Engineering Research Institute
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THIRD PROGRESS REPORT

TO

MATERIALS LABORATORY

WRIGHT AIR DEVELOPMENT CENTER

ON

NOTCH SENSITIVITY OF HEAT RESISTANT ALLOYS AT ELEVATED TEMPERATURES

By

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SUMMARY

Creep and Relaxation Properties

Results previously reported for this joint investigation under Contract No. AF 18(600)-62, Expenditure No. R-605-227 SR-3a, indicated much higher relaxation rates with S-816 at 1350°F for stresses above the proportional limit than for low initial stresses. Further tests have now been conducted with momentary overloading at test temperature to give a plastic strain of about two per cent. The stress was then quickly reduced and a relaxation test started at the resulting lower stress.

Prior plastic straining was found to increase the rate of relaxation of S-816 for all initial stresses tested.

Other tests are planned to investigate stress-relaxation properties of Waspaloy with conventional solution and aging treatment.

Metallurgical Variables Affecting Notch Properties

As the first part of a study of metallurgical variables, Waspaloy specimens were subjected to cold rolling before conventional heat treatment. The resulting grain size varied from coarse (-1 to 0, some finer) for one per cent prior reduction to relatively fine (4 to 5) for ten per cent reduction. When tested at 1500°F, specimens with an A.S.T.M. grain-size range of 1 to 3 (1.5 per cent reduction of original cross section before solution treatment) showed an increase of approximately 2,000 psi in rupture strength above that for the fine-grained material. The alloy in the coarse condition also showed a slight decrease in elongation at rupture. Further work is being carried out with Waspaloy in an attempt to make the alloy more brittle than did the above treatments.

Preliminary tests are also in progress in search of treatments which may tend to embrittle notched bars of S-816 at a temperature of 1350°F.

Work with Inconel X-550 will be started when this material is received.

INTRODUCTION

This report covers work for the quarter between December 15, 1952, and March 15, 1953, under Contract No. AF 18(600)-62, Expenditure Order No. R-605-227 SR-3a. The purpose of the overall program is to examine stress - rupture time properties at elevated temperatures of notched bars and to investigate factors affecting notch sensitivity of heat-resistant alloys. It was planned that this laboratory should concentrate on the significance of creep and relaxation on notched-bar rupture tests and on effects of certain metallurgical variables on such tests.

Past progress reports have presented compilations of pertinent data available from the literature and from industrial sources. It has been reported previously for S-816 alloy at 1350°F that for tests begun at stresses above the proportional limit, the curves of stress against time cross over those for lower stresses, so as to give residual stresses, after several hours, which are below that which would remain after the same relaxation time for a specimen started at a stress below the proportional limit.

In multiple-stress tests for S-816 at 1350°F, it was found that the portion of life expended during a given time period at a given stress is very nearly equal to the fraction: $\frac{\text{actual time at the stress}}{\text{rupture life at that stress}}$.

CURRENT STATUS OF THE INVESTIGATION

Creep and Relaxation Properties

S-816

Four supplementary relaxation tests and two additional creep tests included in this report complete experimental work planned for S-816 with conventional heat treatment. As soon as results for notched bars of this alloy become available, a correlation between notched-bar rupture life and material properties will be attempted. If a satisfactory correlation can be obtained, no further experimentation on S-816 with standard heat treatment is anticipated. This phase of the program has been limited to 1350°F for S-816 alloy because it seemed best to keep the testing temperature as low as possible for this notch-ductile material.

Waspaloy

Specimens of Waspaloy with conventional heat treatment have been prepared, and the first relaxation runs are under way. This alloy will be studied at 1500°F, where it should have border-line notch sensitivity characteristics.

Inconel X-550

As of the date of this report, the Inconel X-550 specified in the original contract as the third alloy to be tested has not reached this laboratory.

Metallurgical Variables Affecting Notch Properties

Abnormal Grain Growth in Waspaloy

During discussions with personnel of the Materials Laboratory, Wright Air Development Center, it was decided to investigate notched-bar rupture properties for specimens treated to form abnormally-large grains. Preliminary tests have been made to determine conditions causing growth of such large grains in Waspaloy. Rupture tests have been performed on smooth bars of Waspaloy with two of the treatments investigated. Further work is in progress for this alloy and for S-816.

EXPERIMENTAL RESULTS

Creep and Relaxation Properties

Tests on S-816 at 1350°F

Results of additional tests to clarify the effect of prior creep and of yielding during loading operations on the relaxation rate of S-816 at 1350°F are shown in figure 1.

