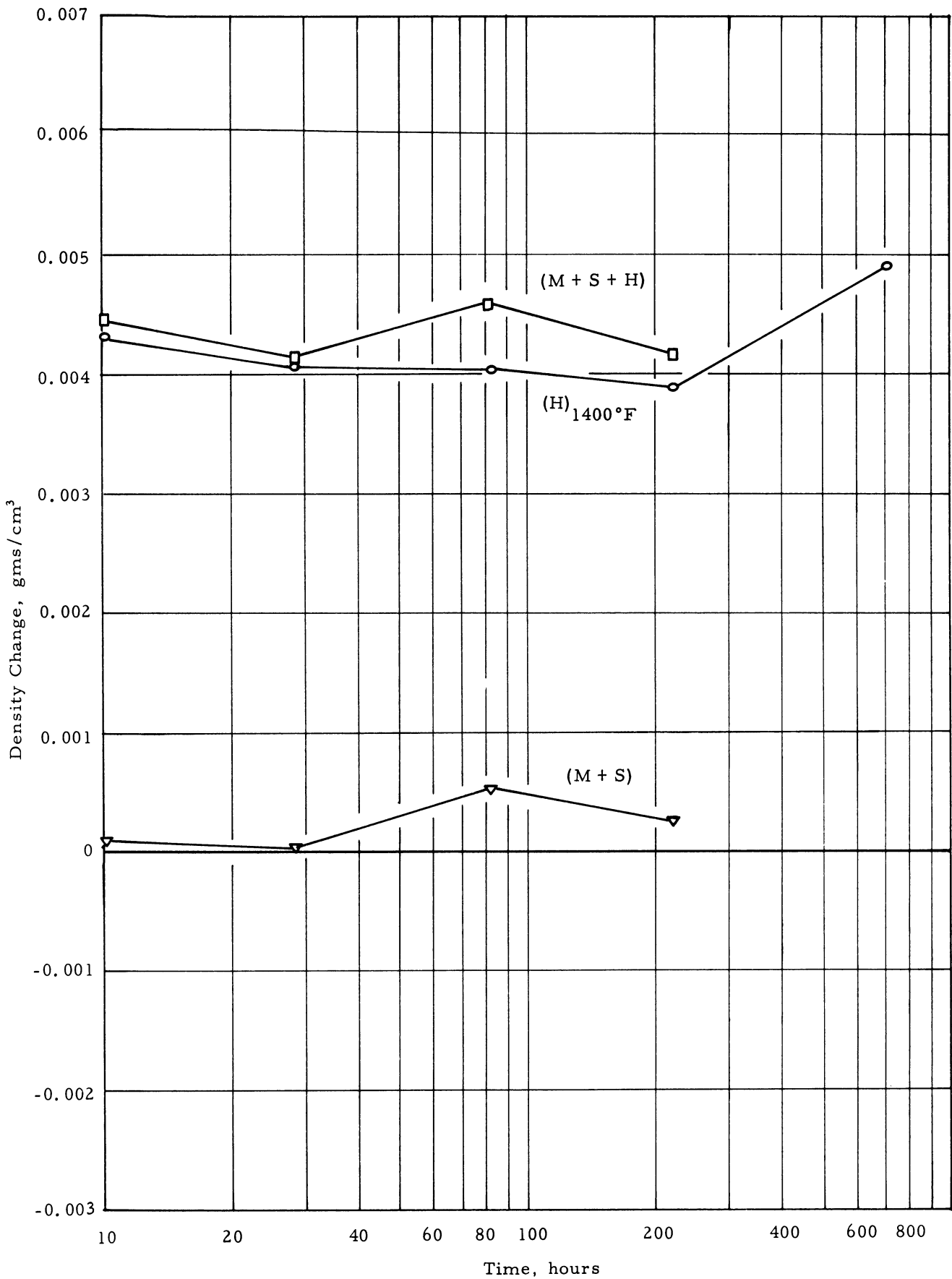46,000 psi. Expected Rupture Life 750 Hours.

