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AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS

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ABSTRACT

One heat of commercial Type-310 stainless steel and one heat of Hastelloy B material were oxidized under stress at temperatures of 1700°, 1800°, 1900°, and 2000°F, and 1200°, 1400°, 1600°, and 1800°F, respectively, and the severity of intergranular penetration determined microscopically. In general, the deeper penetrations increase in depth and frequency as the stress is increased. The weight gained during oxidation (without stress) was determined for the Hastelloy B material. The oxidation of this material followed the parabolic law although the rate was quite high.

INTRODUCTION

This research project has been undertaken under the sponsorship of the Wright Air Development Center of the U. S. Air Force. Its objectives are:

1. To investigate the effect of temperature and stress on the progressive intergranular oxidation and to determine the threshold stress of some stainless steels and high-nickel alloys at temperatures of 1200°F and above.

2. To measure the oxidation rates of these materials by means of the weight gained during oxidation.

3. To study the subsurface oxides.

MATERIAL

The material used during this portion of the investigation consisted of one heat of commercial Type-310 stainless steel and one heat of Hastelloy B. The Type-310 stainless was received in the form of cold-rolled and annealed strip 1.0 x .05 in. and the Hastelloy B in the form of hot-rolled strip 1.0 x .038 in., which had received a light cold-roll pass to improve the surface and a final anneal. The analysis of the Type-310 alloy is 19.26% Ni., 25.42% Cr, 0.62% Si, 1.58% Mn, 0.05% C, remainder Fe, and the Hastelloy B material is 5.19% Fe, 28.01% Mo, 0.26% Cr, 0.45% V, 0.74% Co, 0.24% Si, 0.60% Mn, 0.03% C, remainder Ni.

PROCEDURE

The oxidation procedure for the stress-oxidation tests was the same as that previously used, except that the samples were not quenched, but air-cooled. Since x-ray analysis of the oxides is not now a regular part of the investigation, this simplification was possible. The weight-gain method is the same as that described in the WADC TR 55-470, Part I. The metallographic and penetration-counting procedure was the same as that used in the previous investigation.

In addition to the above procedure, another method of evaluating and presenting the penetration data was used during this quarter. It is known that the most uncertain types of penetrations are the extremely small ones. These represent in the main true intergranular penetration as is shown by high-magnification examination. However, some are the result of polishing artifacts or are surface-roughness effects which usually occur during oxidation. In addition to being somewhat uncertain, these small penetrations dominate the evaluation with respect to total number and mean depth, since they are present to a greater extent than the deeper ones. To make the effect of stress and temperature on the deeper and more significant penetrations more noticeable, it was decided to make a modified number and mean-depth calculation, excluding the smaller penetration. To do this the first group, and in a few instances the first and second groups, were excluded and a modified number and mean-depth calculation made. Each group is 0.0006 in. deep. A complete explanation of the penetration-counting and calculating procedure is given in WADC TR 55-470, Part I. The penetration frequency curves which have been a regular method of reporting data also give these data, since each curve represents the distribution of penetrations of various depths. However, this new method shows the changes in the penetration character in the critical regions to a more marked extent.

RESULTS AND DISCUSSION

The commercial Type-310 stainless steel and Hastelloy B material have been tested at varying stresses at temperatures of 1700°, 1800°, 1900° and 2000°F, and 1200°, 1400°, 1600°, and 1800°F, respectively. The stress levels were chosen so as to give a representative count of intergranular penetration in the low stress regions and were concentrated in the region where the stress caused an increase in the depth of these penetrations. This discussion will be divided into two classifications, commercial Type-310, Heat 25139, and Hastelloy B, Heat B-1400.

COMMERCIAL TYPE-310 ALLOY, HEAT 25139

This material was exceedingly clean in the as-received condition, had a very good surface, and showed no significant surface defects when prepared metallographically in a manner similar to that used for the oxidized specimen.

The variation in the number of intergranular penetrations with depth below the metal oxide interface at constant testing temperature is given in Figs. 1 through 4. These curves follow the general decay type as described in WADC 54-120. As the temperature of testing is increased, the number of deep penetrations increases. When the stress at a given temperature is increased, the number of deep penetrations also increases.

Figures 5 through 12 are the penetration-number and mean-penetration-depth graphs. The number of graphs show the effect of stress at a constant temperature on the total number of penetrations and the number of penetrations, excluding the minor groups. The mean-depth graphs show the effect of stress on the mean depth of penetration and a modified mean depth which is obtained by excluding minor groups. In both types of graphs the elongation observed in 2 in. at the end of the 100-hour test is shown. The elongation is plotted for all samples tested, although it was impossible to obtain penetration data on some of the more highly stressed samples since they contained fissures throughout the matrix. In the 1700°,1800°, and 2000°F tests the number decreases as the stress is increased when all groups are considered. However, the number of deep penetrations is not decreased when the minor groups are excluded, as is shown by the number curves. In Fig. 5 (1700°F) the curve excluding the first group showed an increase in penetration number with increasing stress, while the 1800° and 2000°F graphs showed a decrease even when the first groups were excluded, as shown in Figs. 7 and 11. Excluding the first and second groups showed no appreciable effect of stress on number in the 1800° and 2000°F graphs.

Figure 9 (1900°F) shows an increase in the number of penetrations for the total number as well as the condition when the minor groups are excluded.

Figures 6 and 10 show that the mean depth, considering all penetrations, increases significantly in the 1700° and 1900°F tests and that the deeper penetrations increase even more noticeably. The 1800°F graph shows a shift in the shape of the curve as the number of groups considered is decreased. Considering all groups, the mean depth decreases slightly, while excluding the first group shows no effect of stress, and excluding the first and second groups shows a significant increase in the mean depth.

HASTELLOY B, HEAT B-1400

The Hastelloy B material was tested at temperatures of 1200°, 1400°, 1600° and 1800°F in the stress-oxidation units and weight-gain equipment as described in the procedure portion of this report.

It is interesting to note that the oxide scale obtained during 1400°, 1600°, and 1800°F tests flaked off completely during cooling. The surface after this flaking was powdery, dark blue-grey in the 1400°F samples, powdery, light blue-grey in the 1600°F samples, and shiny copper color in the 1800°F samples. The powdery surface could be rubbed off, exposing a steel-blue surface in each case, while the copper-colored surface was very adherent. The 1200°F tests resulted in a fairly adherent dark blue-black oxide. These colors and conditions were quite reproducible and were independent of stress.

Figure 13 shows the weight gained during oxidation at constant temperatures of 1200°, 1400°, 1600°, and 1800°F. The rate at 1600° and 1800°F is far above any previously encountered in this investigation, as the constants at 1600° and 1800°F are 0.047 and 0.36, the latter being more than 20-fold greater than any value obtained for the stainless steels at any temperatures investigated. This factor, combined with the flaking of the oxide, would limit the use of this material in this temperature range.

Figures 14 through 17 are the penetration frequency graphs giving the relationship between penetration depth and number. These curves are of the decay-curve type previously discussed. The effect of stress in the critical region (that region where the material elongates considerably) could not be determined for the 1200°F tests since the units were not constructed to carry a load greater than 200 lb. A 1/4-in. cross section was the smallest practical specimen size, and therefore the maximum stress obtainable was about 20,000 psi, which was insufficient. The testing at 1200°F was therefore discontinued when 20,000 psi showed negligible elongation at the end of the test. The 1200°, 1400°, and 1600°F graphs are similar, although there are more deep penetrations present in the 1600°F graphs. The 1800°F graph differs from these three in that the distribution is shifted in the direction of the deeper penetrations. In general, increasing the stress causes a greater number of deeper penetrations. This effect is more pronounced at higher temperatures.

Figures 18 through 25 show the effect of stress on the number and mean depth of penetrations. The 1200°F tests were conducted at a stress below the critical range, as previously discussed, and therefore do not show any appreciable effect. Since all of the penetrations encountered were extremely small, the slight increase in number is not considered significant. The large amount of scatter encountered in the 1400°F curve makes the effect of stress on number difficult to determine, while the 1600°F shows an increse and the 1800°F a slight decrease in number. Excluding the first group shows an increase in the number of deep penetrations in the 1800°F graph. This exclusion is of no value in the lower-temperature tests since the penetrations are for the most part in the first group. The mean depth of penetration is not appreciably affected by stress in the 1200°, 1400°, and 1600°F tests as shown in Figs. 19, 21, and 23. In the 1800°F tests, however, the mean depth is increased significantly with increasing stress. This is especially noticeable in the deeper penetrations, as is shown by the curves excluding the minor groups. It was not possible to obtain a great deal of elongation in the 1800°F tests. Some specimens which fractured during the latter part of the test showed only 3.5% elongation. The proposed metallographic analysis of this material should shed some light on this problem.