For a specimen loaded to 30,000 psi stress and then allowed to creep 0.0087 in./in., the relaxation pattern followed very closely that of a specimen with zero prior creep up to three or four hours, but then the stress decreased slightly more slowly than for a bar without prior creep.

Tests were performed in which specimens were strained 0.029 and 0.0265 in./in. respectively by momentary loading to 60,000 psi stress. The overload was immediately removed to give a stress of 30,000 psi, from which stress step-wise relaxation runs were made. The two runs give an average spread of only about 1,000 psi in residual stress indicated at any time during the test and show a considerably faster initial rate of relaxation than is shown for a specimen not initially strained by overloading. At times of from 30 to 80 hours, the curves for "normal" and prestrained conditions seem to cross, with the indication that the prestrained specimens will have the higher residual stress after long time periods.

A test bar similarly prestrained at 60,000 psi before relaxation from a 40,000 psi starting stress exhibits like behavior. It might be noted that the total plastic strain obtained from overloading varied from specimen to specimen, due to slightly different rates of loading and to creep during the loading process. In all cases, the strain was larger than the two per cent predicted by the short-time tensile curve presented in a previous report (1).

Figure 2 presents creep curves at 1350°F for three specimens of S-816, with one prestrained at 60,000 psi (0.0312 in./in. strain) and the others not. Though all three curves are for different stress levels, the plot for the prestrained bar run at 36,000 psi lies about where it would be expected in relation to the other curves and exhibits the same general characteristics as that for the specimen run at 35,000 psi with no prior strain before testing.

As a check on reproducibility between specimens processed at the two laboratories taking part in this project, a conventional stress-rupture time test at 35,000 psi stress was run at the University of Michigan laboratories, using a test bar treated and machined at the second laboratory. The rupture life obtained was 177.5 hours, compared with 180.6 hours reported previously for a test on a specimen made at the University. Agreement appears satisfactory.

Metallurgical Variables Affecting Notch Properties

Abnormal Grain Growth in Waspaloy

The first variation in treatment investigated for Waspaloy involved an attempt to induce abnormally-large grain size by a critical amount of rolling at room temperature before solution treating and aging.

Samples of Waspaloy from heat No. 44,036 were reduced approximately 45 per cent in cross section by rolling at 1950°F, with intermediate re-heat to 1950 between reductions of about 10 to 15 per cent. After the final pass, the bars were air cooled and then given a four-hour solution treatment at 1975°F, followed again by air cooling. After the bars had been machined square, they were then rolled at room temperature to give reductions ranging from zero to ten per cent of the cross section. The cold-rolled bars finally were given the following conventional heat treatment:

1975°F, 4 hours, air cool;
1550°F, 4 hours, air cool;
1400°F, 14 hours, air cool.

Microexamination of representative sections polished and then etched electrolytically with 10 per cent oxalic acid revealed A.S.T.M. grain sizes as listed in table I. Typical microphotographs are shown in figure 3.

After examination of these results had indicated that 1.5 and 10 per cent cold reduction should given rather wide differences in grain size, test specimens (0.250 in. gauge diamter; 1 inch gauge length) were prepared for these reductions and were tested to rupture at 1500°F. Table II lists the data obtained; the results are also plotted in figure 4. In the stress range between 20,000 and 30,000 psi, the material with 1.5 per cent cold reduction prior to conventional heat treatment is found to have somewhat lower elongation to rupture and a significantly longer rupture life than does material with 10 per cent cold working before heat treatment.

Treatments applied to date gave grains considerable smaller than were sought. It was desired to produce a specimen with grains predominantly larger than A.S.T.M. size zero.

DISCUSSION

Creep and Relaxation Properties of S-816

The tests carried out, figure 1, showed that relaxation occurred faster for a given initial stress if the samples were prestrained by overloading. The previous progress report showed the same behavior if the initial stress exceeded the proportional limit. Thus, in all cases to date where a specimen has been loaded initially past the proportional limit, the rate of relaxation of stress has increased during the early stages of the relaxation tests. At longer time periods (after 50 to 500 hours, depending on the stress level), the rate of relaxation after prior strain decreases and the residual stress eventually becomes equal to or higher than for tests started below the proportional limit.

For high starting stresses, a period of creep immediately after loading and before the start of the relaxation run has little measurable effect. (A forty-fold range of creep strain gave rather similar relaxation curves from 40,000 psi starting stress.) At high stresses, creep occurs so rapidly that any initial falling-rate period is difficult to detect. Thus, for 45,000 psi stress, the creep rate shown in figure 14 of the previous progress report (1) appears to be constant between zero and ten per cent creep strain. For this entire range of prior creep, the initial relaxation rate should, therefore, be the same.

