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RELAXATION CHARACTERISTICS OF INCONEL AT ELEVATED TEMPERATURES

C. R. Kennedy
D. A. Douglas

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METALLURGY DIVISION

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C. R. Kennedy and D. A. Douglas

ABSTRACT

A stress-relaxation machine has been developed capable of providing very precise results throughout the useful temperature range of common structural metals. Among the advantages claimed for this machine are: dynamic response, sensitive control, and low cost.

It is demonstrated that with the use of a single specimen and relatively short testing times, a complete set of stress-strain rate values can be obtained at any desired temperature. The reliability of the testing device and the analytical model is shown in the comparison of values predicted from relaxation tests with those obtained in constant-stress creep tests.

It should be emphasized that the relaxation test is not a substitute for the creep test. However, it does provide a check on the accuracy of the law assumed for the constant-stress creep data and provides additional information which assists the metallurgists in establishing more exact analytical models to describe the deformation process.

INTRODUCTION

Many of the structural problems which arise in high-temperature design stem from the occurrence of time-dependent plastic deformation. A material's resistance to deformation can be measured by a variety of test types such as the constant-stress creep test and the stress-relaxation test. The present discussion deals primarily with the relaxation characteristics of a common nickel-base alloy, Inconel (nominal composition 15% Cr, 5% Fe, bal Ni). As used in this report, relaxation means the replacement of an initial elastic strain with plastic strain through creep. Since only the elastic strain can transmit stresses, the relaxation process can be measured through changes in the stress required to maintain a constant total strain.

As in the case of creep, the phenomenon of stress-relaxation is time and temperature dependent and assumes importance in the performance of materials under a number of types of stress conditions. The data produced by relaxation testing can be applied directly to problems where stresses are maintained by elastic strain. Some of the typical design applications involving the use of elastic strain to maintain stress levels above a minimum working stress are the use of bolts to sustain flange seals, shrink or press fits, rolled in tubes, and springs. In these examples, lowering of the working stresses during service at elevated temperatures due to relaxation could impair the service performance. Likewise, stress gradients caused by design geometries or by temperature gradients are subject to redistribution through relaxation so that a more uniform gradient is achieved. The rate at which this will occur is determined by the relaxation characteristics of the material. Thus, elastic-stress concentration can be lowered without appreciable damage to the structure if rapid relaxation occurs. The removal of residual stresses by annealing is controlled by stress-relaxation and, therefore, relaxation data can be used to develop stress-relief treatments after fabrication by welding, forging, cold working, and machining.

Relaxation testing can also be used as a sensitive method of providing reliable information as to the long-time constant stress behavior of a material. Under certain conditions, these results, through direct computation, yield exact constant-stress creep strength values. It is not proposed, however, that the relaxation test surplant the constant-stress creep test, but rather, that it provides a means for rapid evaluation of new materials or old materials under more extreme conditions. Results from both types of testing enable the metallurgist to establish more exact analytical models to describe the deformation process and they provide the designer with strength values of higher quality.

PROCEDURE

Many different testing machines and methods have been used by previous experimenters[1-5] to determine the relaxation characteristics of metals; however, most of the work has been at relatively low temperatures. Since extrapolation of these results to higher temperatures does not give reliable values, actual test data must be obtained at the desired temperature. For this purpose, it was necessary to devise new experimental techniques to achieve the accuracy desired in the temperature range of 1200 to 2000°F.

The primary effect of increasing temperature in this type of test is to increase the initial rate of relaxation. The test machine must, therefore, incorporate a low-inertia loading mechanism capable of loading a specimen at a rapid rate and then immediately reversing and unloading at a rate sufficiently rapid to maintain a constant total strain. In the case of Inconel at 1650°F, half of the initial stress is relaxed in 18 sec. A pneumatic motor, similar to a valve operator, was found to have the required features and was incorporated in the relaxation machine design shown in Fig. 1. The motor has a diaphragm of approximately 330 sq. in. and is capable of applying a 10,000-lb. force. A schematic drawing of the motor with the solenoid control circuit is shown in Fig. 2.

The method of controlling the load required to achieve and maintain a desired strain depends upon the flow rate of air to the motor. Because of the large volume of air in the motor, the pressure can be regulated by simple solenoid valves operating off and on. An extensometer similar to the one developed at Westinghouse[1] is used to measure strain and was modified so as to control the load. This extensometer, shown in Fig. 3, has a 120 to 1 magnification ratio and measures strain over a 6-in. gauge length specimen shown

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[1] A. Nádai and S. Boyd, "Relaxation of Steels at Elevated Temperatures. A New Automatic Relaxation Machine," J. Appl. Mechanics, Trans. ASME 60, A-118 (Sept. 1938).

[2] J. J. Kanter, "Interpretation and Use of Creep Results," Trans. ASM 24, 900 (Dec. 1936).

[3] E. L. Robinson, "Metals at High Temperature — Test Procedure and Analysis of Test Data," J. Appl. Mechanics, Trans. ASME 55, 145–150 (1933).

[4] John H. M. Draper, "Creep Relaxation Testing — Tests at Constant Strain and Decreasing Load," Eng. J., p 564–565 (May 6, 1955).

[5] W. Barr and W. E. Bordgett, "An Accelerated Test for the Determination of the Limiting Creep Stress of Metals," Trans. Inst. Mech. Engrs. 122, 285–297 (1932).

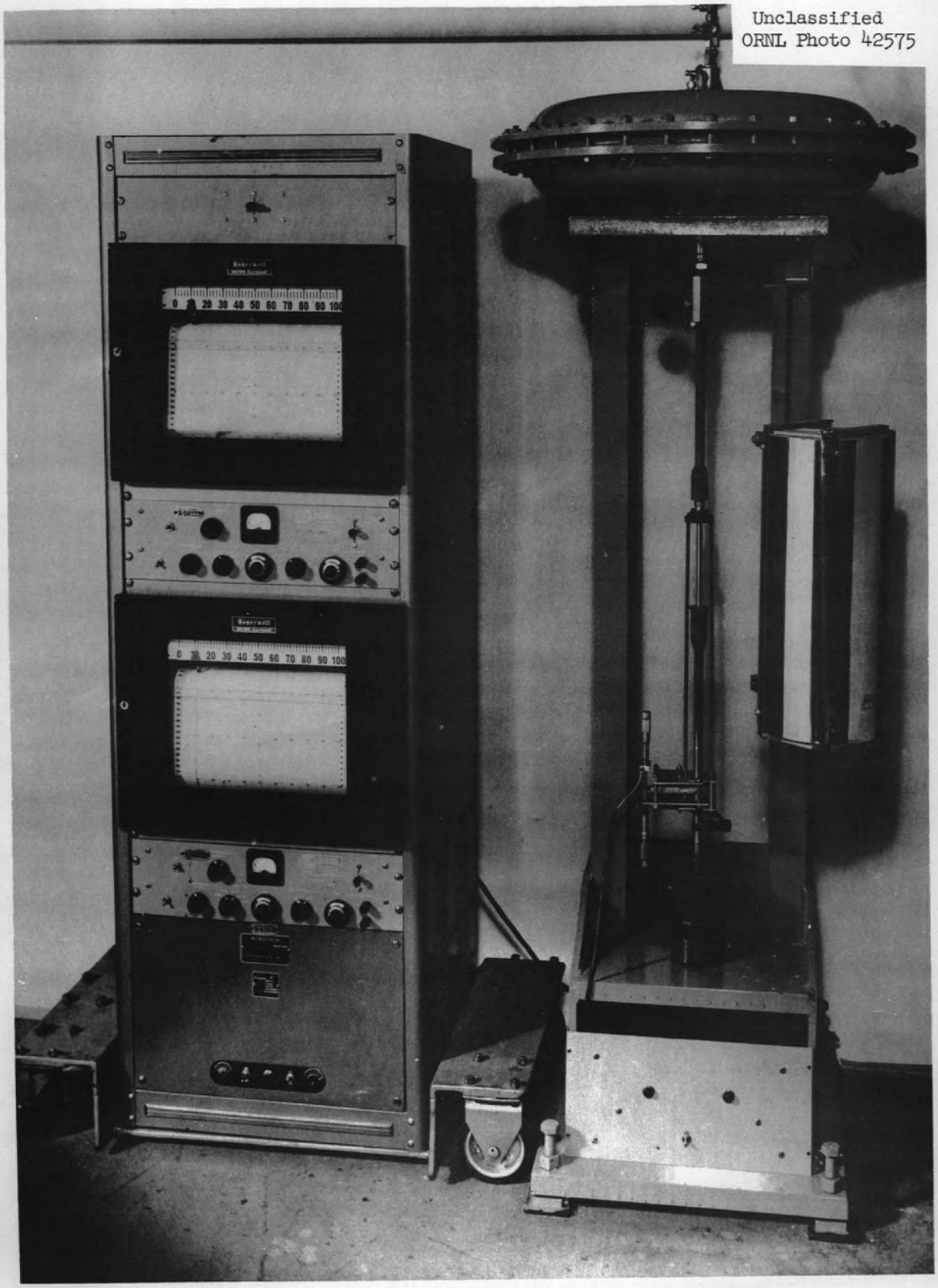

Fig. 1 Relaxation Machine.