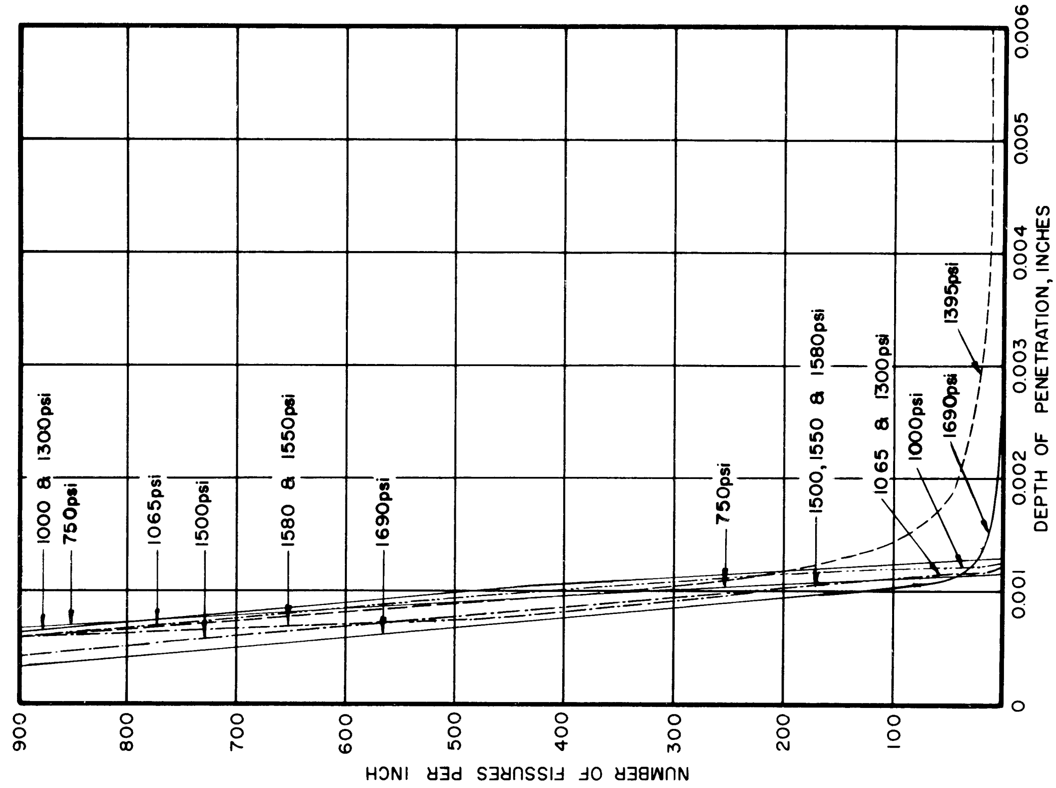

FIG. 2 PENETRATION VS. DEPTH BELOW SURFACE.
TYPE 310 STAINLESS, HT. 25139. 1800°F, 100 HOURS.

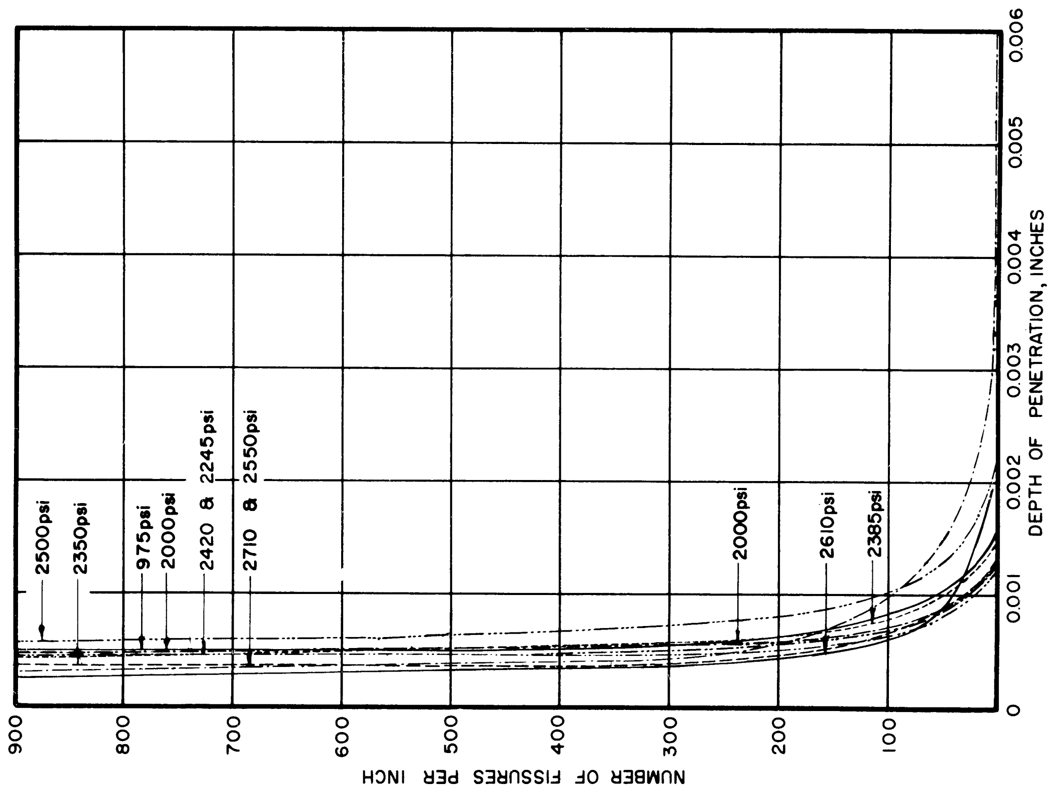

FIG. 1 PENETRATION VS. DEPTH BELOW SURFACE.
TYPE 310 STAINLESS, HT. 25139. 1700°F, 100 HOURS.

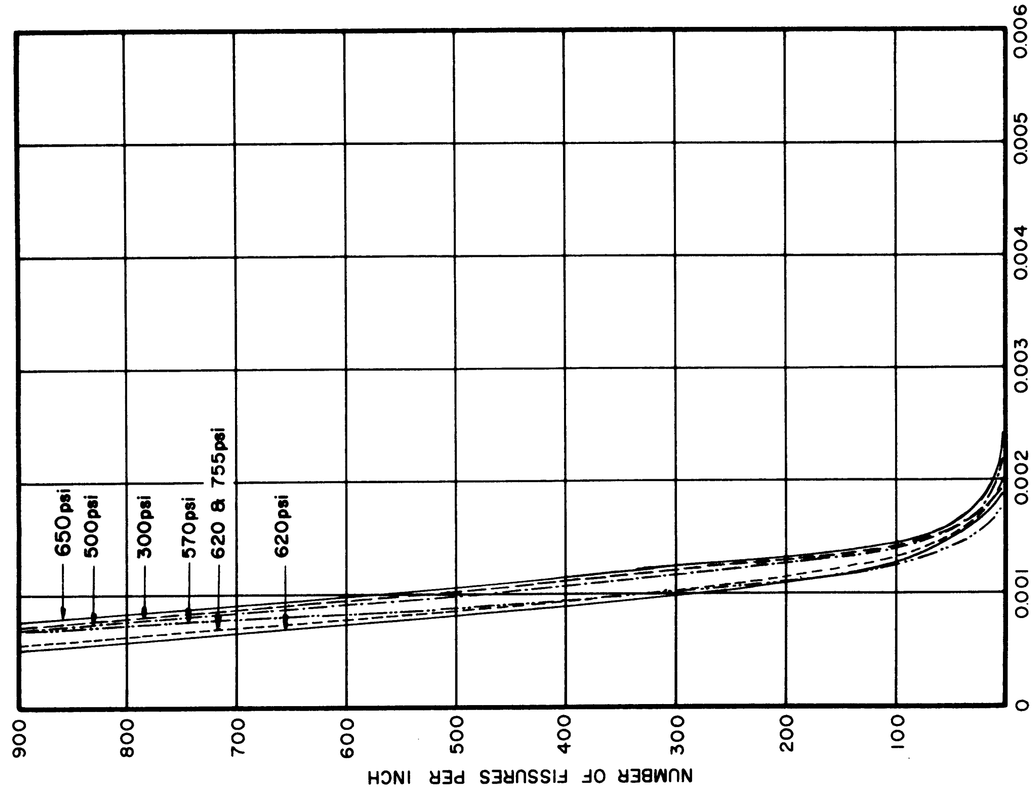

FIG. 4 PENETRATION VS. DEPTH BELOW SURFACE.
TYPE 310 STAINLESS, HT. 25139. 2000°F, 100 HOURS.

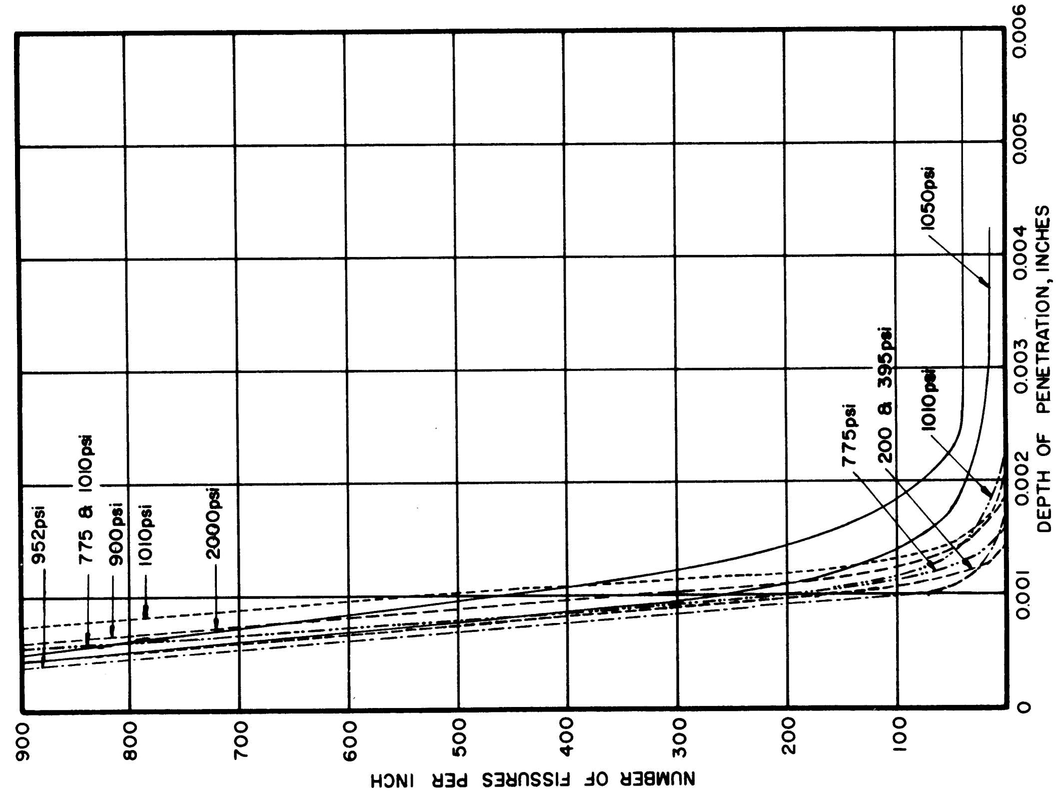

FIG. 3 PENETRATION VS. DEPTH BELOW SURFACE.
TYPE 310 STAINLESS, HT. 25139. 1900°F, 100 HOURS.