For low initial stresses, on the other hand, a measurable decrease in creep rate (and, therefore, in the related relaxation rate) occurs between zero creep and a finite creep of, say, one to three per cent prior to the start of relaxation measurements.

Such an explanation for the decrease in relaxation rate after large prior creep strains is supported by the behavior reported in figure 1 for specimen PR-18 with a smaller amount of initial creep. At first, the plot for this specimen parallels the relaxation curve for no creep previous to relaxation, but after a few hours when the steady-state creep period is finally reached for the stress level present, the residual stresses fall less rapidly than do those for a specimen relaxed immediately after loading to the starting stress of 30,000 psi.

Such a simple accounting for relaxation behavior may not be complete, but the important point is not the explanation for, but rather the significance of, these findings. In a sharply-notched bar, the metal at the notch root is strained plastically on loading, even when the nominal stress is somewhat below the proportional limit. With S-816 at 1350°F, this local plastic strain actually promotes the tendency for concentrated high stresses to level out by rapid relaxation. Moreover, with a high ductility to rupture for all stresses, the alloy should be capable of extreme local plastic strains during loading and still be able to accomodate the creep strains accompanying relaxation processes without failure of fibers in the critical region near the notch root.

A very low ductility to rupture, on the other hand, should seriously affect life of notched bars. When the sum of loading strain and creep strain reaches the limiting value, failure of critical fibers should begin. This, in turn, raises the stress on other fibers, promoting general failure.

A material with high elongation in the rupture test and for which prestrain favors rapid stress relaxation at the stress and temperature of test should not be severely affected by stress concentrations.

Metallurgical Variables

Abnormal Grain Growth in Waspaloy

Results reported above show that large grains can be induced in Waspaloy with a critical amount of cold working before the solution treatment and aging normally provided. Excessive growth of grains has been reported to be associated with low ductility, which, in turn, has been shown to occur in many instances of notch sensitivity at elevated temperatures.

Small reductions by cold rolling are difficult to control exactly and may give a variable amount of work in different parts of the bar. However, for specimens given like reductions, the grain size variation found from specimen to specimen appears to be acceptable.

When the rupture test data of table II are compared for 1.5 and ten per cent cold reduction before final solution treatment, the coarser material shows a definite increase in rupture life and a drop in elongation during the test, as would be expected. Elongation values at rupture of 3.5 to seven per cent are probably still too high to indicate notch sensitivity. Further work is planned to seek a means of obtaining coarser grains.

In preliminary tests, a bar reduced one per cent in cross section showed the largest grain, but a small portion in the center had fine grains with a size typical of that for bars with no cold working after preparatory homogenizing by hot reduction of about 45 per cent. The occurance in immediate juxtaposition of the coarsest and finest grains indicates that the very center of the bar may have received less than the critical amount of cold working. It is hoped that other bars now in process with 1-1/4 per cent cold work before heat treating may receive just enough more work to place the center in the critical range for grains of the size looked for.

FUTURE WORK

Experimental work for the coming quarter is to concentrate on creep and relaxation properties at 1500°F of Waspaloy with normal heat treatment. The studies are to include relaxation following pre-strain and rupture tests under multiple stress levels, as was done for S-816 at 1350°F.

Meanwhile, tests on Waspaloy and on S-816 with a variety of non-standard treatments are to be continued in the next three months in search of metallurgical conditions favoring notch brittleness for these two alloys.

Whenever the Inconel X-550 stock arrives, a comparable program will be initiated for that material.

BIBLIOGRAPHY

(1) Voorhees, H. R., and Freeman, J. W., "Second Progress Report to Materials Laboratory, Wright Air Development Center, on Notch Sensitivity of Heat Resistant Alloys at Elevated Temperatures," University of Michigan Research Institute Project 2024, Dec. 15, 1952.