Figure 15.   Creep Exposure Density Change versus Time for Rene' 41 at 1400°F,
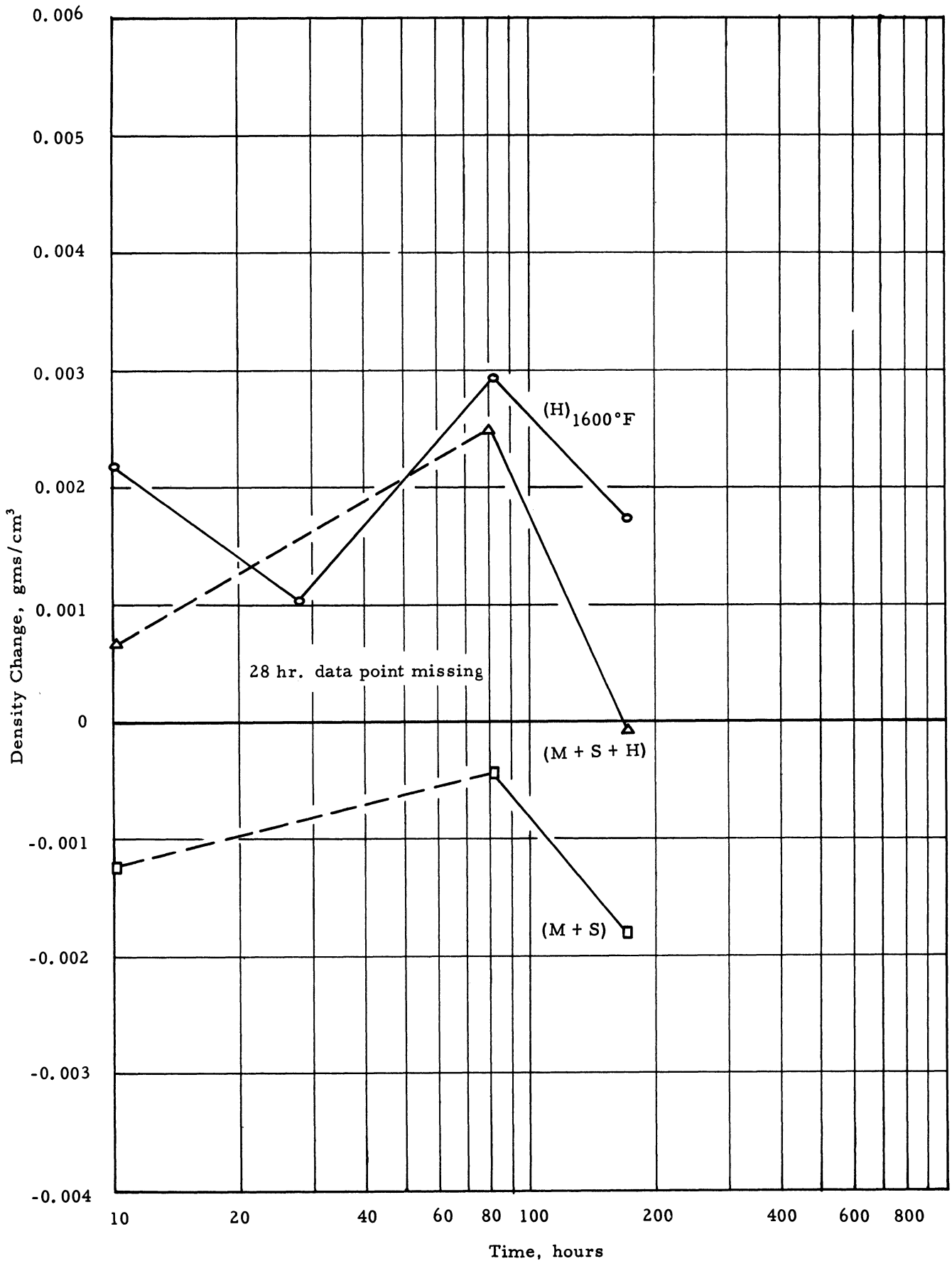59,000 psi.   Expected Rupture Life 238 Hours.

Figure 16. Creep Exposure Density Change versus Time for Rene' 41 at 1600°F, 24,000 psi. Expected Rupture Life 200 Hours.