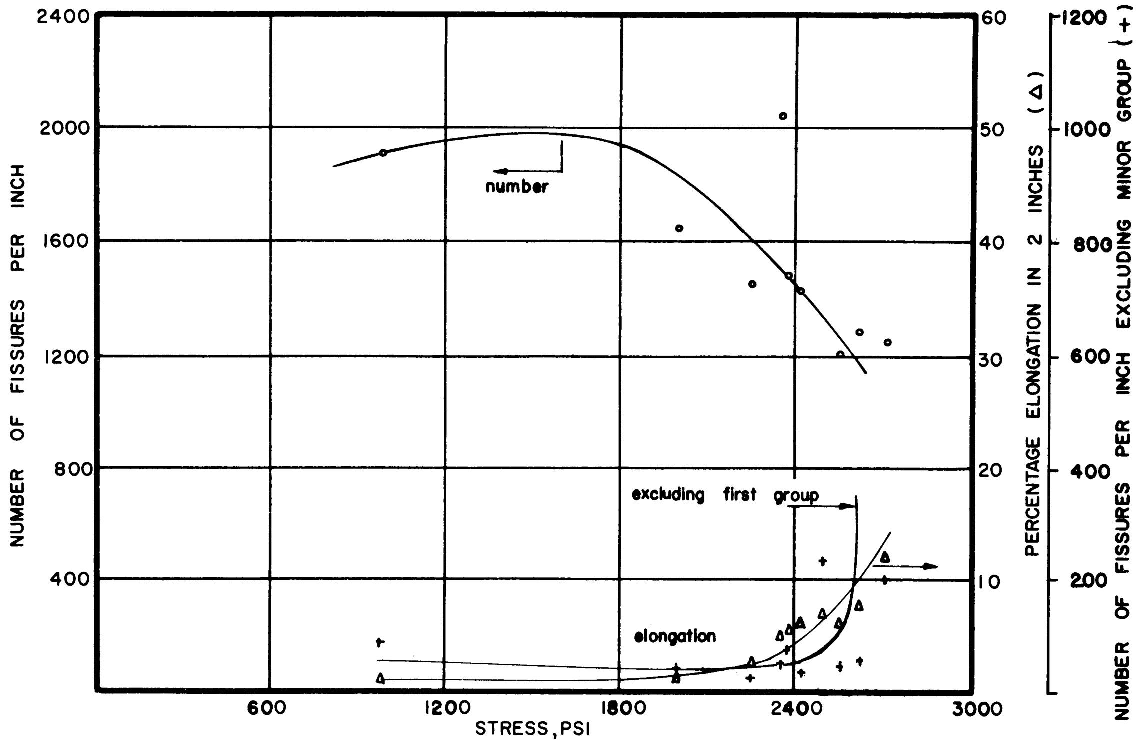

FIG. 5 SUMMARY PENETRATION-FREQUENCY CURVES.
TYPE 310 ALLOY, HT. 25139, 1700°F, 100 HOURS.

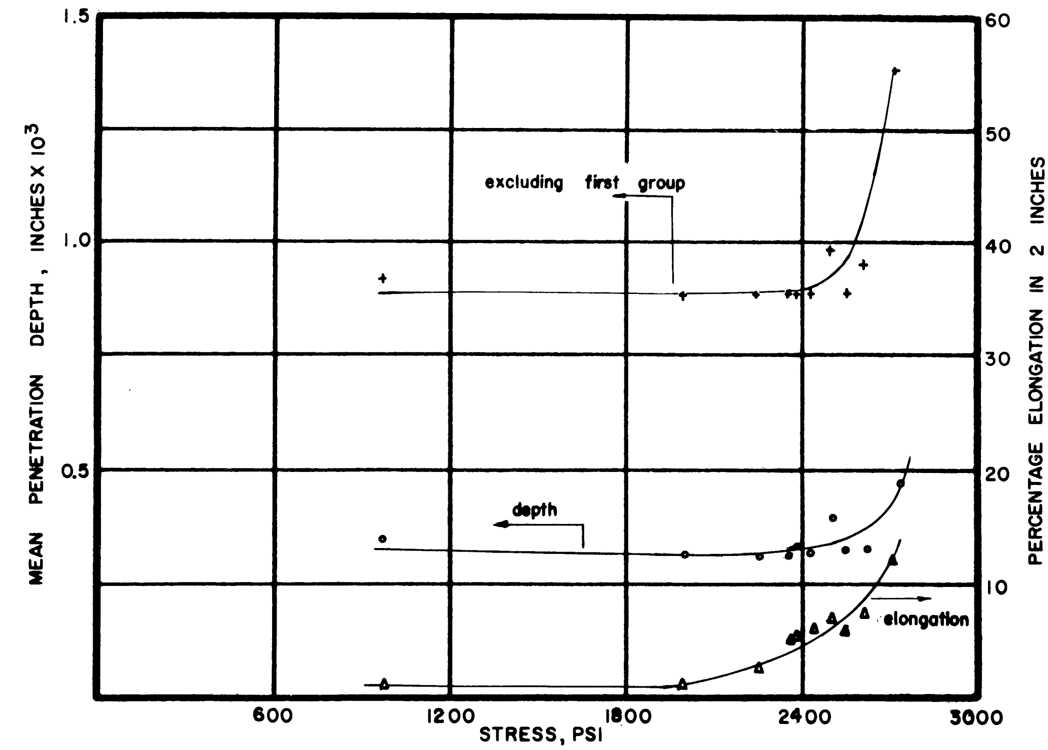

FIG. 6 SUMMARY PENETRATION-DEPTH CURVES.
TYPE 310 ALLOY, HT. 25139, 1700°F, 100 HOURS.

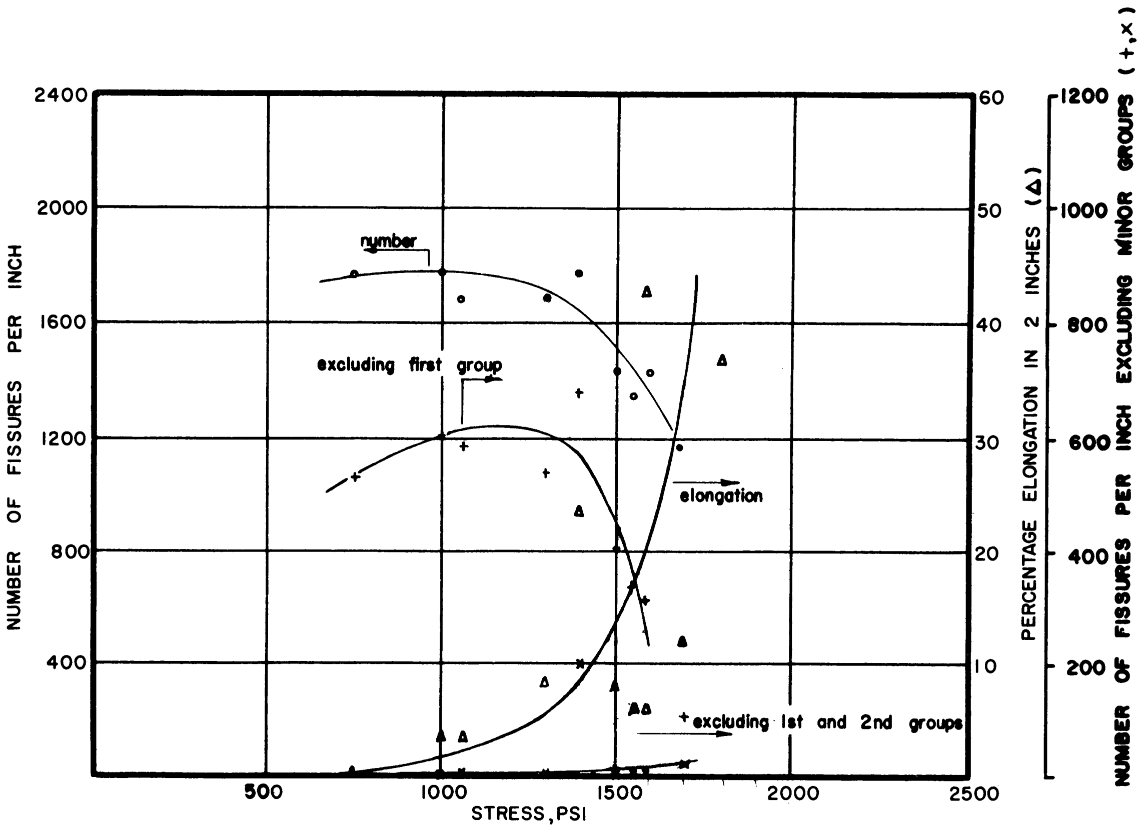

FIG. 7 SUMMARY PENETRATION-FREQUENCY CURVES.
TYPE 310 ALLOY, HT. 25139, 1800°F, 100 HOURS.