TABLE I

EFFECT ON GRAIN SIZE OF COLD WORK PRIOR TO FINAL SOLUTION TREATMENT OF WASPALOY

Specimen No.	Cold Reduction (per cent)	A. S. T. M. Grain Size Range
W 115	1	-1 to 0, some 2 to 3 (4 to 6 in small area near center of the original bar)
W 109	2	0 to 2, some -1 and 3 to 4
W 118	2	1 to 2, some 0 and 3 to 4
W 103	4	3 to 5, some 6
W 108	6	3 to 5
W 116	10	4 to 5, some 2 to 3 and 6 to 7
W 111	0	4 to 5, some 3 and some 6

TABLE II

STRESS - RUPTURE DATA AT 1500°F FOR WASPALOY WITH 1.5 AND 10 PER CENT COLD REDUCTION PRIOR TO FINAL SOLUTION TREATMENT (Specimen gauge diameter 0.250", gauge length 1.00")

Specimen No.	Cold Reduction (per cent)	ASTM Grain Size	Stress (psi)	Life (hrs)	Elongation (per cent)	Red. Area (per cent)
W 102	10	4 to 5, (3, 6)	25,000	260.5	7.5	9.0
W 117	10	4 to 5, (6)	30,000	98.9	8.5	7.9
W 112	1.5	1 to 3, (4)	20,000	1203.3	3.5	7.0
W 114	1.5	1 to 2, (0, 3 to 5)	25,000	479.1	7.	7.4
W 113	1.5	2 to 3, (1,4)	30,000	132.6	5.	7.1

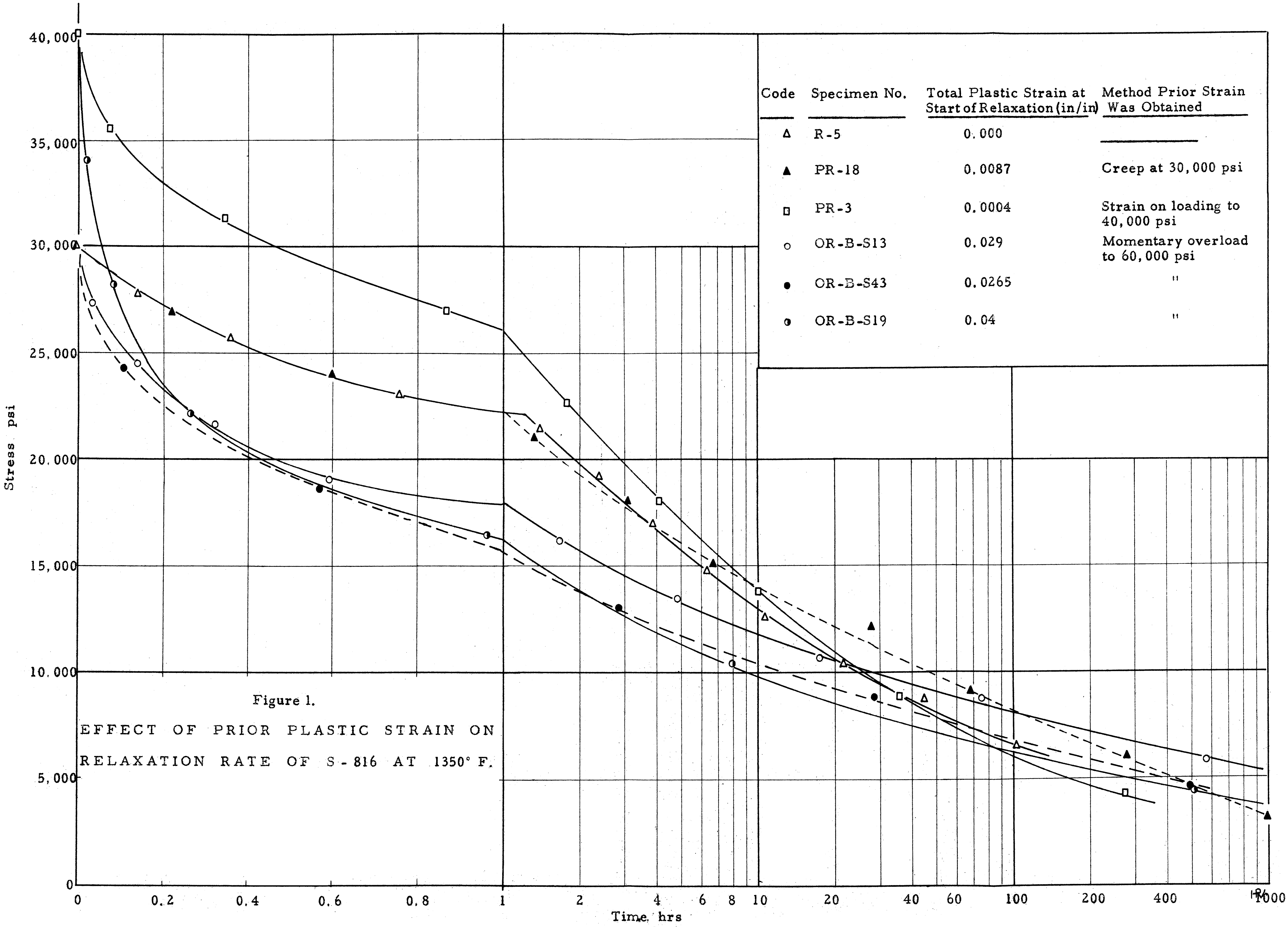


Figure 1.