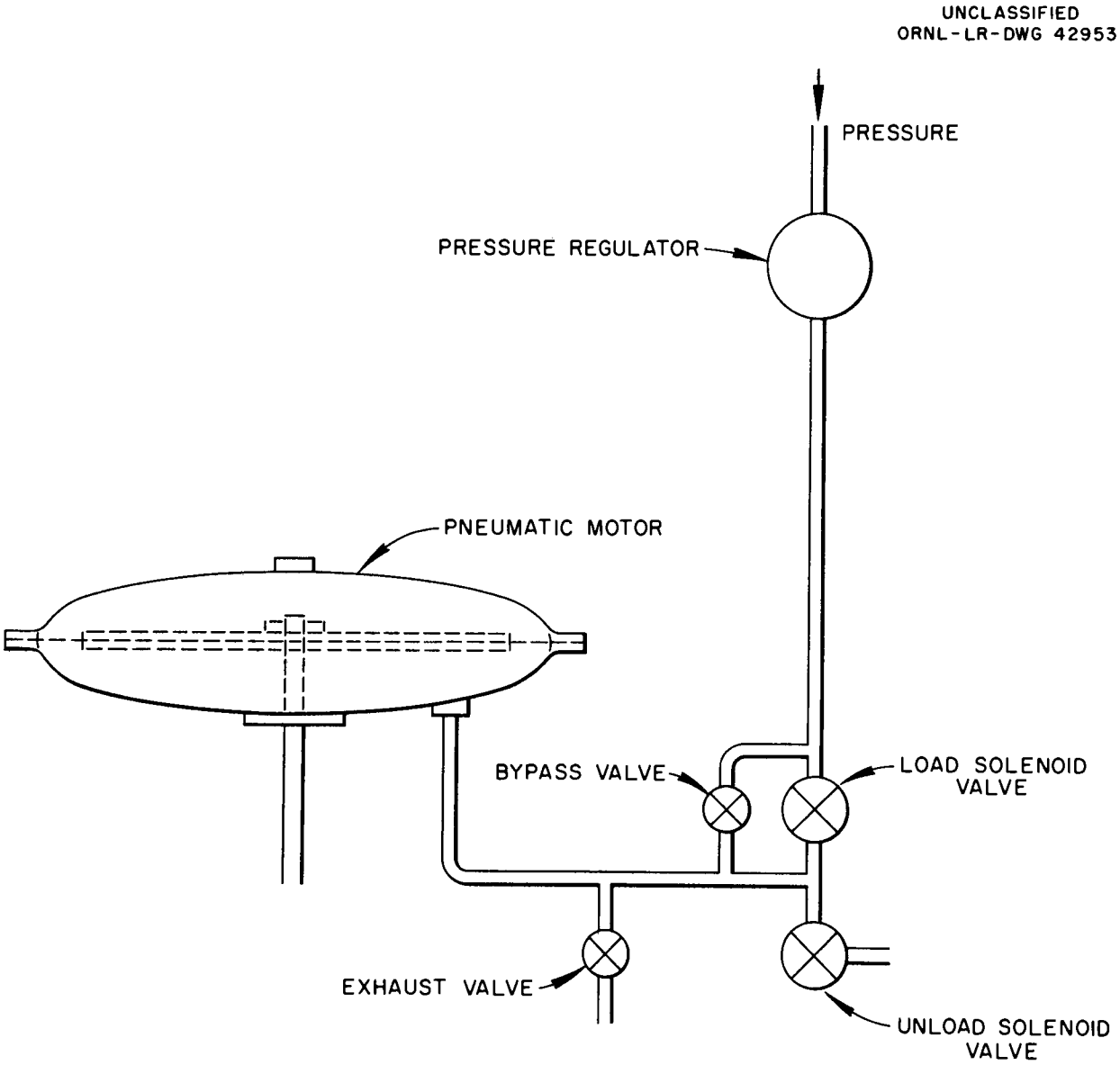

Fig. 2. Schematic Drawing of the Solenoid Valve Circuit.

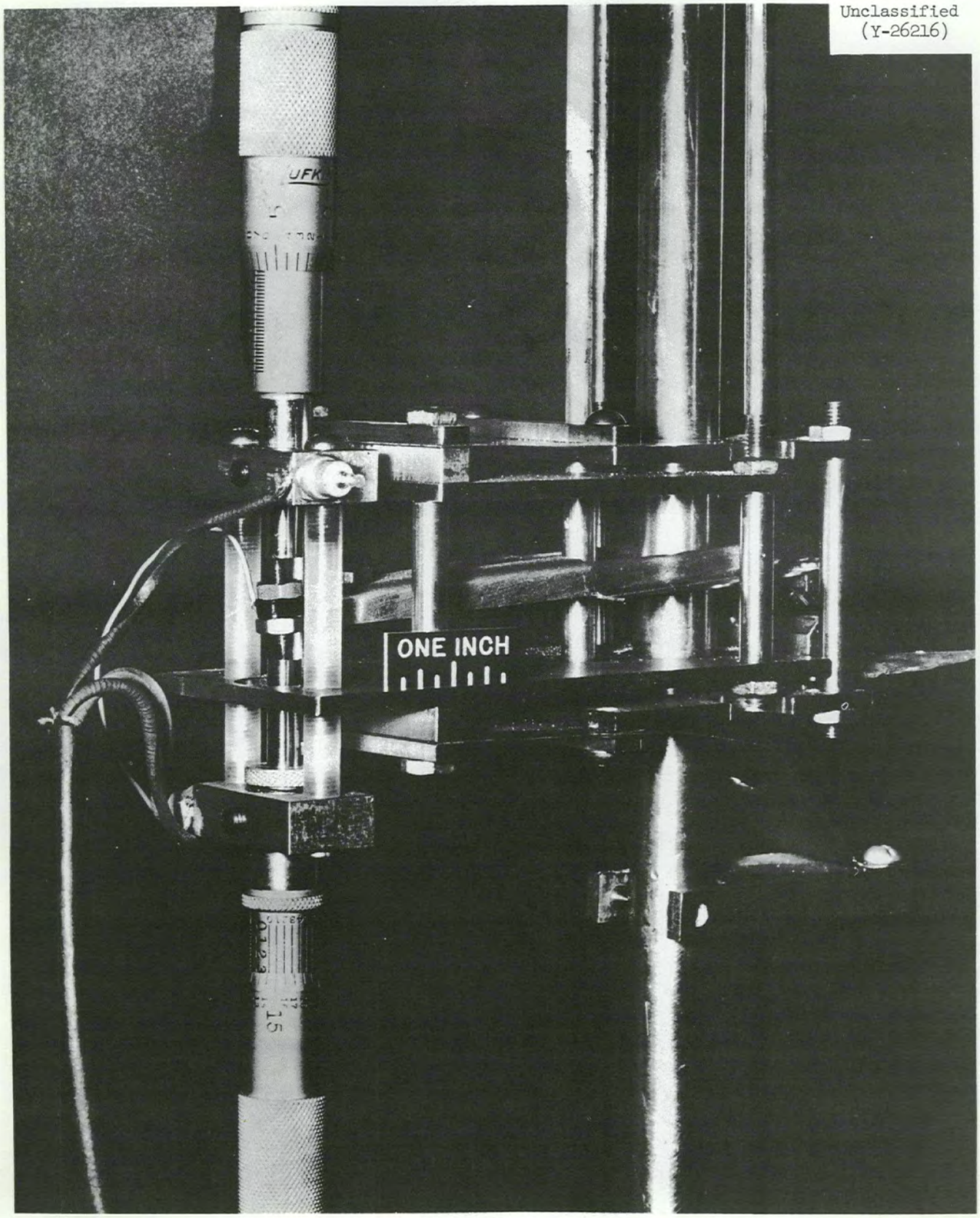

Fig. 3 Extensometer Used in Relaxation Testing.

in Fig. 4. The micrometer heads establish the position of electrical contact; the top contact signals for load application and the lower contact signals for the release of the load. When the extensometer makes contact, the grid bias in a grid glow relay is cut out. The relay then actuates the proper solenoid valve and thereby controls the pressure in the pneumatic motor. A view of the solenoid valve control circuit is shown in Fig. 2. By proper adjustment of the supply pressure one can obtain strain rates on loading from 0.001 to 0.1 in./in./min.

The furnace is shunt-wound and is controlled by a proportioning type controller capable of maintaining the temperature within ± 2°F. By proper shunting of the furnace a gradient of less than 5°F can be achieved over the 6-in. gauge length of the specimen.

Before the test is initiated, it is necessary to hold the specimen at temperature until the gradient is acceptable and the furnace has reached thermal equilibrium. The test is then run by setting the micrometer screws to obtain the desired strain, regulating the supply pressure to produce the proper strain rate, and by turning on the electronic relay. The test from this point is completely automatic and requires only periodic checks of the temperature control and gradient.

A load cell of the appropriate capacity is used to measure the stress applied to the specimen. The signal from the load cell is calibrated by a modified Transducer Calibration Adapter Model Q-902 built by the Oak Ridge National Laboratory's Instrument Division. The signal is continuously recorded on a strip-type potentiometer with a two-speed chart drive. A high chart-speed of 12 in./min is used to record the first 30 min of each test and a low chart-speed of 6 in./hr is used for the remainder of the test. In most cases, the tests are discontinued after 100 hr.

The listed results are from tests performed on Inconel, Heat NX 4364, with two grain sizes — a fine-grain size in the as-received material comparable to a 1650°F anneal, and a coarse-grained structure obtained by re-annealing the as-received material at 2050°F for 2 hr. Photomicrographs of the Inconel in the two conditions are shown in Figs. 5 and 6. Most of the data were obtained from single specimens at one temperature. Each specimen was first tested at a