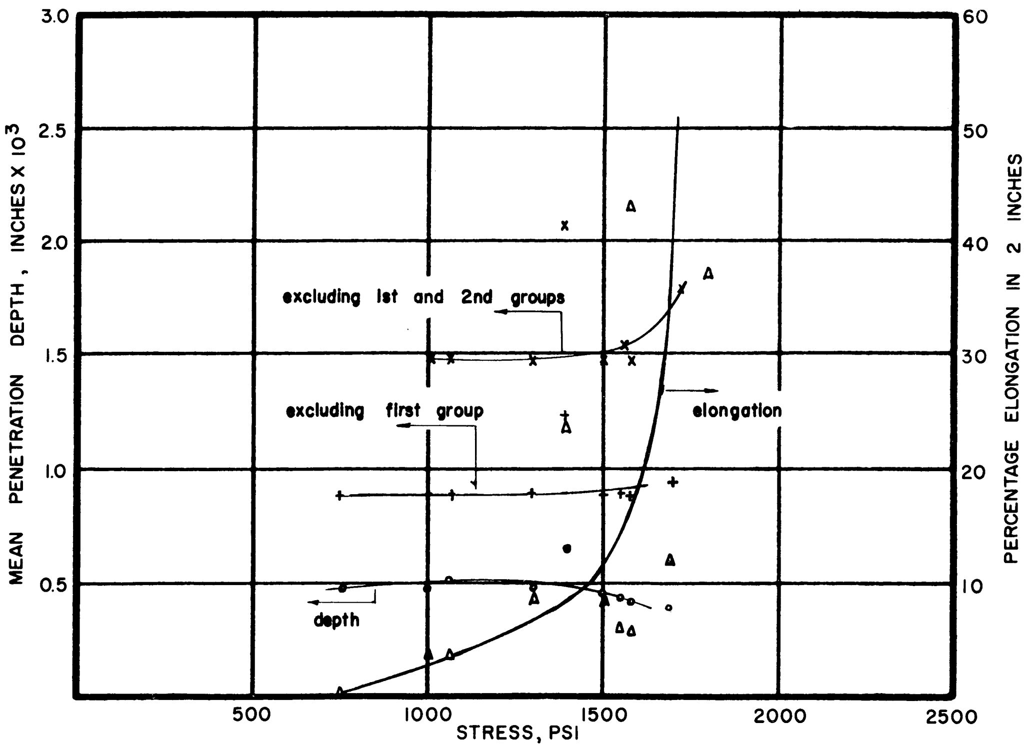

FIG. 8 SUMMARY PENETRATION-DEPTH CURVES.
TYPE 310 ALLOY, HT. 25139, 1800°F, 100 HOURS.

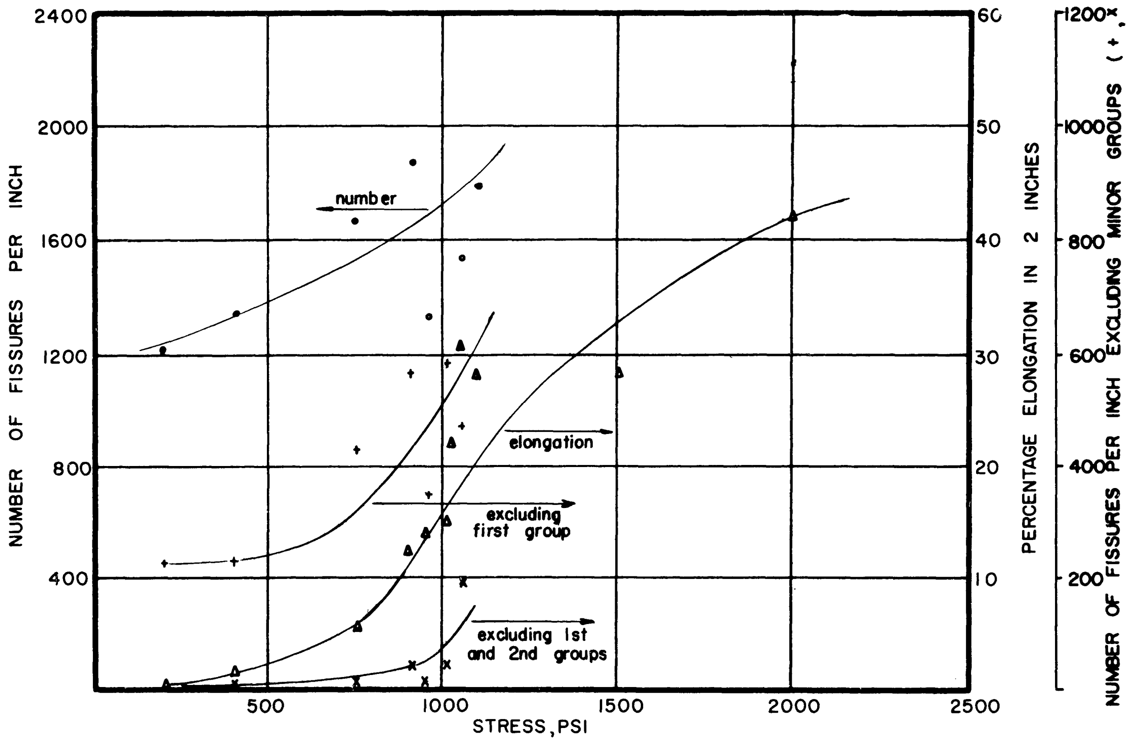

FIG. 9 SUMMARY PENETRATION-FREQUENCY CURVES.
TYPE 310 ALLOY, HT. 25139, 1900°F, 100 HOURS.

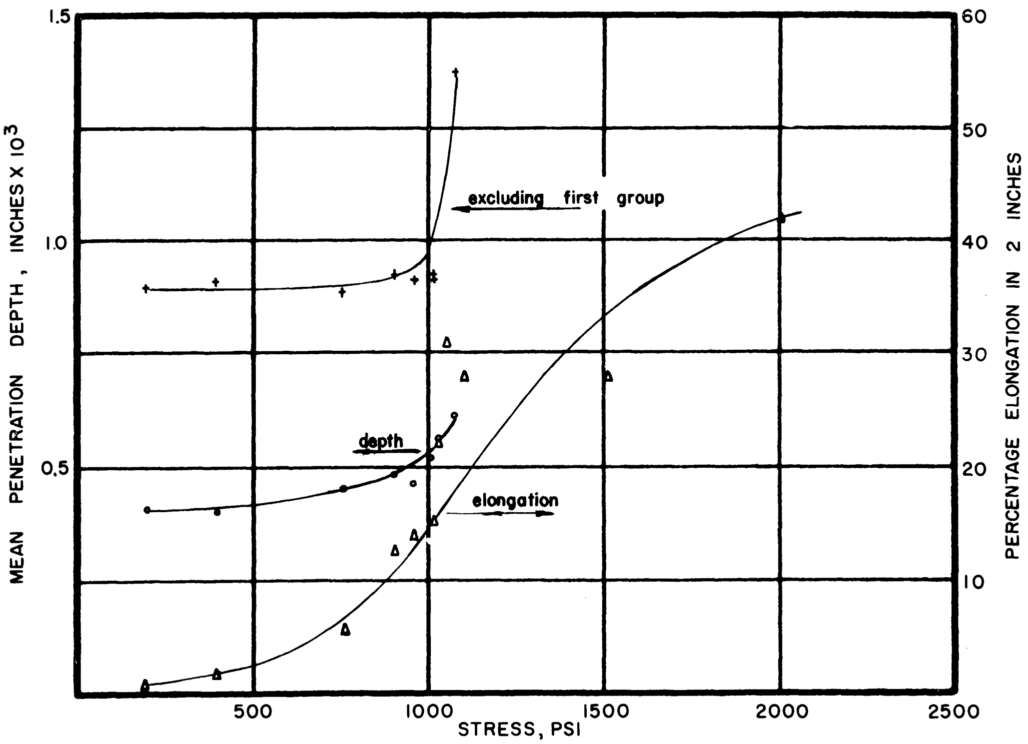

FIG. 10 SUMMARY PENETRATION-DEPTH CURVES.
TYPE 310 ALLOY, HT. 25139, 1900°F, 100 HOURS.

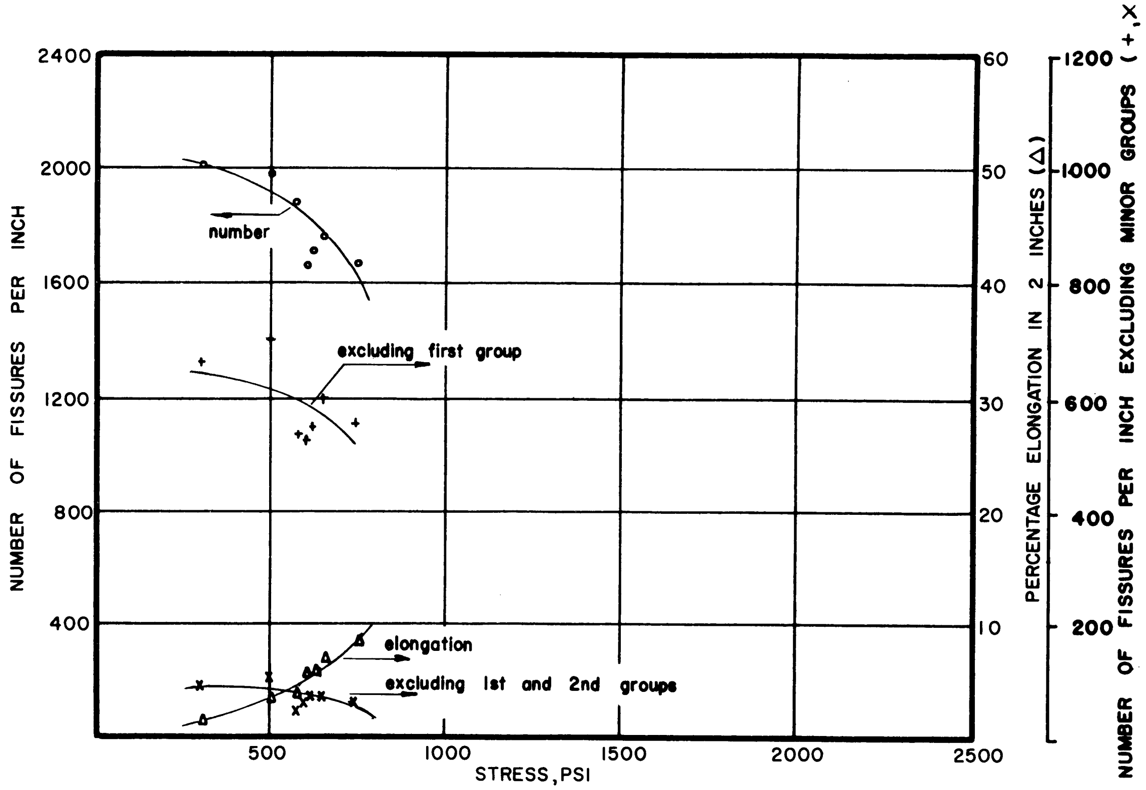

FIG. 11 SUMMARY PENETRATION-FREQUENCY CURVES.
TYPE 310 ALLOY, HT. 25139, 2000°F, 100 HOURS.