EFFECT OF PRIOR PLASTIC STRAIN ON RELAXATION RATE OF S-816 AT 1350° F.

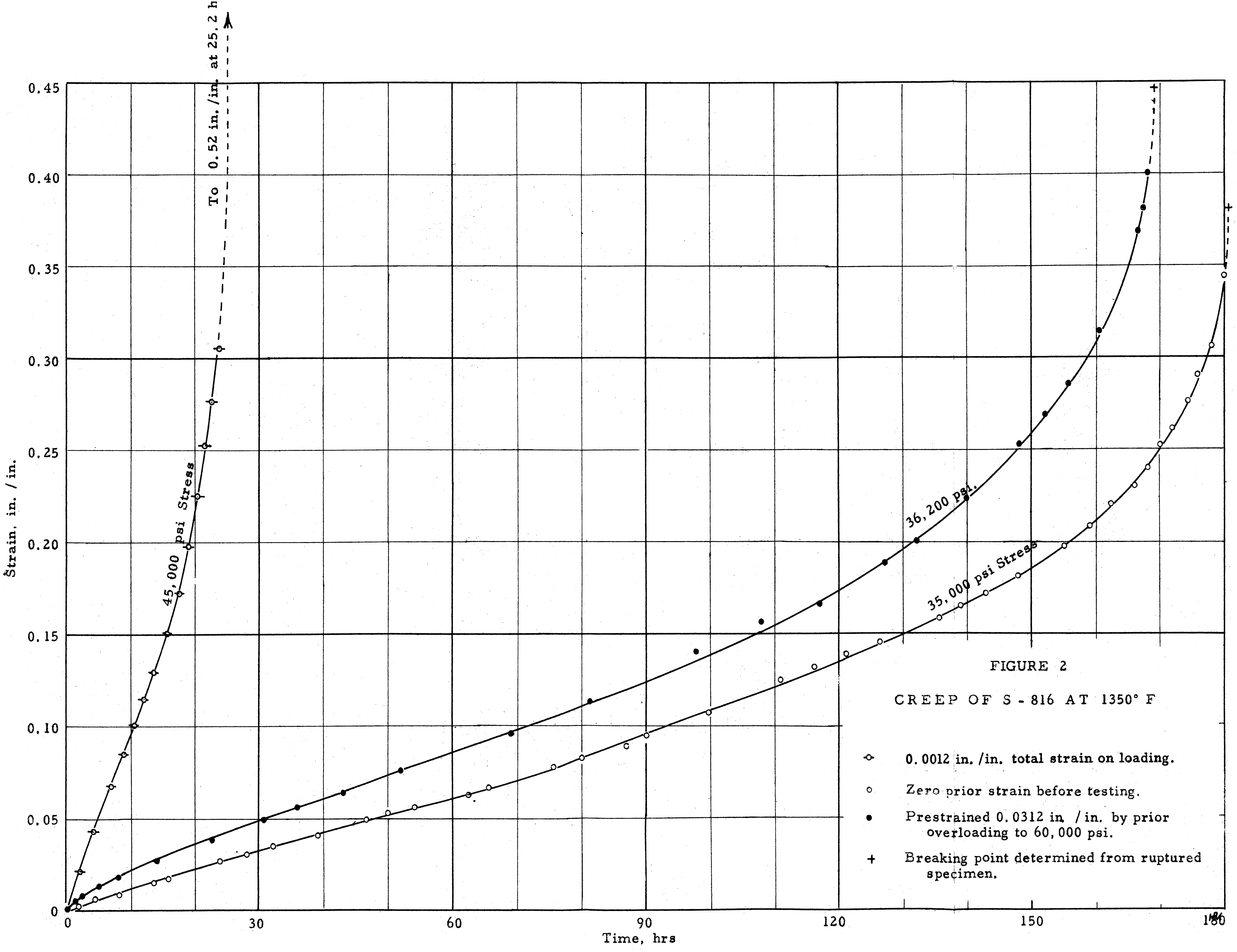
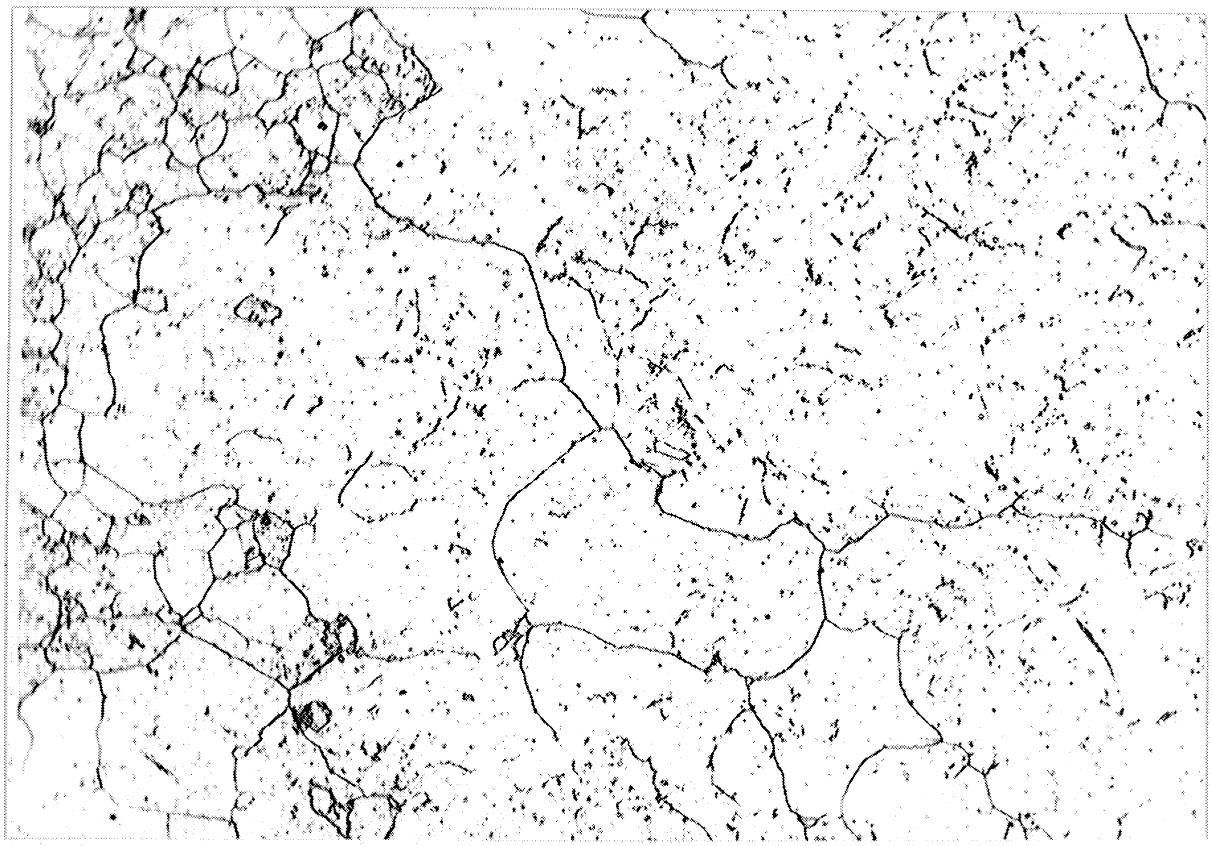