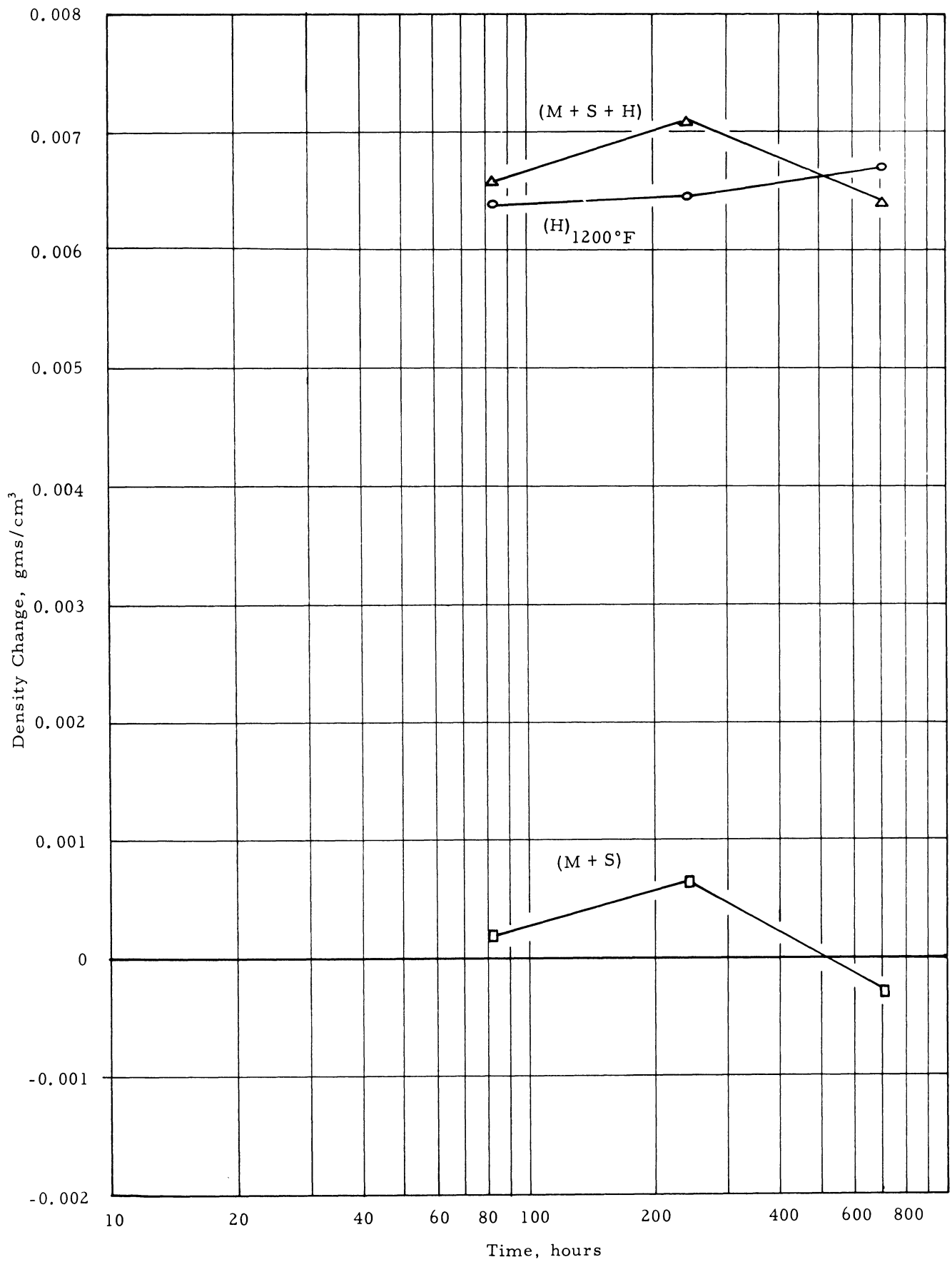
Figure 17. Creep Exposure Density Change versus Time for Rene' 41 at 1200°F, 105,000 psi. Expected Rupture Life 800 Hours.