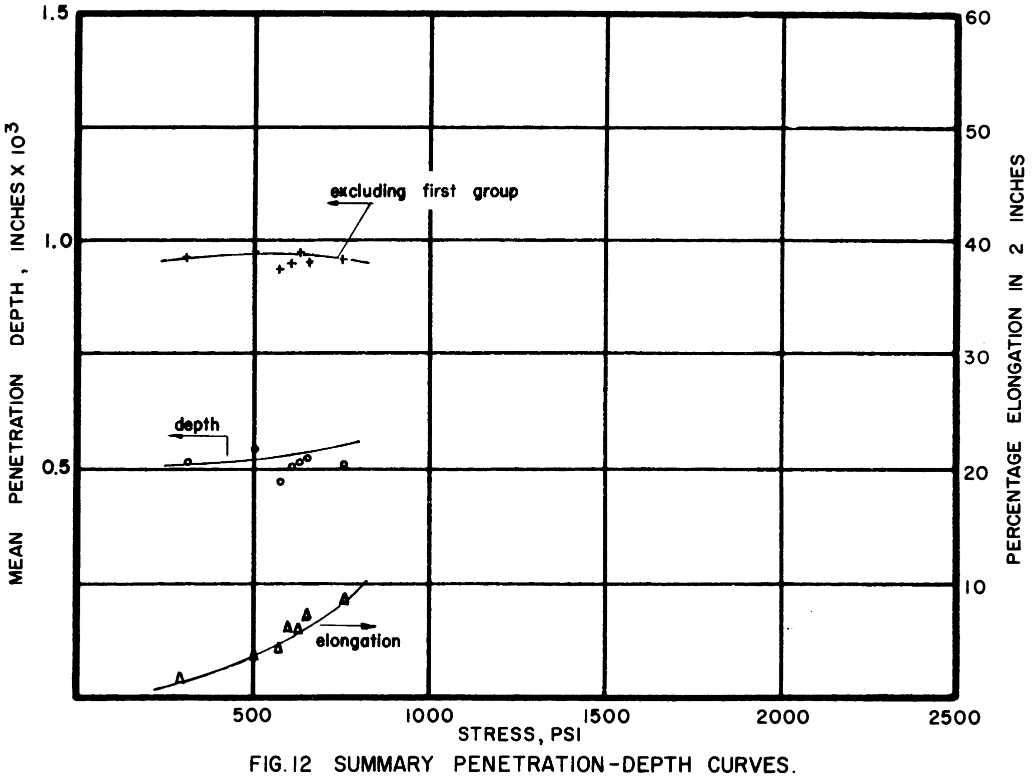

FIG. 12 SUMMARY PENETRATION-DEPTH CURVES.
TYPE 310 ALLOY, HT. 25139, 2000°F, 100 HOURS.

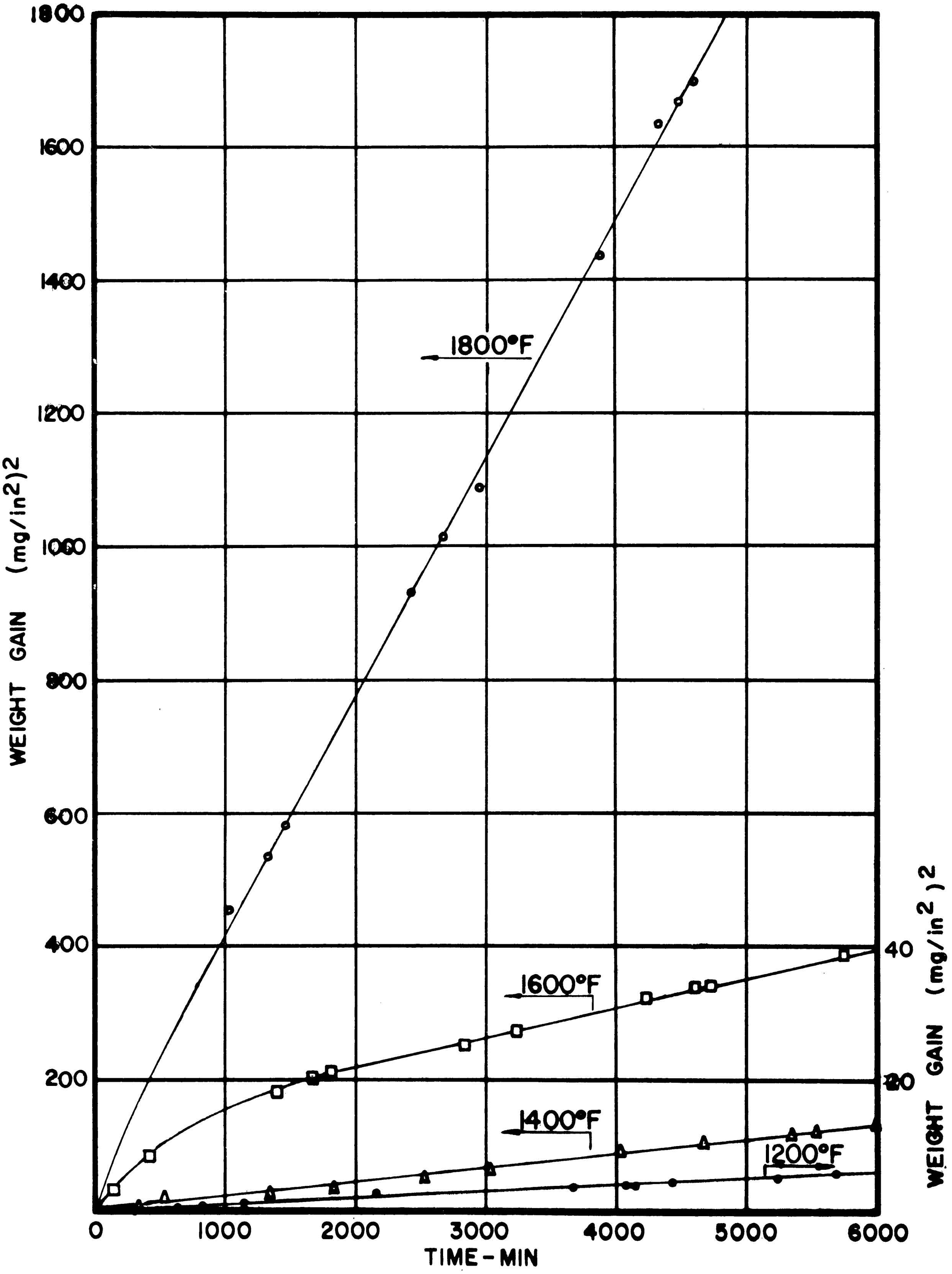

FIG. 13 APPLICATION OF PARABOLIC RELATIONSHIP TO WEIGHT-GAIN DATA; HASTALLOY B, HT. B-1400.

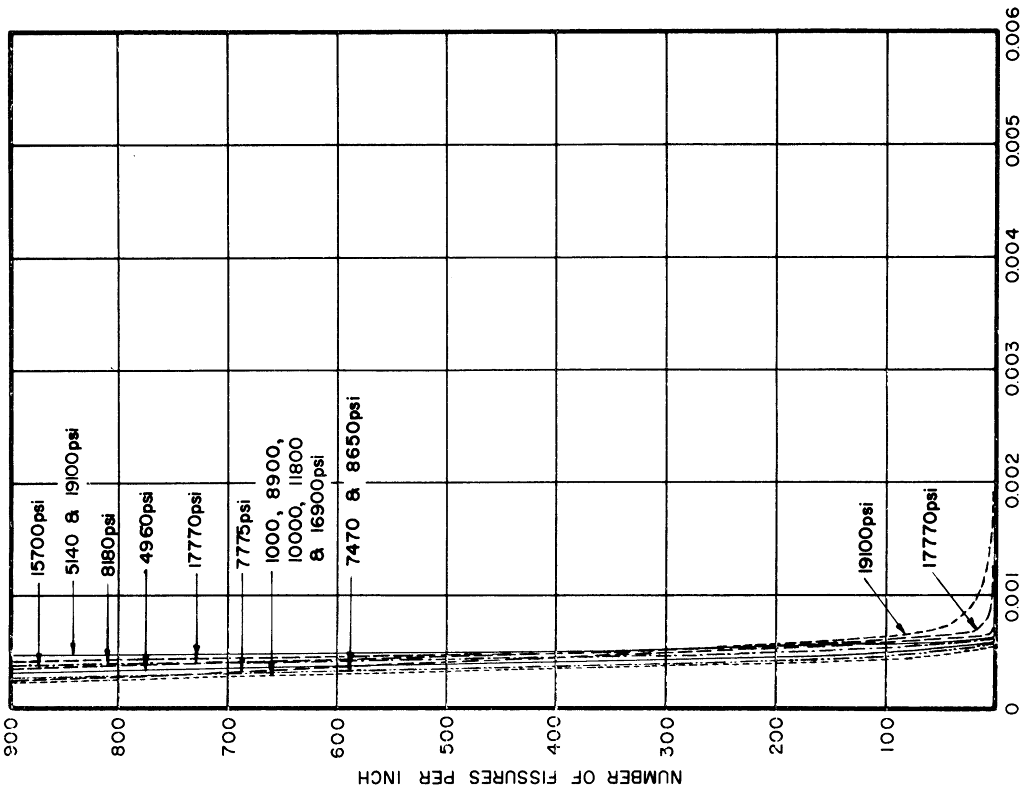

FIG. 15 PENETRATION VS. DEPTH BELOW SURFACE.
HASTALLOY B, HT. B-1400, 1400°F, 100 HOURS.