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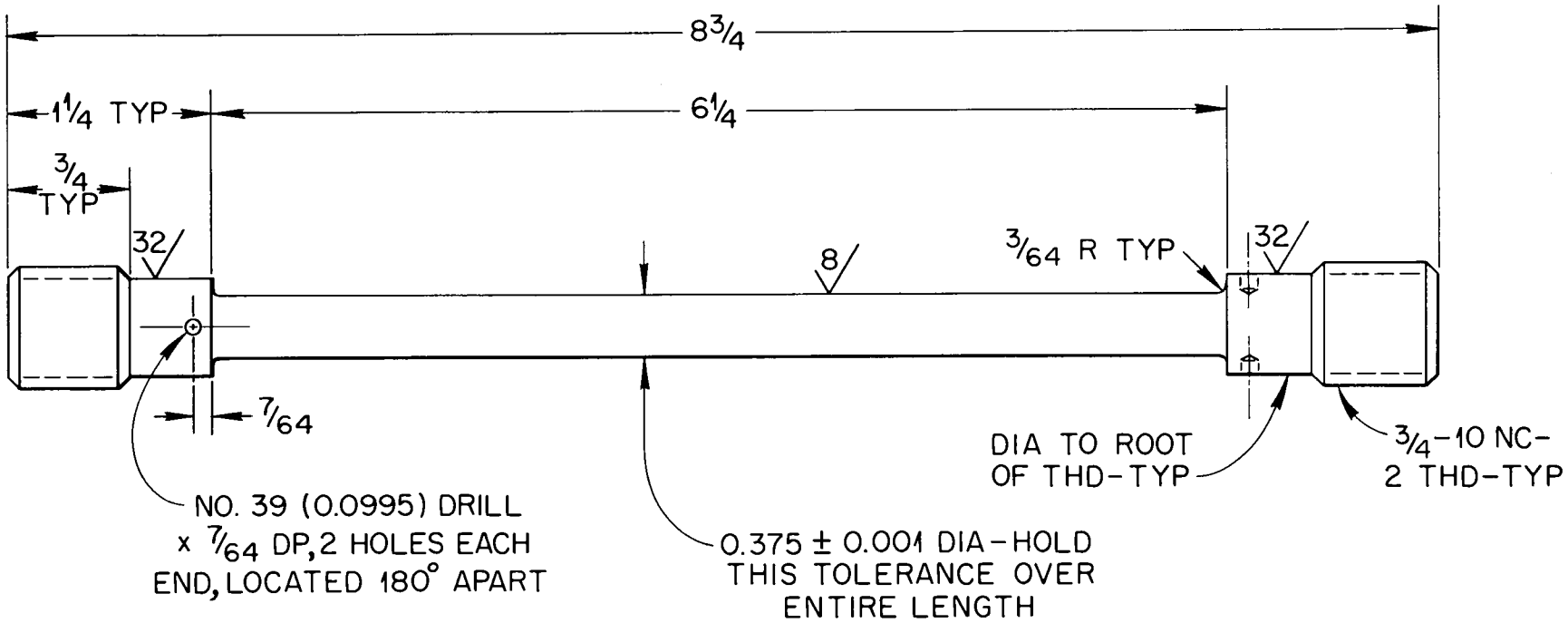

Fig. 4. Relaxation Specimen.

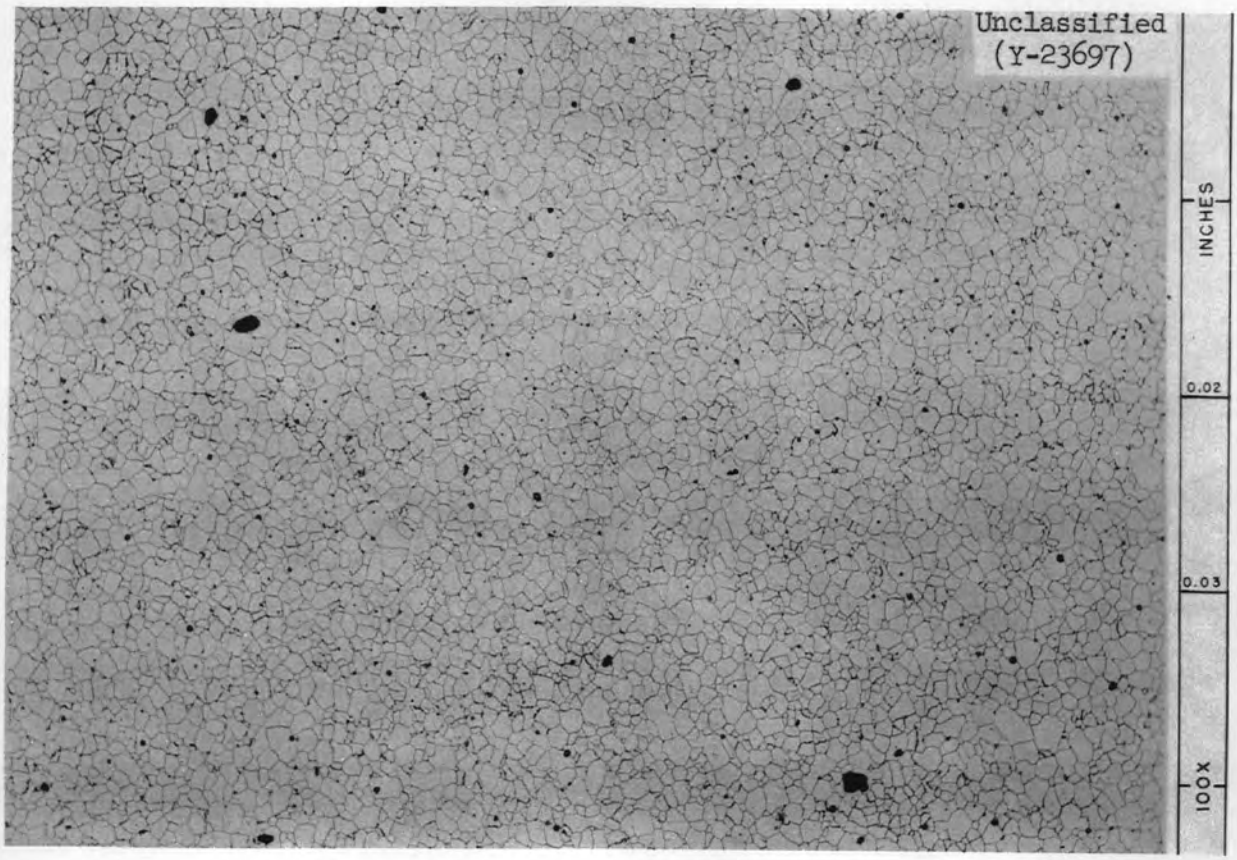

Fig. 5 Photomicrograph of As-Received Inconel.

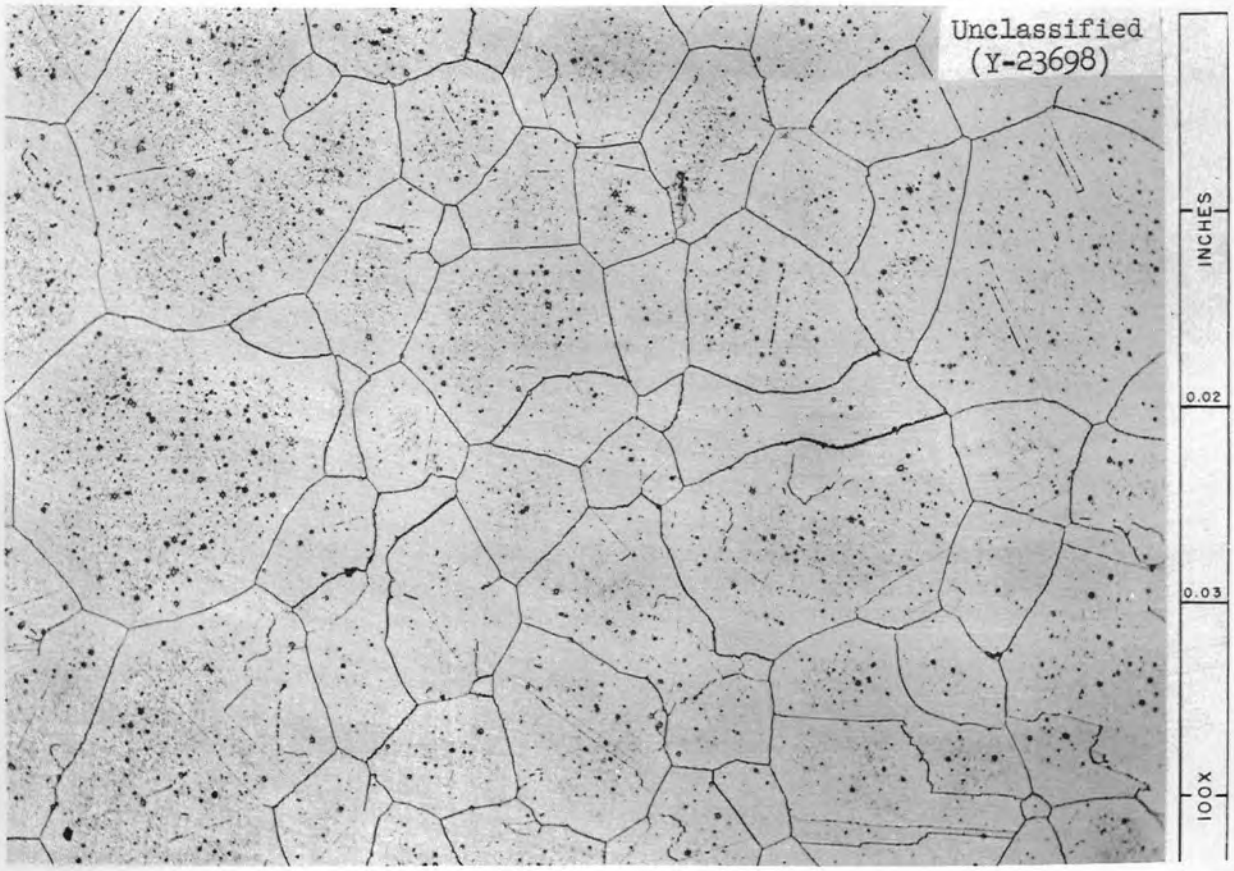

Fig. 6 Photomicrograph of As-Received Inconel Annealed at 2050°F for 2 Hr.

strain of 0.05% and then retested at this same strain to determine the effect of prior plastic strain on the relaxation rate. When no effects were observed, the same specimen was tested with higher strains, duplicating each test.

RESULTS AND DISCUSSION

The results of relaxation testing of Inconel at temperatures of 1300, 1500, and 1650°F are shown in Figs. 7 through 12. Results shown in Figs. 7 through 9 are those obtained from testing as-received material and in Figs. 10 through 12 are those obtained from testing Inconel annealed at 2050°F for 2 hr in hydrogen. The differences between the results obtained from testing Inconel in these two conditions are not very great and, as will be shown later, less than the differences shown in creep properties.

Creep and relaxation are manifestations of the same deformation processes in metals. Although an adequate fundamental theory of creep has yet to be developed, there should exist for any given temperature an empirical law which will express the creep rate, $\frac{d\epsilon_c}{dt}$, as a function of the variables: stress, strain, and time. Both creep and relaxation measurements give only the results of the integration of such a differential law. In the past, creep test results have been used exclusively to determine these differential laws. Such tests impose the limitations of constant stress. The relaxation test has the more involved limitation of a constant total strain.

$$\epsilon_t = \epsilon_c + \epsilon_e = \text{constant} \tag{1}$$

where,

ϵ_t = total strain

ϵ_e = elastic strain

ϵ_c = plastic strain of creep.