FIGURE 2

CREEP OF S-816 AT 1350° F



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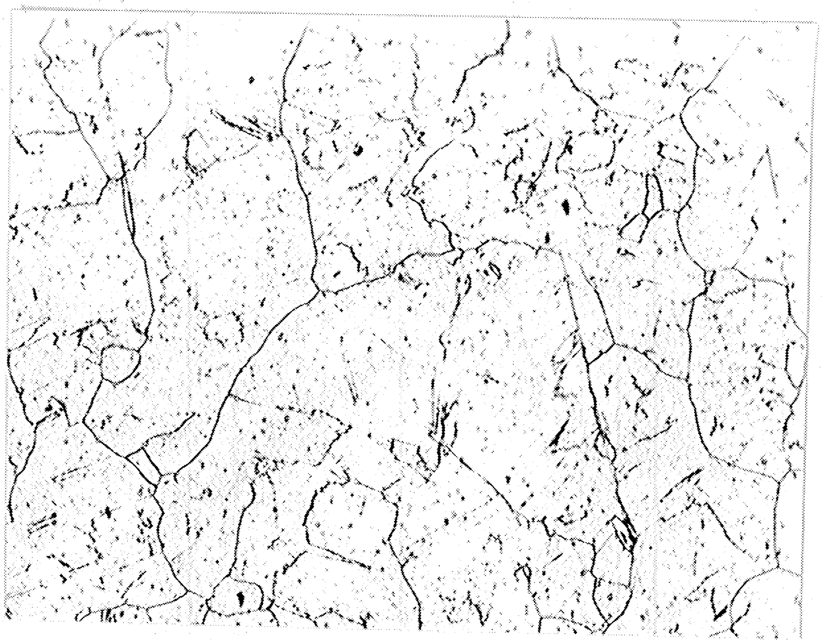
a. One per cent cold reduction before final heat treatment. The finer grain shown in the upper left was found at the center of the bar as rolled.