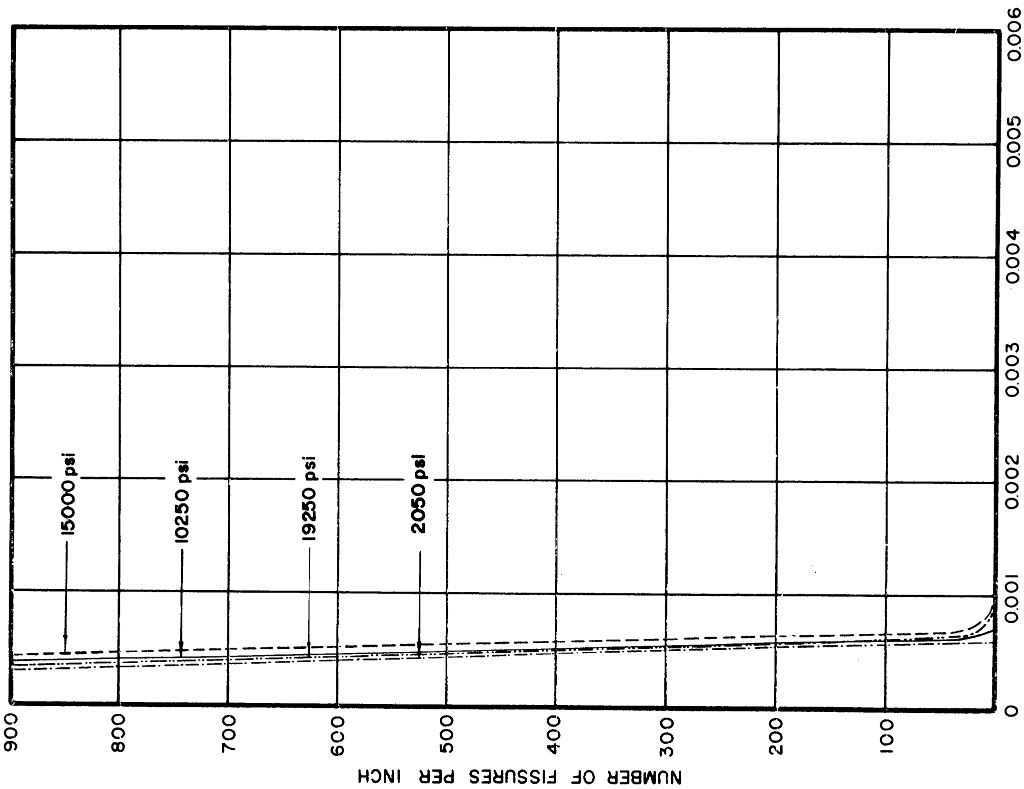

FIG. 14 PENETRATION VS. DEPTH BELOW SURFACE.
HASTALLOY B, HT. B-1400, 1200°F, 100 HOURS.

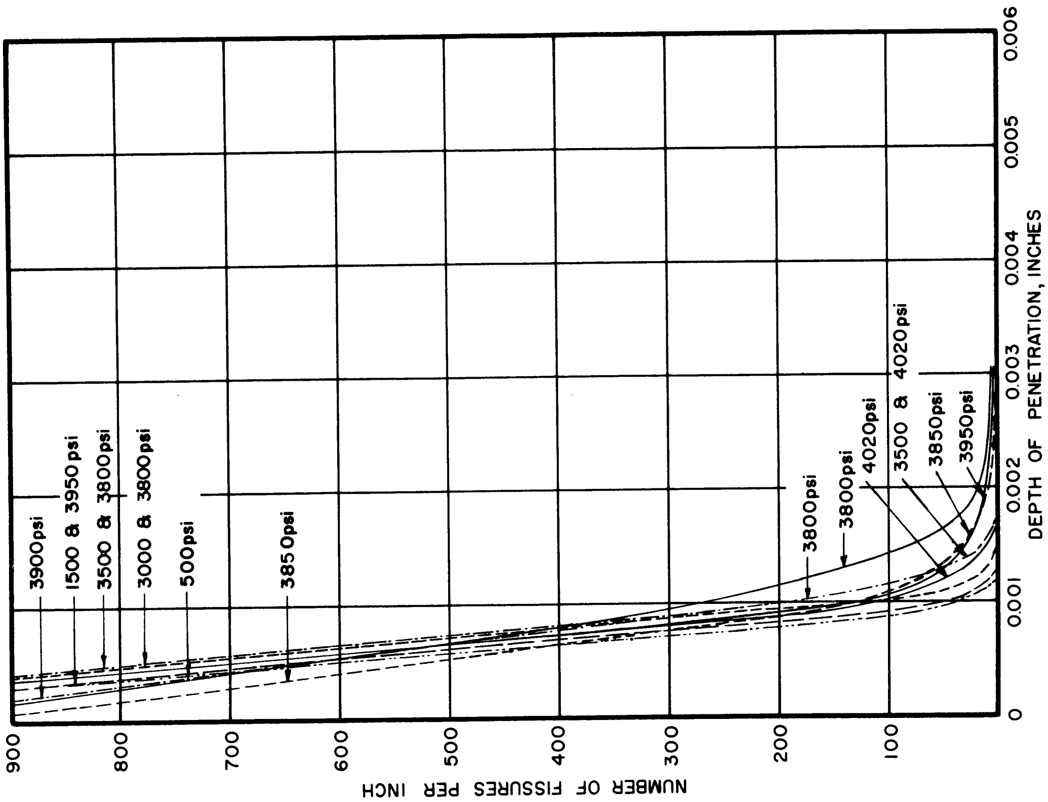

FIG. 17 PENETRATION VS. DEPTH BELOW SURFACE.
HASTALLOY B, HT. B-1400, 1800°F, 100 HOURS.

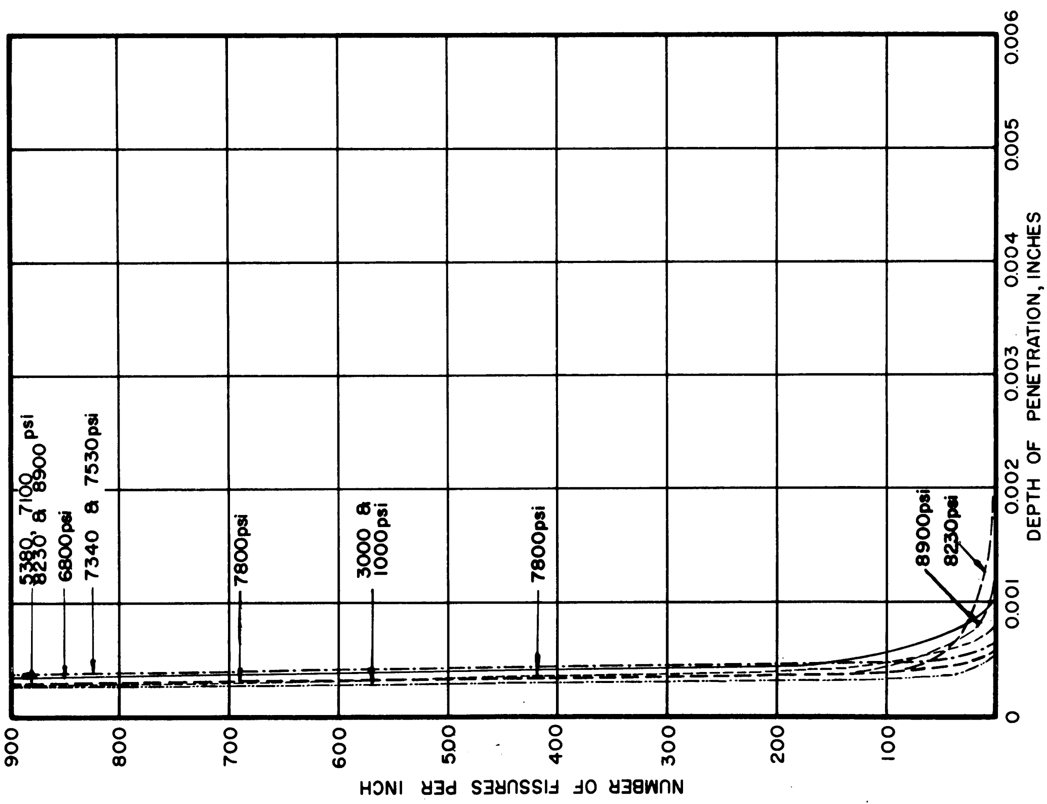

FIG. 18 PENETRATION VS. DEPTH BELOW SURFACE.
HASTALLOY B, HT. B-1400, 1600°F, 100 HOURS.

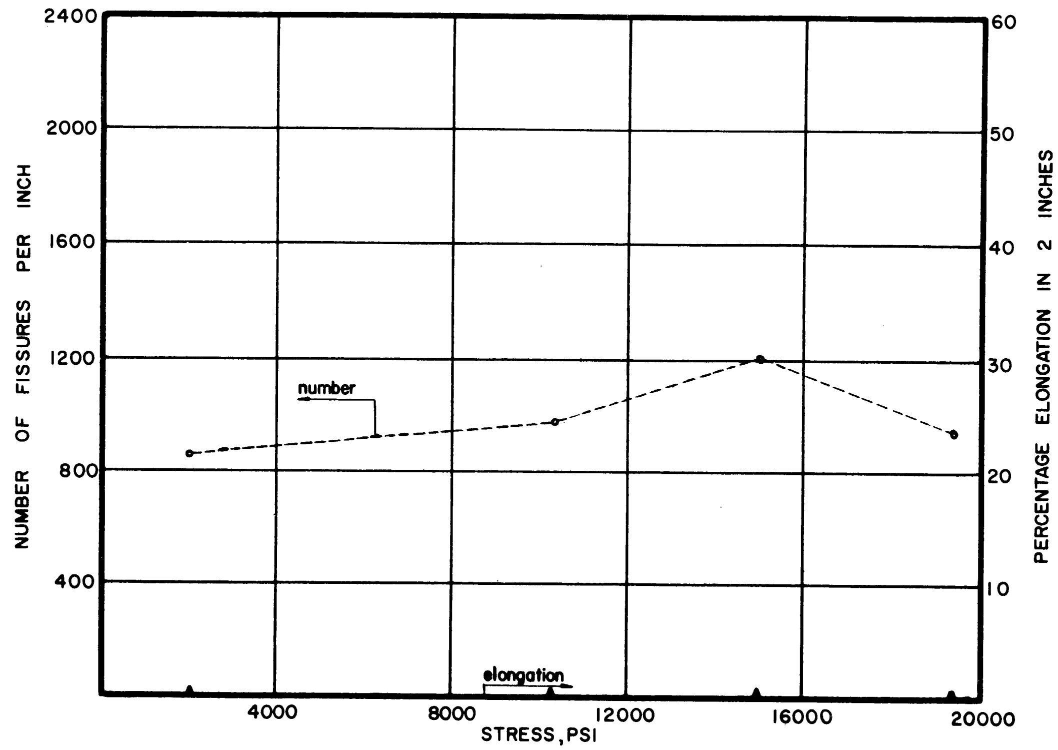

FIG. 18 SUMMARY PENETRATION-FREQUENCY CURVES
HASTALLOY B, HT. B-1400, 1200°F, 100 HOURS.