Therefore, the determination of the differential law for creep rate by relaxation testing is extremely difficult. However, in many cases, a particular differential law is either known to fit the material or for ease of calculation a simple expression is used. Therefore, there are two reasons for attempting to correlate the processes of creep and relaxation: (1) to check the accuracy of the differential laws assumed for creep, and (2) to demonstrate the use of the relaxation test on a short-time determination of creep strength.

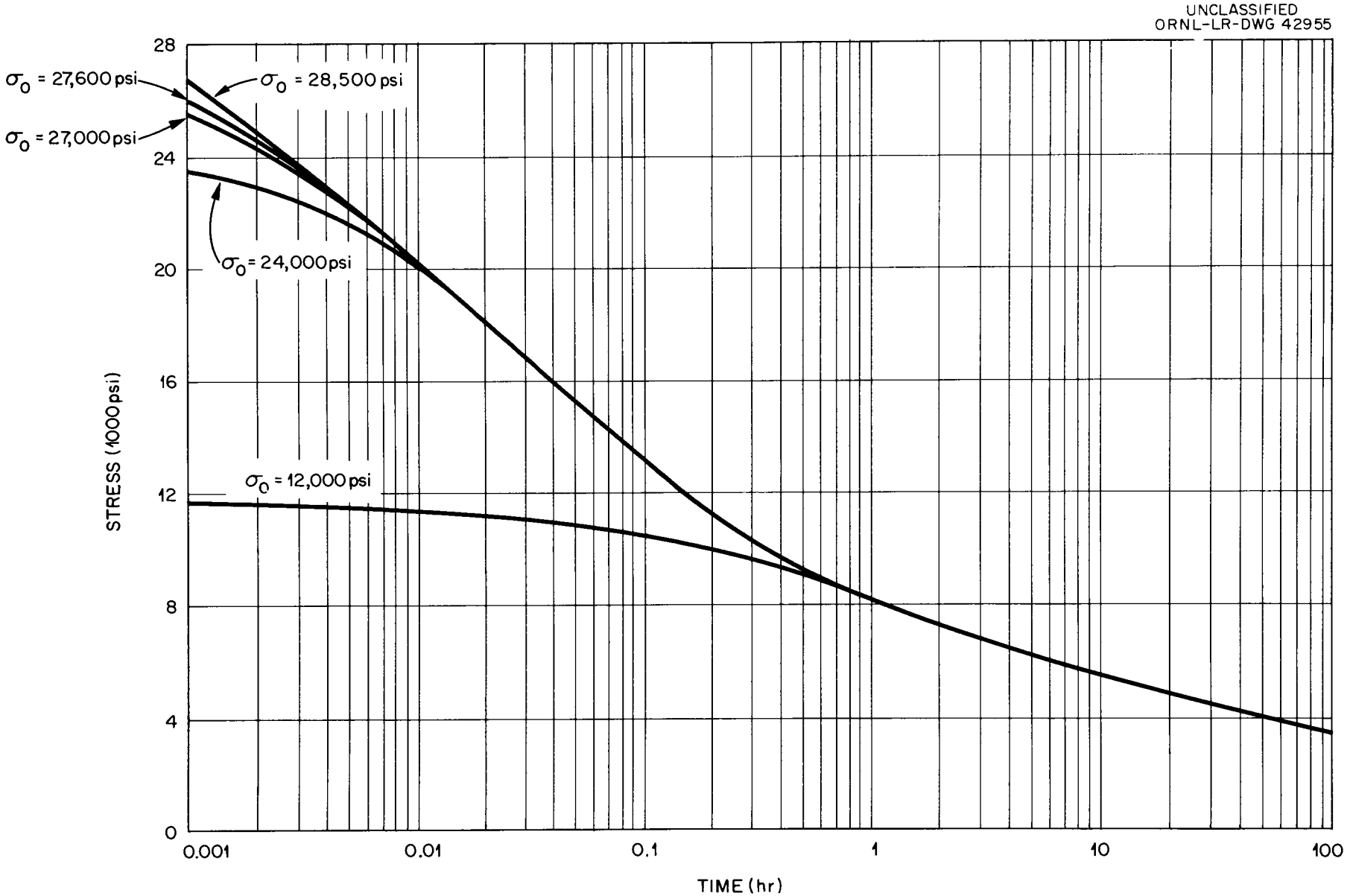

Fig. 7. Relaxation Characteristics of As-Received Inconel at 1300° F.

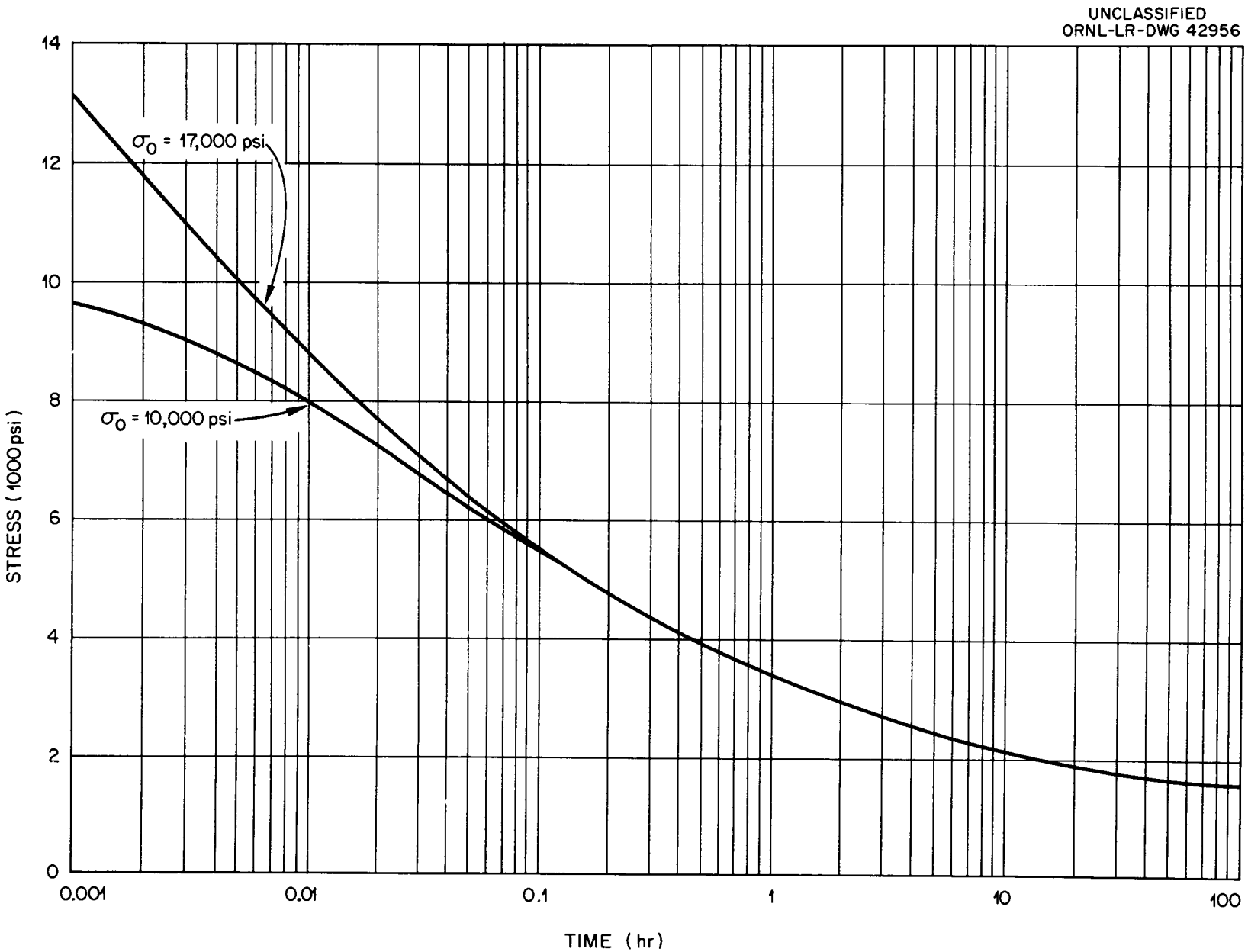

Fig. 8. Relaxation Characteristics of As-Received Inconel at 1500°F.