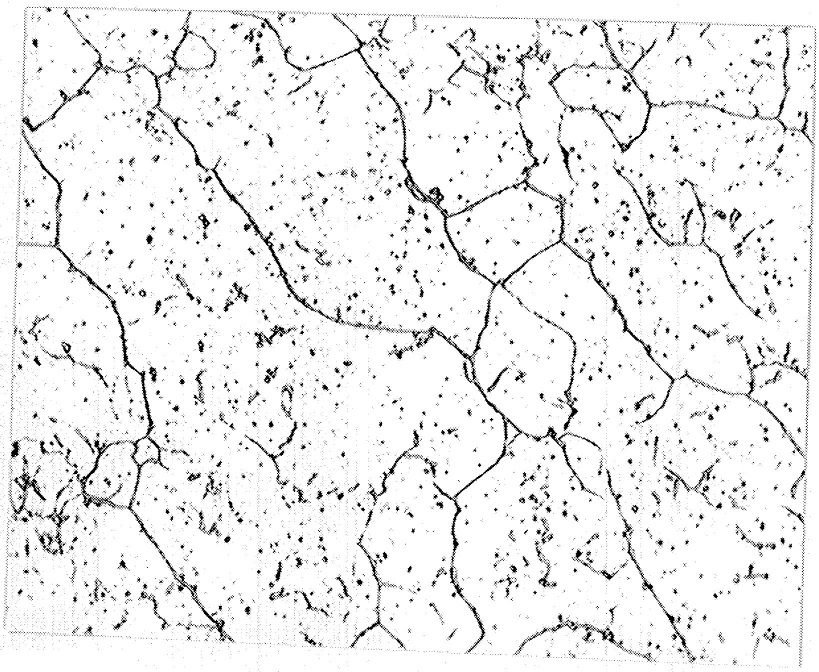
Fig. 3. - TYPICAL GRAIN SIZES OBTAINED WITH DIFFERENT AMOUNTS OF COLD REDUCTION OF WASPALOY PRIOR TO FINAL HEAT TREATMENT.

(All 100 X)



b. 1.5 per cent cold reduction before final heat treatment.

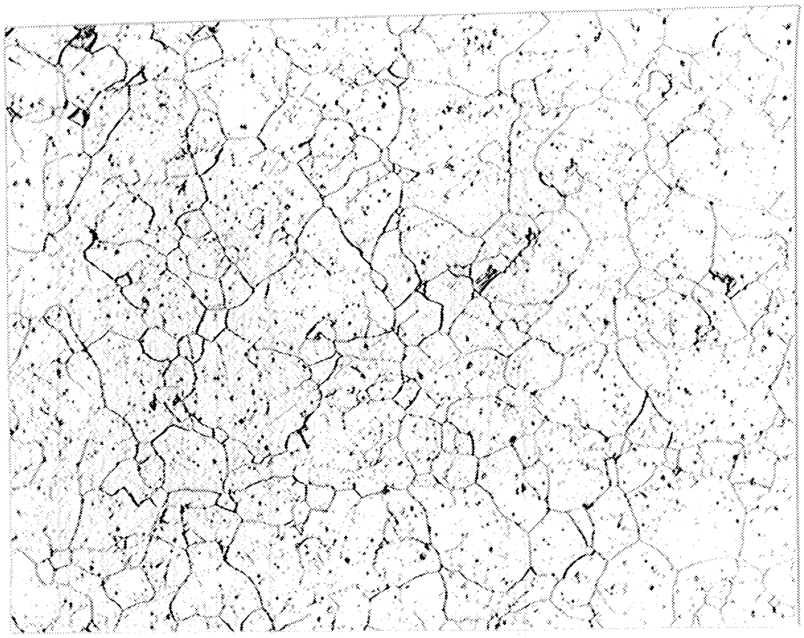
53-383



c. Two per cent cold reduction before final heat treatment.