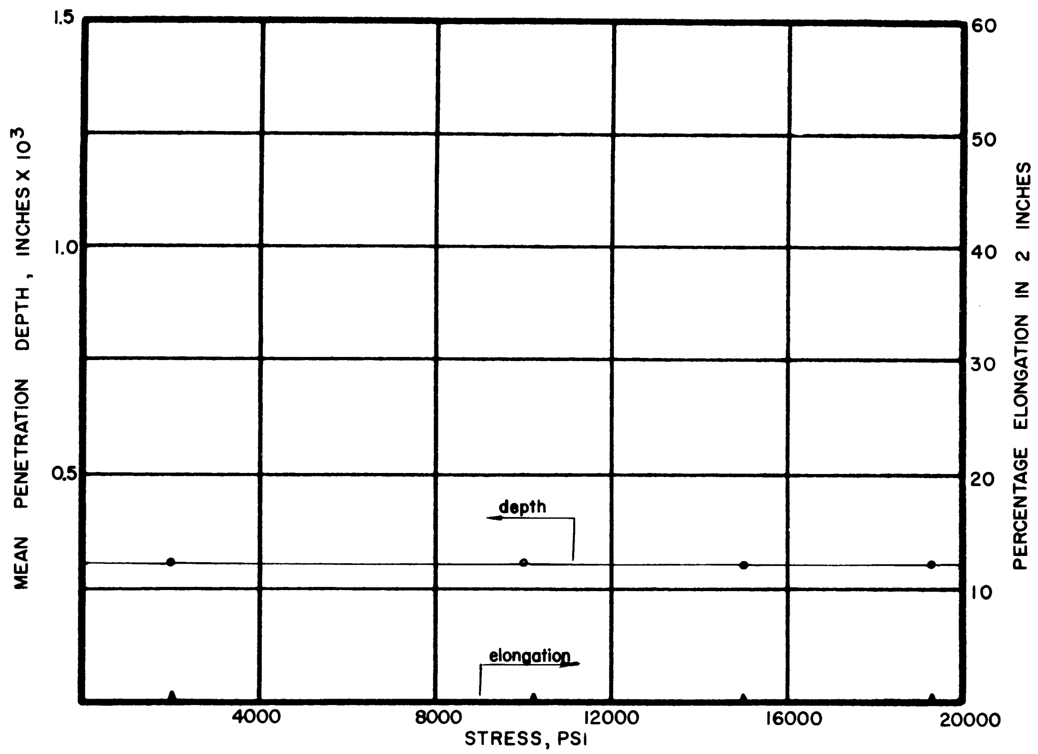

FIG. 19 SUMMARY PENETRATION-DEPTH CURVES.
HASTALLOY B, HT. B-1400, 1200°F, 100 HOURS.

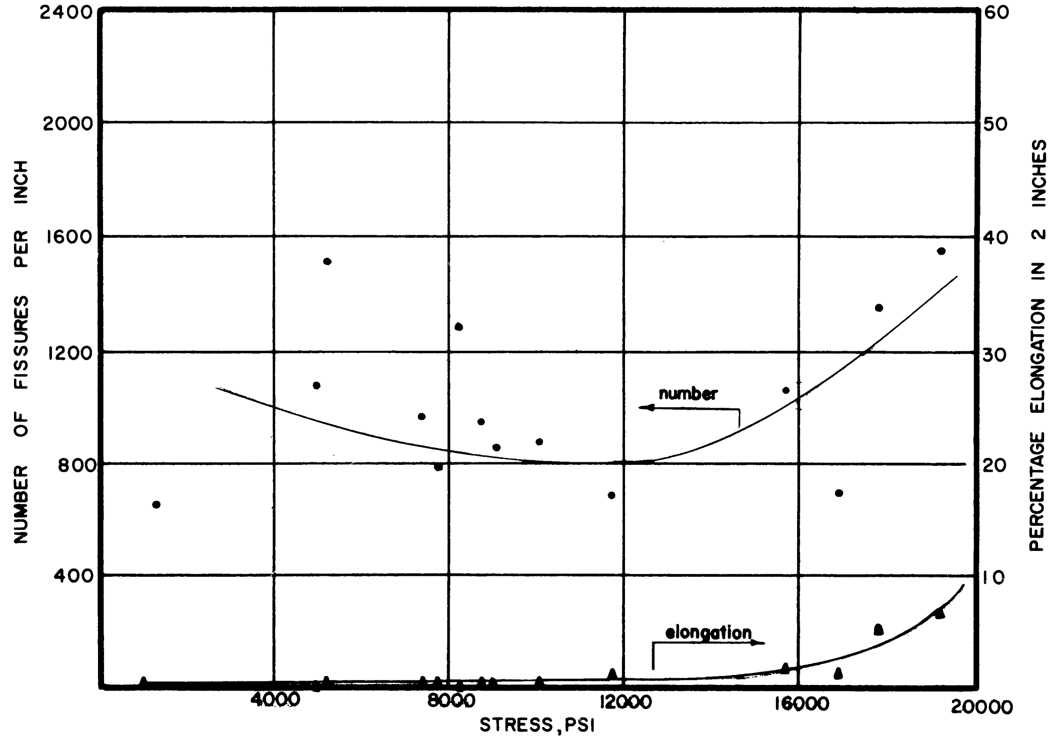

FIG. 20 SUMMARY PENETRATION-FREQUENCY CURVES.
HASTALLOY B, HT. B-1400, 1400°F, 100 HOURS.

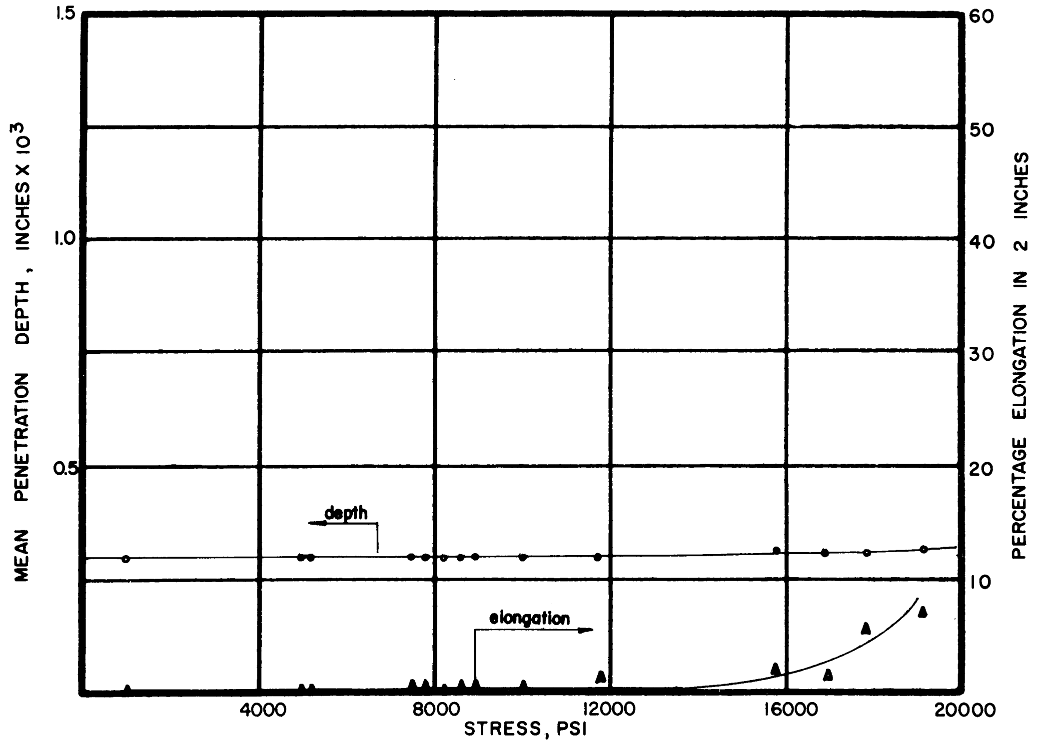

FIG 21 SUMMARY PENETRATION-DEPTH CURVES.
HASTALLOY B, HT. B-1400, 1400°F, 100 HOURS.