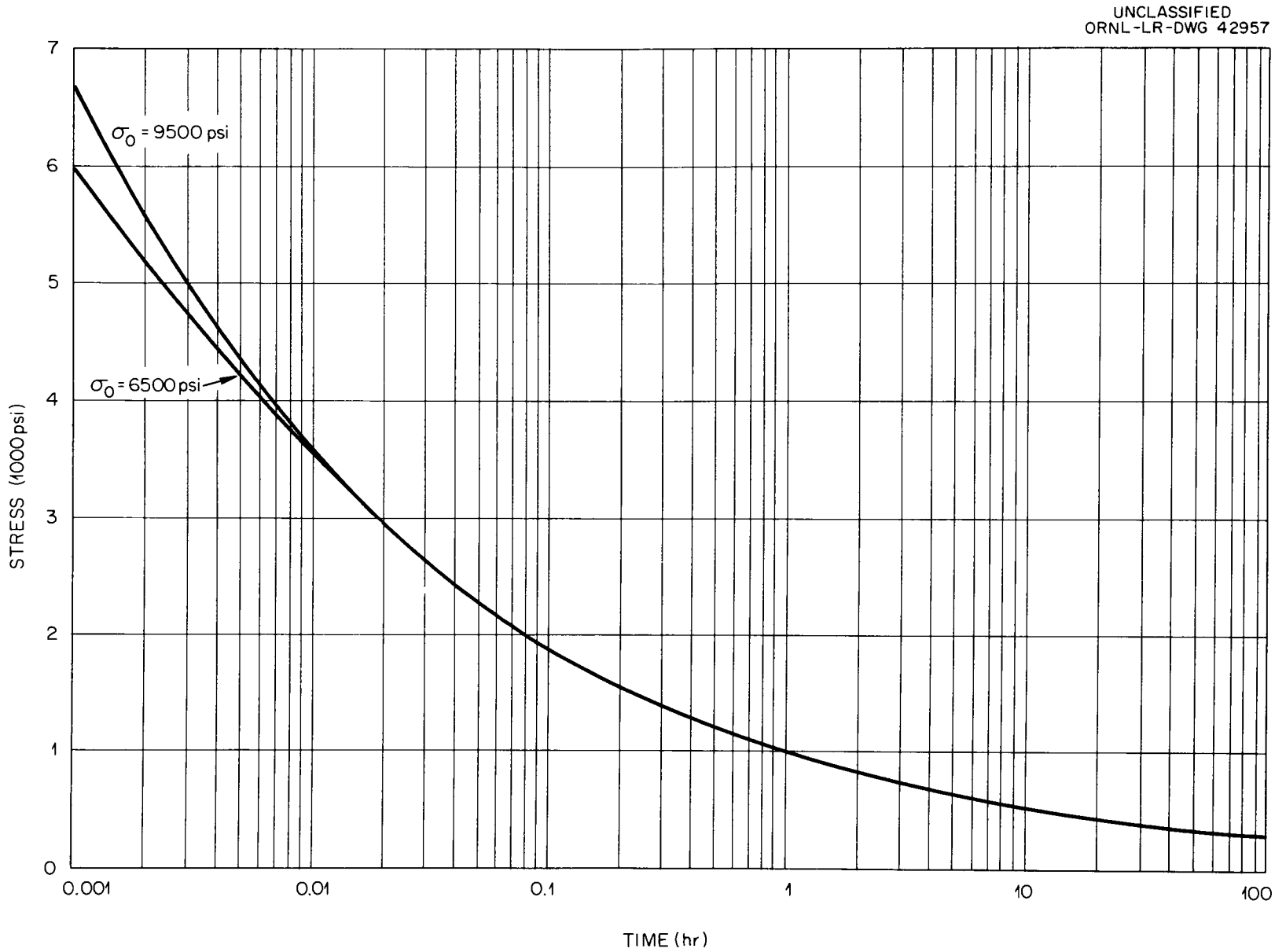

Fig. 9. Relaxation Characteristics of As-Received Inconel at 1650°F.

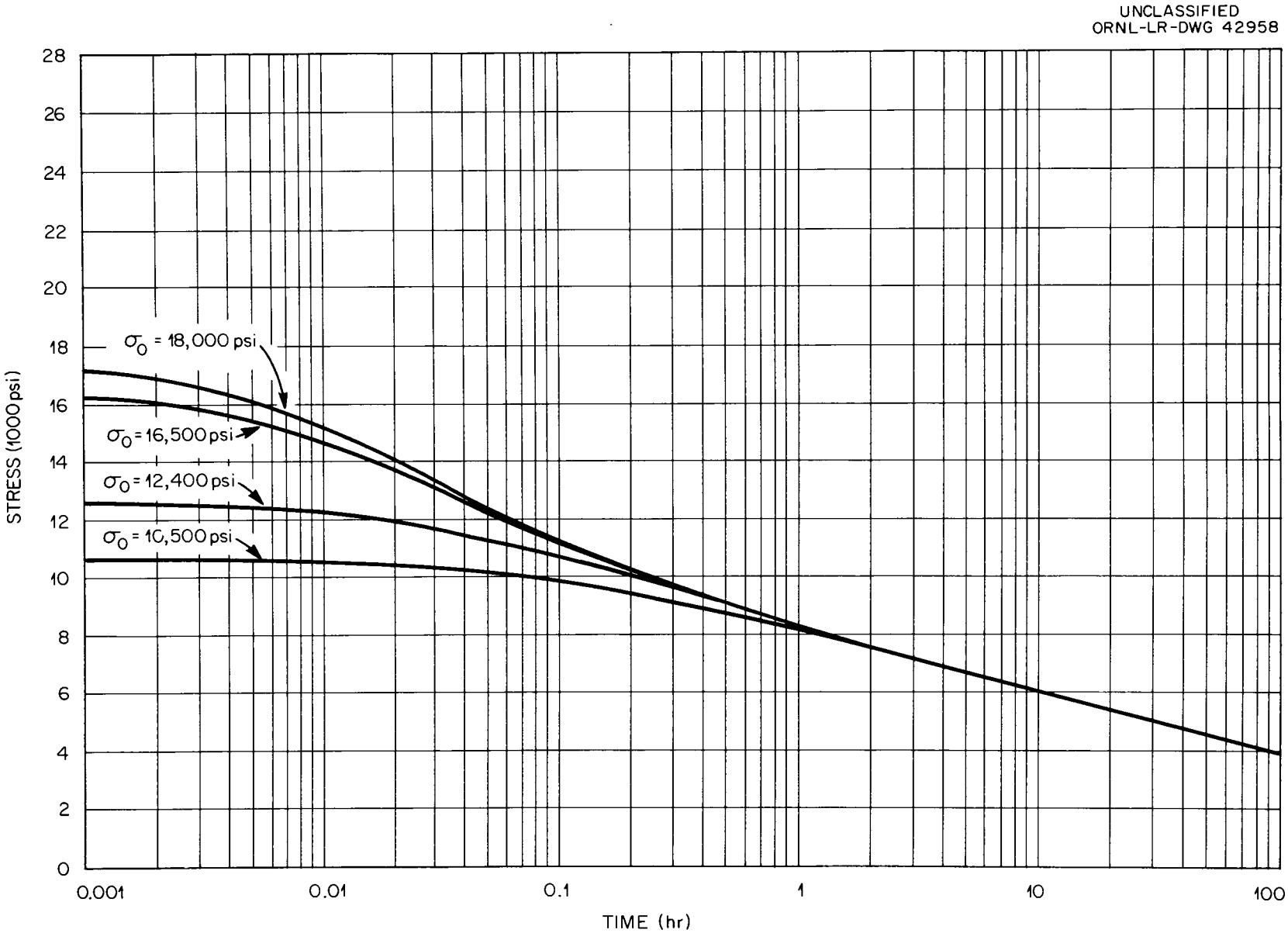

Fig. 10. Relaxation Characteristics of Inconel Annealed at 2050°F for 2 hrs. Tested at 1300°F.

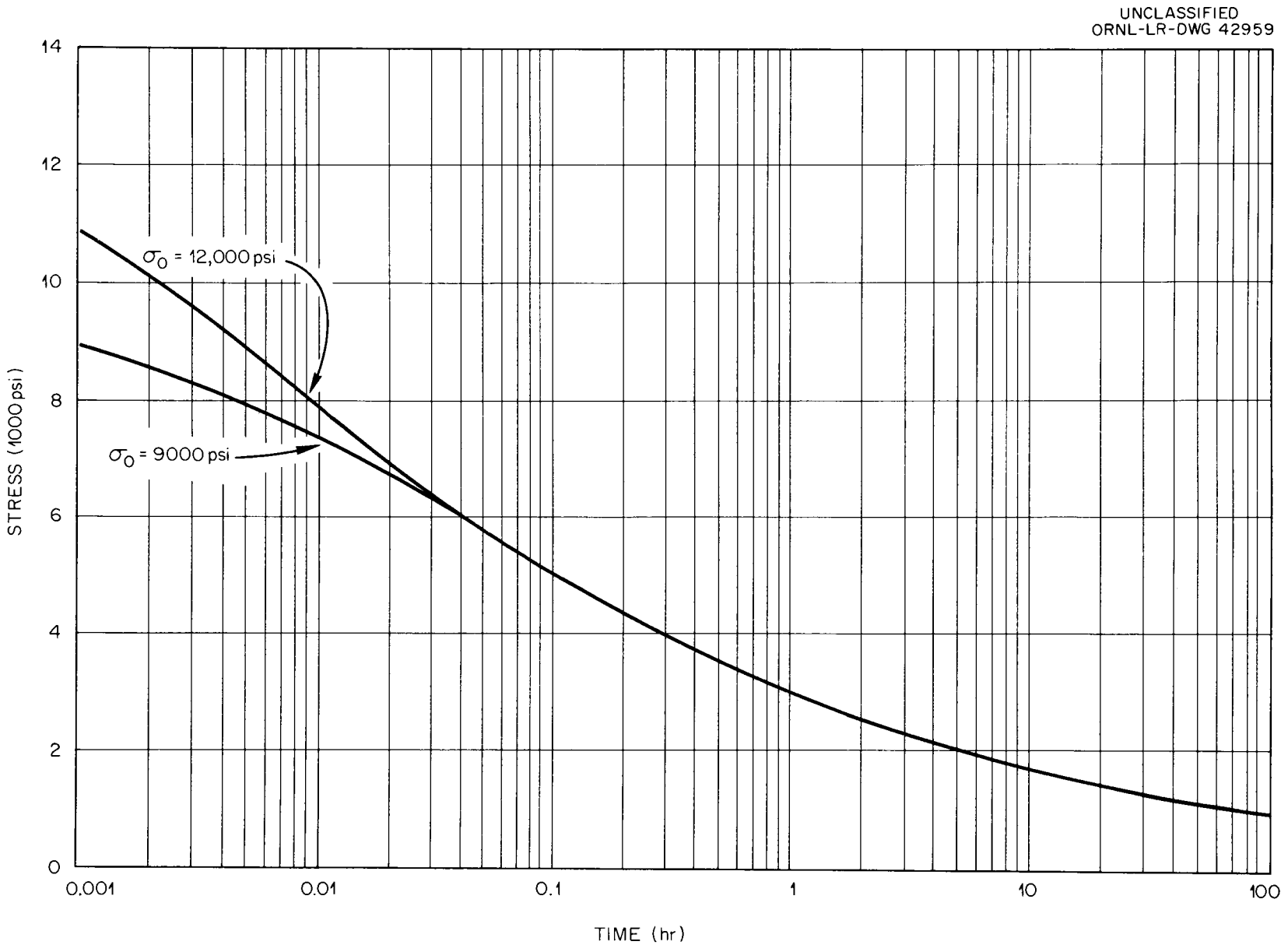

Fig. 11. Relaxation Characteristics of Inconel Annealed at 2050°F for 2 hrs. Tested at 1500°F.