53-385

Fig. 3. - Continued.



53-384

d. Ten per cent cold reduction before final heat treatment.

Fig. 3. - Concluded.

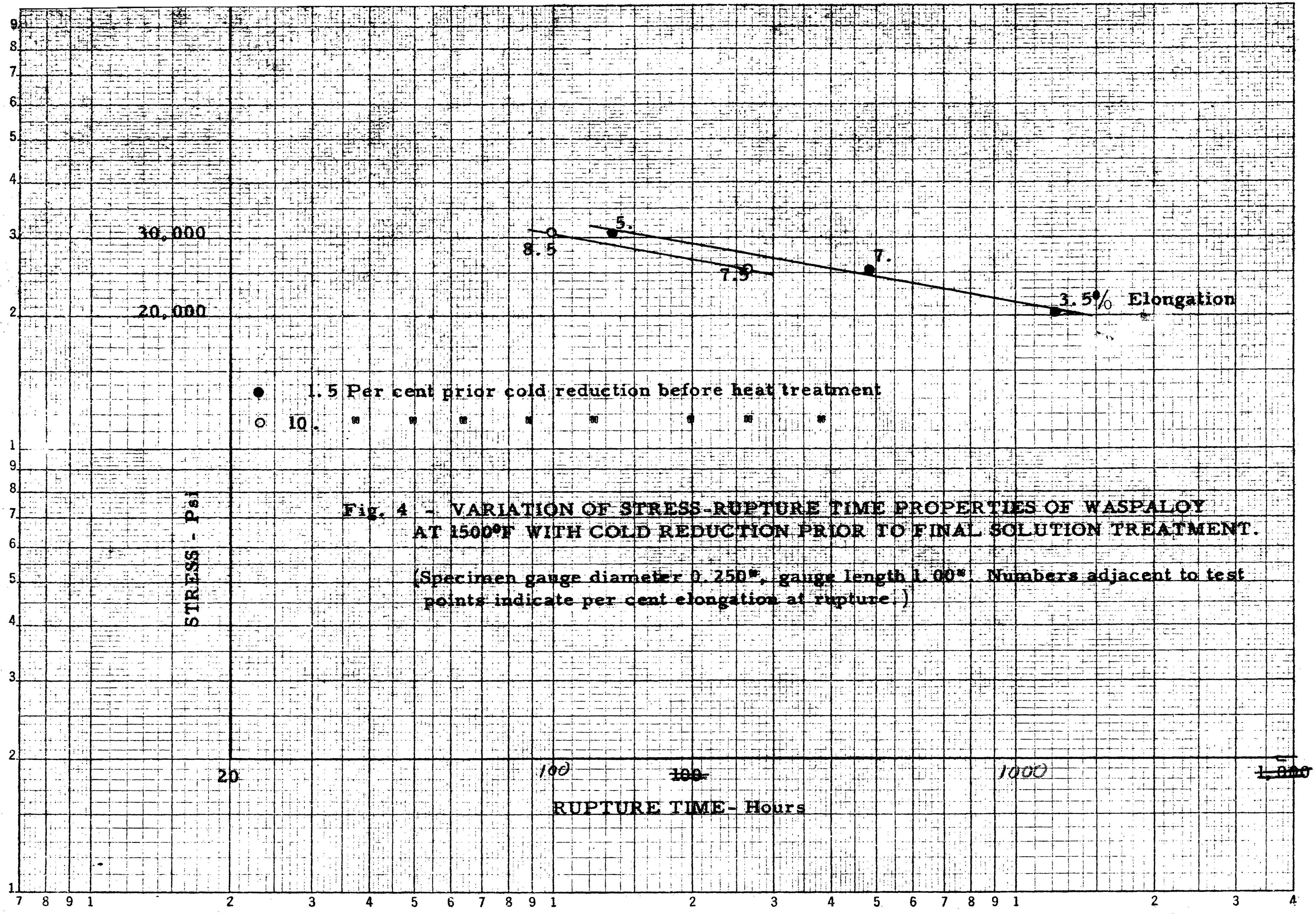


Fig. 4 - VARIATION OF STRESS-RUPTURE TIME PROPERTIES OF WASPALOY AT 1500°F WITH COLD REDUCTION PRIOR TO FINAL SOLUTION TREATMENT.

(Specimen gauge diameter 0.250", gauge length 1.00". Numbers adjacent to test points indicate per cent elongation at rupture.)