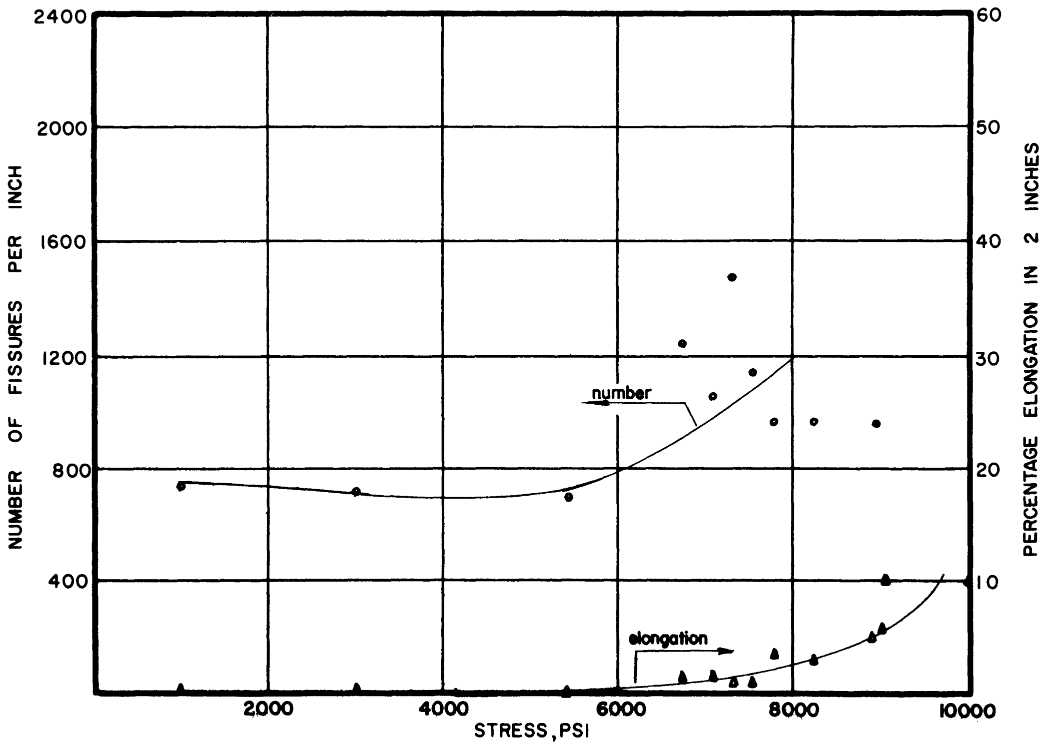

FIG. 22 SUMMARY PENETRATION-FREQUENCY CURVES.
HASTALLOY B, HT. B-1400, 1600°F, 100 HOURS.

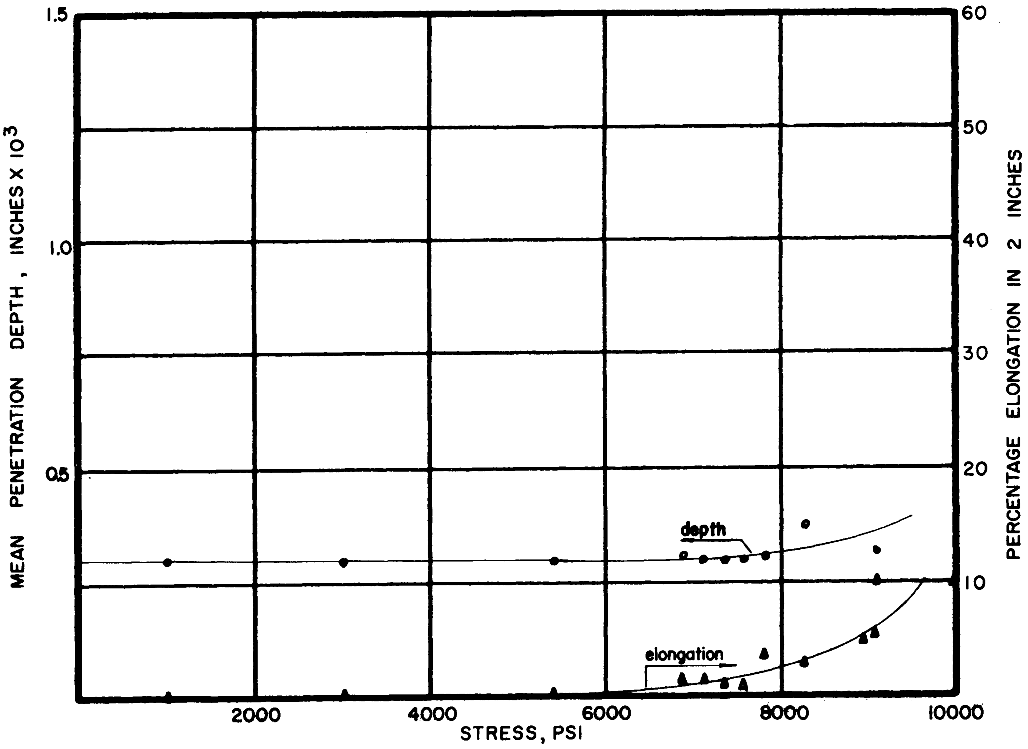

FIG. 23 SUMMARY PENETRATION-DEPTH CURVES.
HASTALLOY B, HT. B-1400, 1600°F, 100 HOURS.

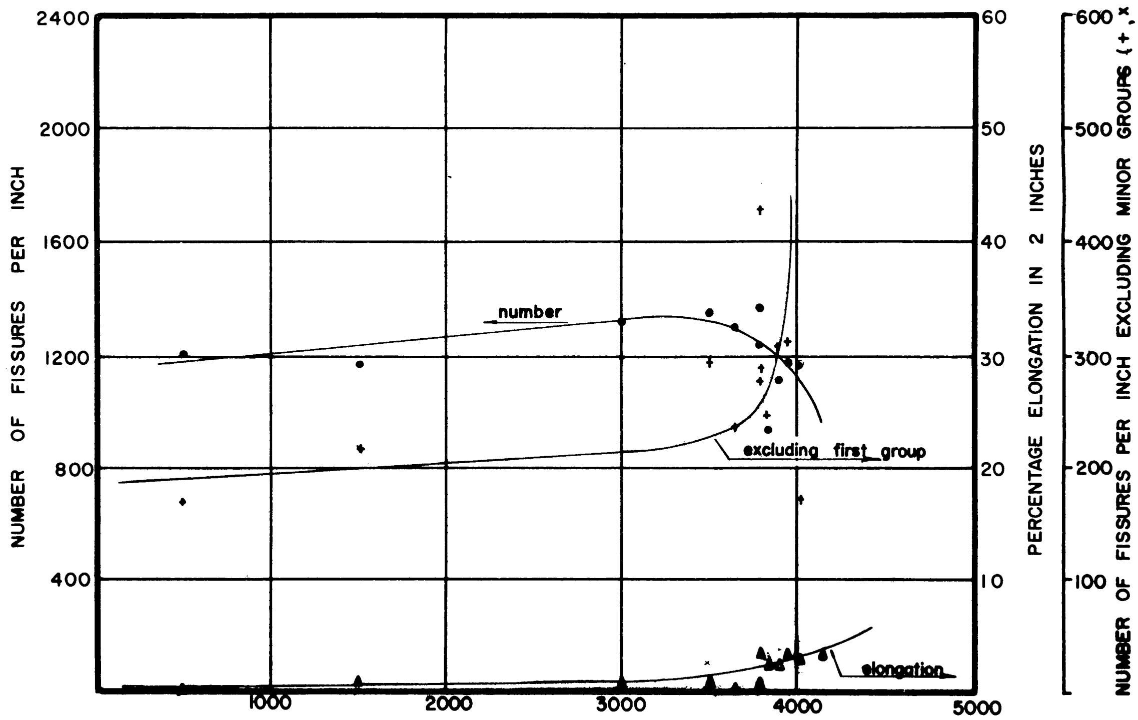

FIG. 24 SUMMARY PENETRATION-FREQUENCY CURVES.
HASTALLOY B, HT. B-1400, 1800°F, 100 HOURS.

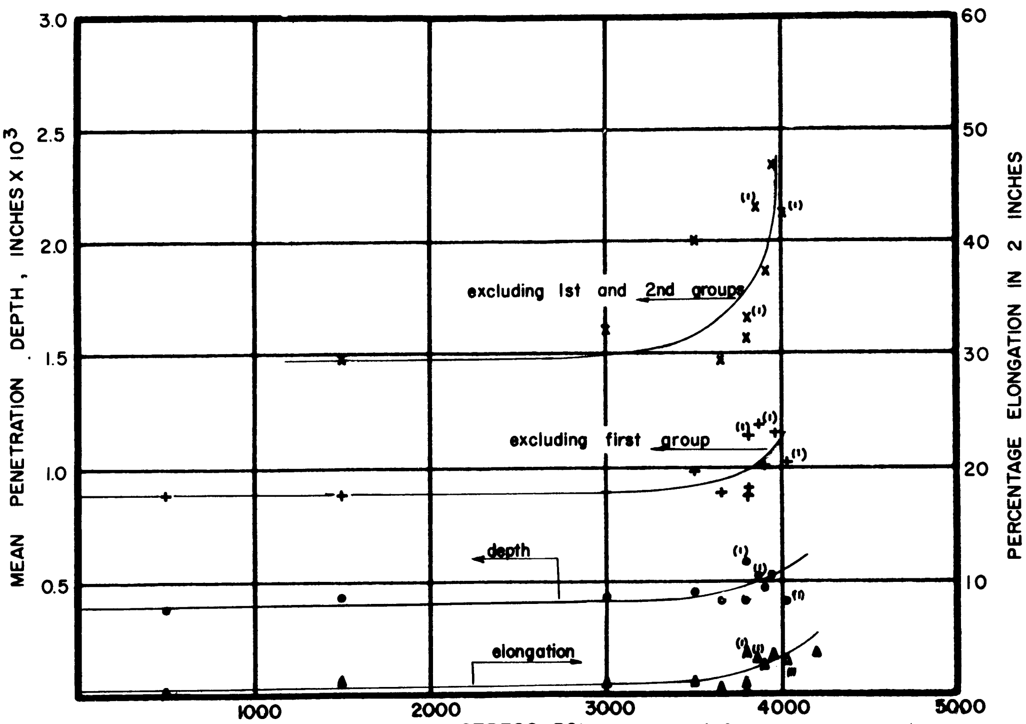

FIG. 25 SUMMARY PENETRATION-DEPTH CURVES.
HASTALLOY B, HT. B-1400, 1800°F, 100 HOURS.