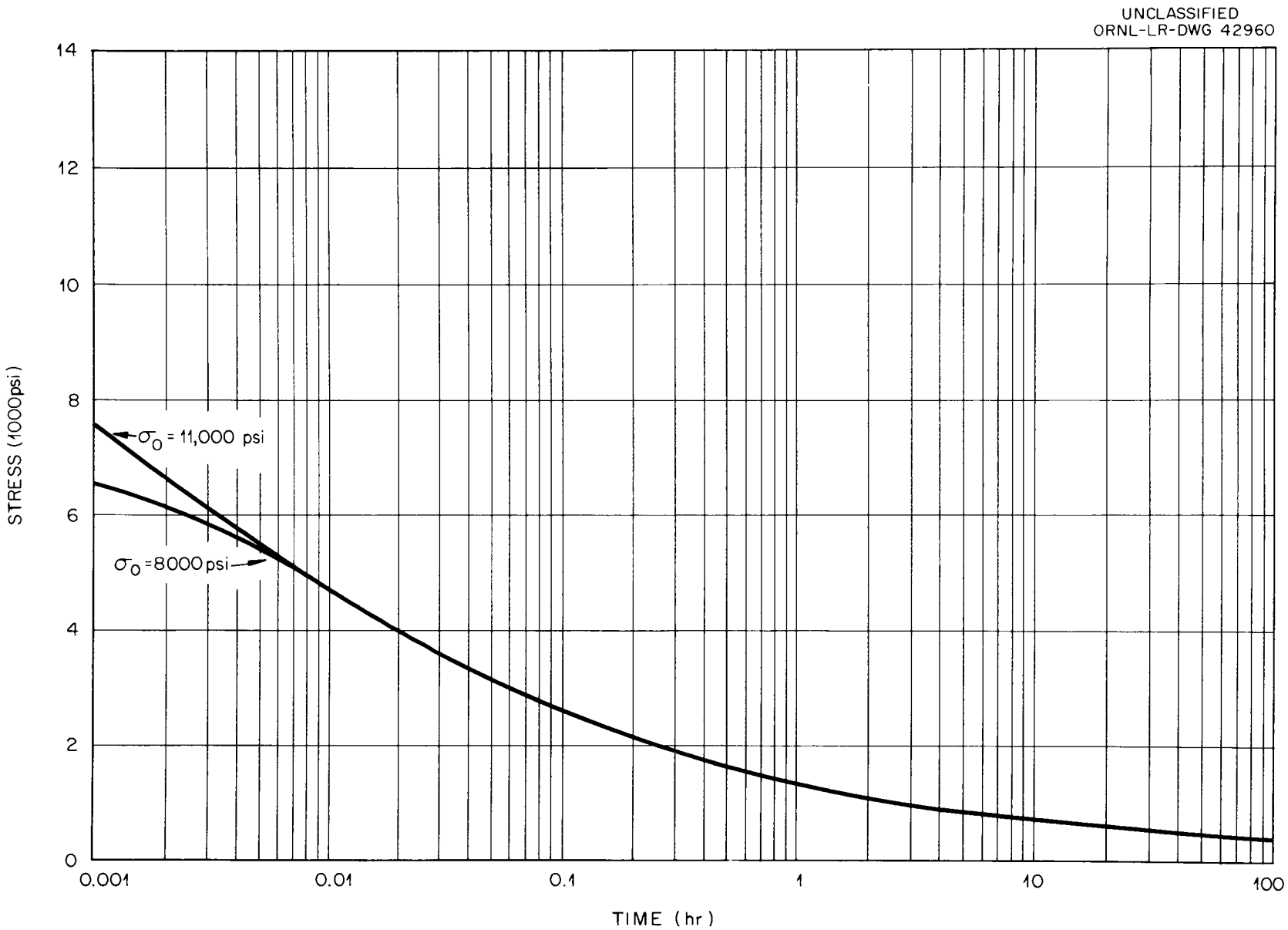

Fig. 12. Relaxation Characteristics of Inconel Annealed at 2050°F for 2 hrs. Tested at 1650°F.

The general methods for correlating the creep and relaxation data have been summarized by Roberts,[6] Soderberg,[7] Popov,[8] and Fennie,[9] and in all cases excellent agreements are found if the proper differential law is used. The one major distinction in the various expressions used for creep rate are those which include the effect of strain hardening or primary stage creep and those which ignore the primary stage of creep. One such frequently used equation which ignores the primary stage of creep is

$$\epsilon_c = \left(\frac{\sigma}{A}\right)^n t \tag{2}$$

where,

σ = applied stress

t = time

A, n = material constants

This equation, of course, does not accurately describe the creep strength of materials except under conditions where primary creep is negligible or non-existent. For many metals as the temperature increases the degree of primary creep diminishes; therefore, Eq. 2 is assumed to be accurate for high temperature. Temperatures of 1300°F and above for Inconel are sufficiently high that primary creep can be neglected; therefore, in this paper Eq. 2 can be assumed to be correct.

The general method of correlating the two processes of creep and relaxation using Eq. 2, originally proposed by Kanter,[2] demonstrates the simplicity of using this type of equation. Since, in Eq. 1 the total strain is constant, then by differentiated with respect to time, $\frac{d\epsilon_t}{dt} = 0$,

and

$$\frac{d\epsilon_c}{dt} = -\frac{d\epsilon_e}{dt} . \tag{3}$$

This states that the plastic strain rate is equal to the elastic strain rate

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[6] I. Roberts, "Prediction of Relaxation of Metals from Creep Data," Proceedings ASTM 51, 811–825 (1951).

[7] C. R. Soderberg, "The Interpretation of Creep Tests for Machine Design," Trans. ASME 58, 733–743 (1936).

[8] E. P. Popov, "Correlation of Tension Creep Tests with Relaxation Tests," J. Appl. Mechanics, Trans. ASME 69, A-135 (1947).

[9] I. Fennie and W. H. Heller, Creep of Engineering Materials, McGraw-Hill, New York (1959).

during the relaxation test. The elastic strain at any time is directly proportional to the strain by the modulus of elasticity, E.

$$\epsilon_e = \frac{\sigma}{E} \tag{4}$$

Differentiating with respect to time

$$\frac{d\epsilon_e}{dt} = \frac{1}{E}\frac{d\sigma}{dt} \tag{5}$$

Differentiating Eq. 2 with respect to time

$$\frac{d\epsilon_e}{dt} = \left(\frac{\sigma}{A}\right)^n \tag{6}$$

Combining Eqs. 3, 5, and 6

$$\left(\frac{\sigma}{A}\right)^n = -\frac{1}{E}\frac{d\sigma}{dt} \tag{7}$$

or

$$dt = -\left(\frac{A}{\sigma}\right)^n \frac{1}{E}\, d\sigma \tag{8}$$

Equation 7 is now integrated between limits of σ_o and σ, where the σ_o is the initial stress at t = 0, and σ is the stress at time, t.

$$\int_o^t dt = -\frac{A^n}{E}\int_{\sigma_o}^{\sigma}\frac{d\sigma}{\sigma^n} \tag{9}$$

or

$$t = \frac{A^n}{E(n-1)}\left(\sigma^{(1-n)} - \sigma_o^{(1-n)}\right) \tag{10}$$

Equation 9 yields the time for an initial stress, σ_o, to relax to any given stress if the creep constants A and n are known. It is equally as easy to obtain the creep constants A and n, if the relaxation data are given. This is done by noting that when σ is small compared to σ_o, the value of $\sigma_o^{(1-n)}$ becomes insignificant providing $n > 2$ as is usually the case. Therefore, Eq. 8 may be written

$$t = K\,\sigma^{(1-n)} \tag{11}$$

where,

$$K = \frac{A^n}{E(n-1)}\,. \tag{12}$$

Using Eqs. 11 and 12, the values of A and n can readily be obtained from the relaxation results. The data plotted on logarithmic coordinates should yield a straight line with a slope of (1 - n) and a zero intercept of K.

Shown in Figs. 13 and 14 are logarithmic plots demonstrating the validity of Eq. 14. The deviation of the straight line for very short values of time are areas where $\sigma_o^{(1 - n)}$ is significant and alters the straight line. The values of A and n obtained from the relaxation results are given in Table I. The strain rate versus stress curves thus established by the relaxation data are compared in Figs. 15 and 16 to constant-stress creep data obtained by testing fine- and coarse-grained Inconel 0.060-in. sheet, Heat NX 8004. These data, obtained from testing NX 8004 in argon, have been reported previously, including the testing procedure.[10] The agreement obtained is outstanding, realizing that the comparison is made for two heats of Inconel with different specimen geometries. The one exception is the comparison of properties of the fine-grained Inconel at 1650°F.

The lack of agreement between the data at 1650°F demonstrates that Eq. 2 is not the correct differential law for this material and temperature. Examination of the creep curves of fine-grained NX 8004 at 1650°F, shown in Fig. 17, demonstrate that the creep rates are not linear, but are accelerating with strain or time. When the slope of the creep curve is greater than one, the creep rate is accelerating, and when it is lower than one, the creep rate is decelerating. Generally, this type of behavior indicates a condition of metallurgical instability. One fairly common type of structure instability develops when a metal is tested at temperatures higher than the temperature at which it was annealed. In this particular case the fine-grained Inconel was given what is called an equivalent 1650°F anneal. This treatment is given to the material by the vendor and consists of a short-time exposure at 1850°F. Apparently this treatment does not produce a structure which remains stable under stress at 1650°F. This instability can be corrected by a higher

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[10] J. R. Weir, D. A. Douglas, and W. D. Manly, Inconel as a Structural Material for High-Temperature Fused-Salt Reactor, ORNL-2264 (June 4, 1957).

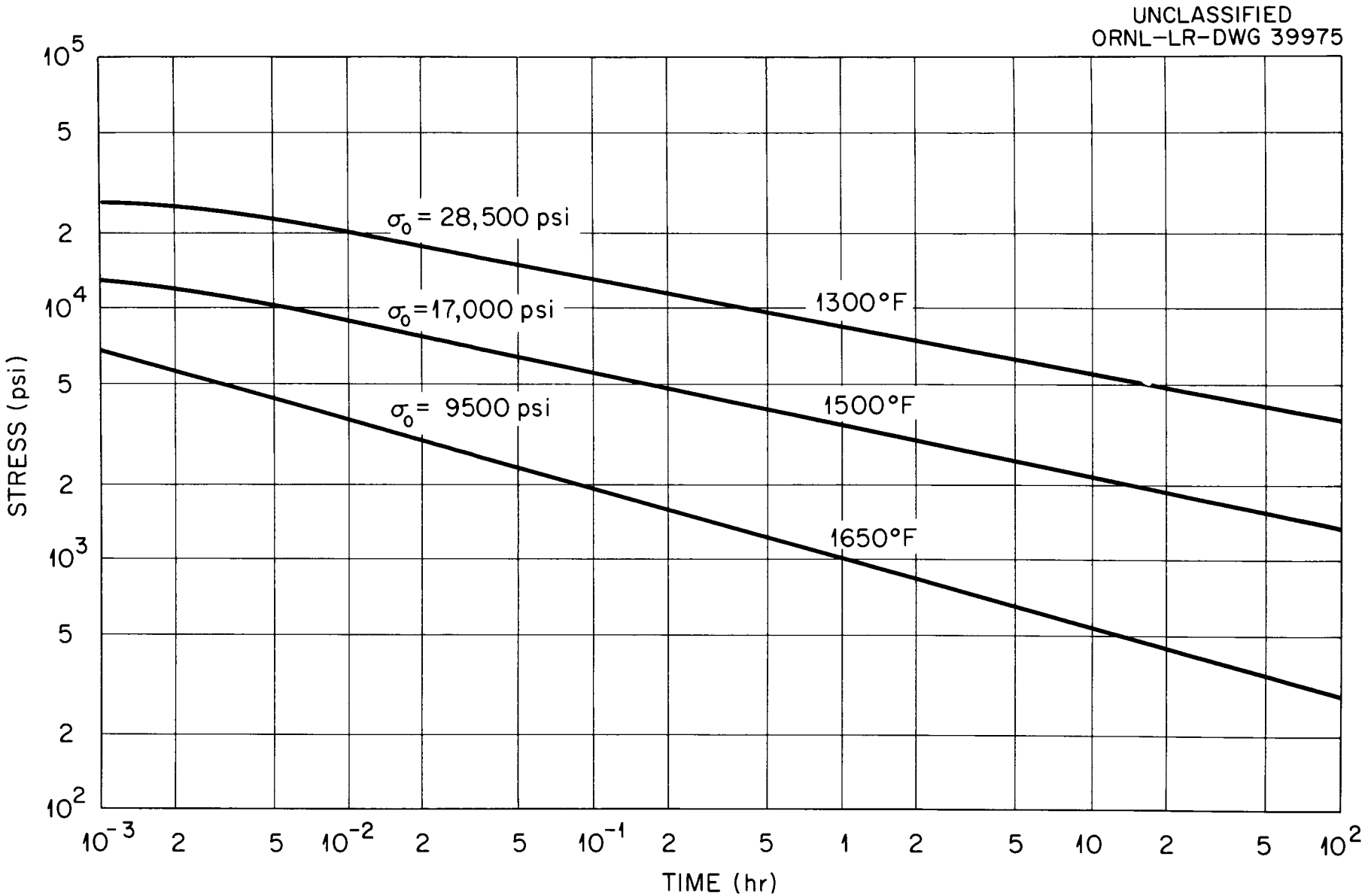

Fig. 13 Stress-Relaxation Characteristics of as-Received Inconel.

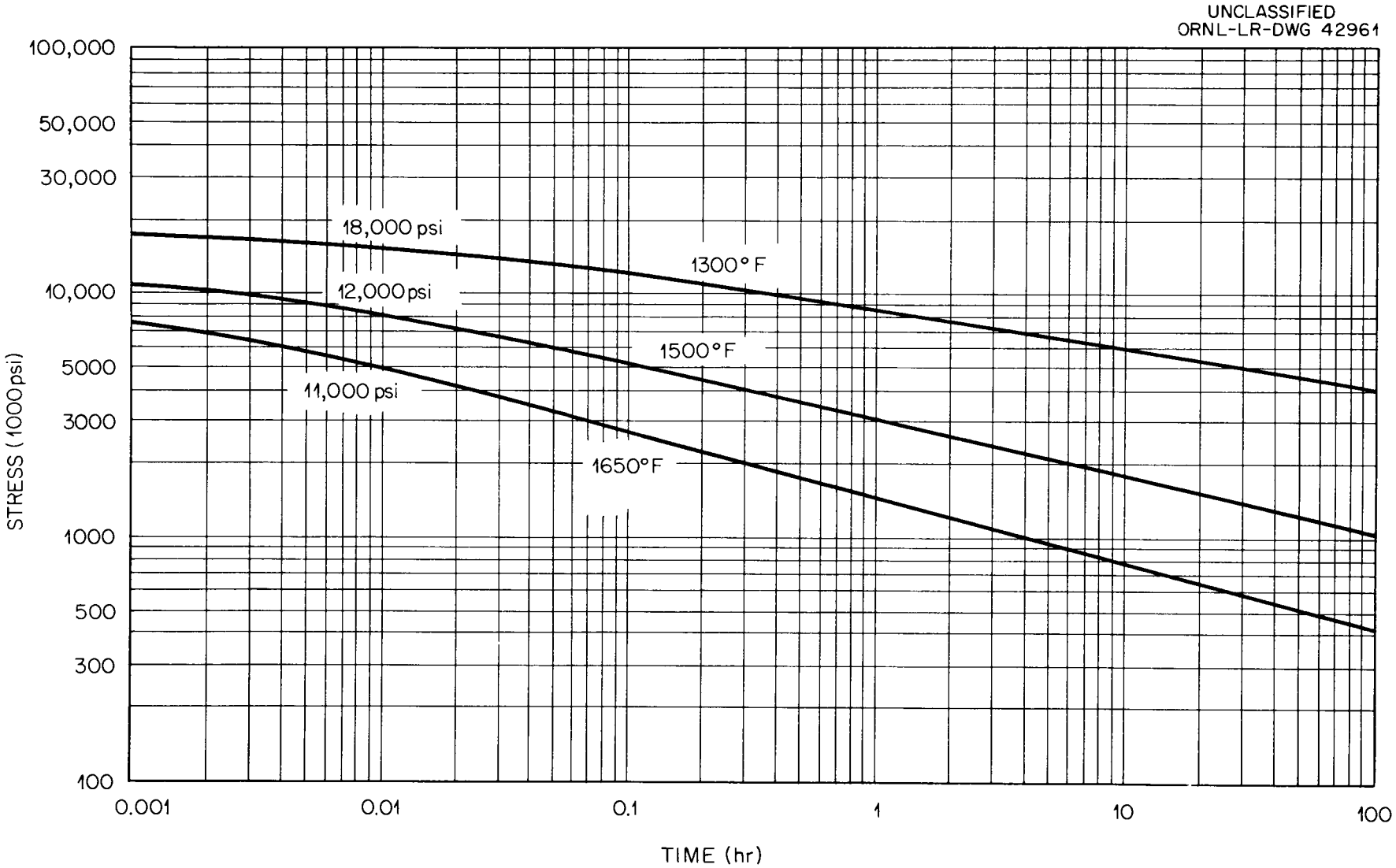

Fig. 14. Stress-Relaxation Characteristics of Inconel Annealed at 2050° F for 2 hrs.

TABLE I

VALUES OF CREEP CONSTANTS DETERMINED BY CONSTANT-STRESS CREEP TESTING AND BY RELAXATION TESTING

Test Type	Heat Treatment	Test Temperature (°F)	Creep Constants A	n
Creep	*	1300	43,000	6.18
Relaxation	*	1300	38,700	6.31
Creep	*	1500	20,500	5.95
Relaxation	*	1500	20,100	5.81
Creep	*	1650	16,800	4.70
Relaxation	*	1650	10,800	4.61
Creep	**	1300	31,600	6.85
Relaxation	**	1300	30,700	7.37
Creep	**	1500	17,900	5.50
Relaxation	**	1500	19,600	5.42
Creep	**	1650	10,500	5.20
Relaxation	**	1650	13,100	4.79

*As received.

**Annealed at 2050°F for 2 hr.

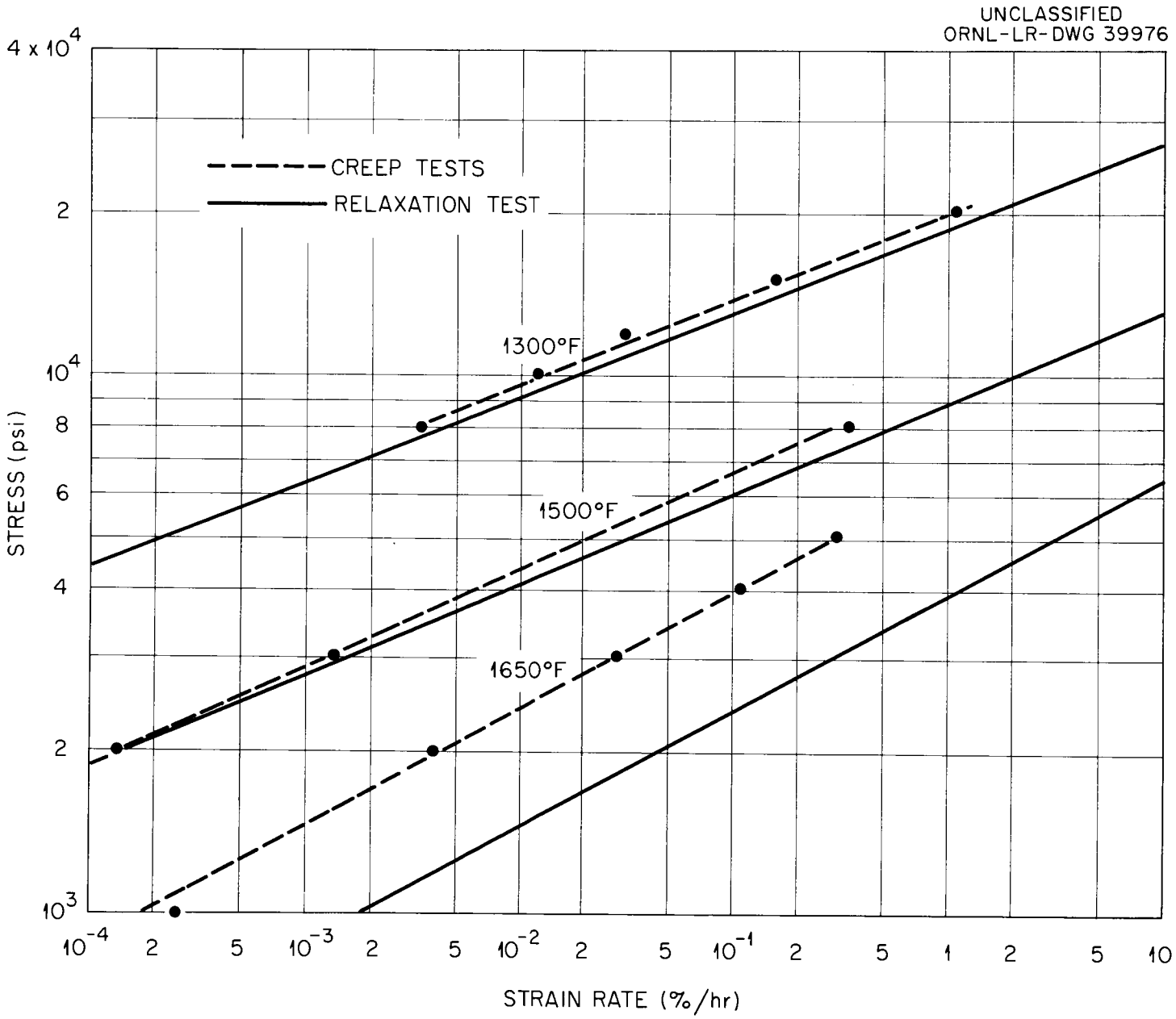

Fig. 15 Stress vs Strain-Rate for as-Received Inconel

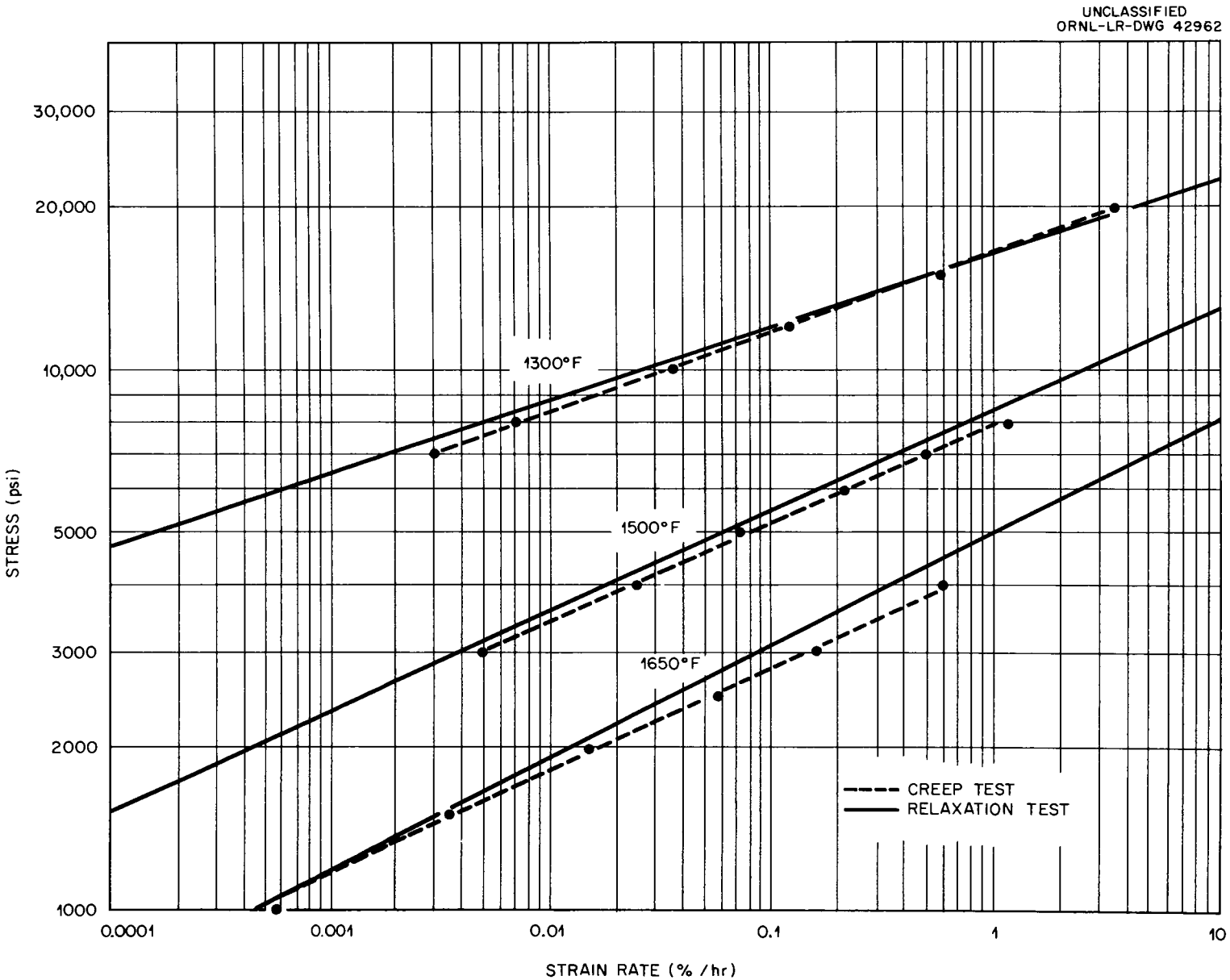

Fig. 16. Stress vs Strain-Rate for Inconel Annealed at 2050°F for 2 hrs.

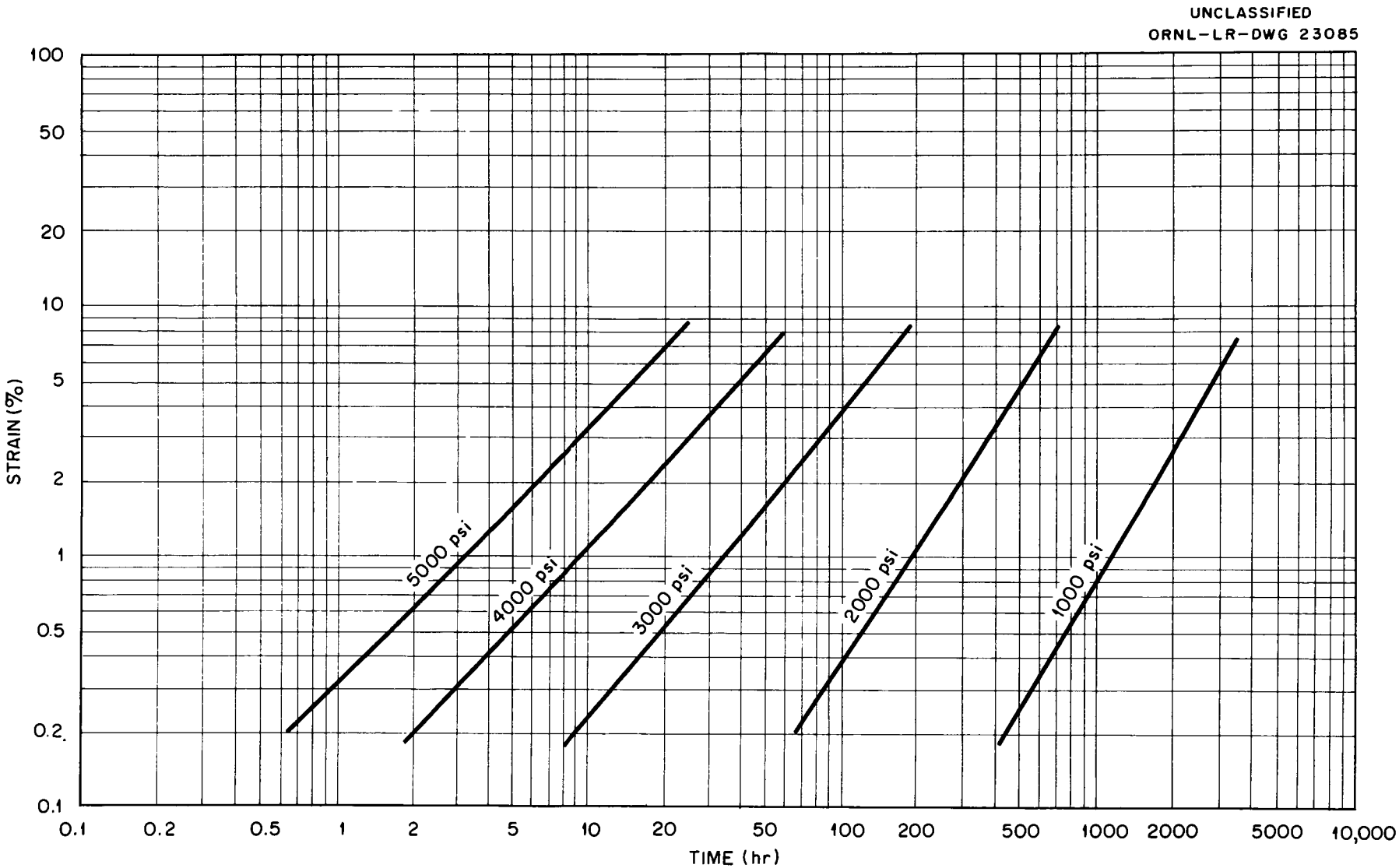

Fig. 17 Creep Curves of Inconel Sheet (NX8004, as rec) Tested at 1650°F in Argon.

temperature anneal as shown by the results of testing Inconel annealed at 2050°F for 2 hr. When this is done, the 1650°F results demonstrate a very close agreement using Eq. 2 as shown in Fig. 16.

Although this equation or others which ignore the primary stage of creep are accurate only for specific conditions, the simplicity of the expressions warrants its use in many complex design problems where the use of the exacting solutions are sometimes unmanageable. Also, in view of the many indeterminate factors influencing creep, these solutions seem adequate. It should be remembered that the values of the creep constants, A and n, determined by constant-stress creep tests always tend to produce optimistic values of the creep strength. This has been demonstrated by the test results above for materials tested with an unstable structure. Less conservative values will be obtained by using the second-stage creep rate values where materials exhibit large strain in the primary stages. It would appear that the values of the creep constants obtained by relaxation testing, which include the effect of primary creep, may be more indicative of the creep strength of the material.

ACKNOWLEDGMENT

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