Engineering Research Institute
University of Michigan
Ann Arbor

PROGRESS REPORT NO. 2
(through January 31, 1953)

INVESTIGATION OF THE INFLUENCE OF Ti-Al-B ON THE HIGH-TEMPERATURE PROPERTIES OF Cr-Ni-Mo-Fe AUSTENITIC ALLOYS

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Project 2061

TO

FLIGHT RESEARCH LABORATORY (WCRRL)
RESEARCH DIVISION
WRIGHT AIR DEVELOPMENT CENTER
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Contract No. AF(616)-173
Expenditure Order No. R-463-8 BR-1

February 1953

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SUMMARY

This is the second quarterly progress report under Contract Number AF 33(616)-173. Data in this report cover further results from a study of the mechanism by which boron increases the high-temperature properties of a lean austenitic alloy with titanium boron additions; the material used in this part of the investigation was bar stock from an arc-furnace Heat A-5764 of the following composition:

0.069C, 1.43Mn, 0.58Si, 0.011S, 0.014P, 12.50Cr,
16.20Ni, 0.60W, 2.42Mo, 0.58Ti, 0.027B.

The effect of the solution treating temperature on various properties of the alloy has been studied. The low melting point phase which appeared in the grain boundaries of laboratory induction Heat D42 (reported in First Progress Report) after solution treating at 2200°F was also observed in the arc-furnace Heat A-5764. This led to the belief that 2150°F was the maximum safe solution treating temperature. Solution treating at 2150°F produced a 5,000 psi higher 100-hour rupture strength than a solution treatment at 1900°F.

Hot-cold working resulted in higher strengths for this alloy than any other treatment yet tried; higher strengths resulted from hot-cold working after a 2150°F solution treatment than after a 1900°F solution treatment.

Aging of this alloy resulted in a reduction in rupture strength. Trends still indicate that the beneficial effect of boron is due to solid

solution strengthening. Limited lattice parameter data have not yet established whether boron enters the austenite lattice substitutionally or interstitially.

INTRODUCTION

This second quarterly progress report covers the progress made through January 31, 1953, on the research work authorized under Contract Number AF 33(616)-173 (Expenditure Order No. R-463-8 BR-1).

The work covered in this report is a continuation of the study of the mechanism by which boron and titanium improve the properties of high-temperature alloys.

All testing in this period was done on an arc-furnace heat of material, Heat A-5764, supplied by the Thompson Laboratory of the General Electric Company. A program was undertaken to compare the results of the two laboratory heats, D41 and D42, reported in the First Progress Report under this contract with the Heat A-5764. Emphasis was placed on the determination of solution treating temperatures and the effect of aging treatments on the hardness and rupture properties of the alloy. Following this initial work a more extensive program was outlined for studying the effects of solution treating conditions and hot-cold work on the high-temperature strength of this alloy.

GENERAL PROCEDURE

The specific objectives of the work reported here were to:

(1) determine the response of the commercial material to solution treatments,

(2) evaluate the effect of aging, and

(3) establish the influence of hot-cold working conditions on the rupture strength of the alloy A-5764.

Metallographic examinations, hardness tests, and X-ray diffraction studies were used to follow the response of the alloy to solution treatments and aging. Rupture tests at 1200°F were performed to establish the influence of the various treatments on high-temperature strength.

Test Materials

The alloy used in the work covered by this report was an arc-furnace material, Heat A-5764, processed from an eight-inch billet to 1-3/4-inch square bars by the Thompson Laboratory of the General Electric Company. Further processing to 7/8-inch square bars was done at the University of Michigan in the following manner:

A suitable length of 1-3/4-inch square bar was heated at 2050°F for 1/2 hour and rolled on a two high rolling mill using closed square passes. Four passes were used with reheats between each pass, after which the bar was water quenched, cut in half, and reheated at 2050°F for 1/2 hour. Two more passes were then made to reach the final size of 7/8-inch square.

This alloy had the following composition:

Heat No.	C	Mn	Si	S	P	Cr	Ni	Mo	W	Ti	B
A-5764	0.069	1.43	0.58	0.011	0.014	12.50	16.20	2.42	0.60	0.58	0.027

RESULTS

The influence of solution treating conditions, aging, and hot-cold work on the properties of the commercial heat of Haynes 88 was determined. The resulting characteristics were compared with the results of the investigation on the laboratory induction heats, as reported in the First Progress Report.

Solution Treatment

The microstructure of the bar stock from arc-furnace Heat A-5764 as-rolled at 2050°F to 7/8-inch sqaure is shown in figure 1. Structures after solution treating at 2150°F and 2200°F are shown in figures 2 and 3. It was observed that:

(1) The heat exhibited a notable decrease in the amount of excess phase or phases present as stringers of small particles, as compared to the laboratory boron Heat D42.

(2) No noticeable solution of the excess phase resulted at 2200°F, as was observed for the laboratory heat.

(3) Grain sizes remained constant at all solution treating temperatures, up to 2150°F for the times investigated.

(4) As was previously reported for the laboratory induction heat, the commercial heat exhibited a low melting point eutectic at the grain boundaries (see fig. 3) when subjected to a solution treatment of four hours at 2200°F. The low melting point constituent was not as extensive as previsouly encountered in the laboratory heat; furthermore, in some sections of the metallographic sample examined it existed only along the lines of stringers. This is shown in figure 4.

A comparison of the hardness of the Heat A-5764 and the laboratory Heat D42 after solution treating at different temperatures is shown in figure 5. The results on the two heats can be said to check quite well. Heat A-5764 solution treated for four hours at 1900°F had a hardness somewhat higher than was anticipated.

A one-hour treatment at 2150°F followed by water quenching has been established as the maximum solution-treating condition for further work for 0.03-percent boron material. Further work will also be conducted on samples heat treated at 1900°F for four hours and water quenched, on the basis that there should be little solution of titanium compounds at this temperature. The solution of titanium compounds at the higher temperature could well be the cause of many of the observed results.

Age Hardening

Samples of Heat A-5764 were aged at 1350°F for varying time periods up to 48 hours. The increases in hardness for these aging treatments are shown in figure 6. Aging for 48 hours at 1350°F after a solution treatment of 1 hour at 2150°F resulted in a Brinell hardness increase of 28 points. Further aging would seem to result in no great increase in hardness as is shown by the slope of the aging curve. Stock solution treated at 2200°F and then aged exhibited a higher hardness, approximately 10 Brinell hardness points, than the 2150°F solution condition. Since the maximum hardness increase amounted to approximately 35 points, an aging mechanism does not seem to be a means of increasing the strength to any great extent.

The microstructures of the aged samples of the commerical heat are presented in figures 7 and 8. This alloy, as was found for the laboratory heat D42, exhibited a slight precipitation at the grain boundaries when aged 48 hours at 1350°F. If the microstructures of the laboratory boron heat, presented in the First Progress Report, and the Heat A-5764 are compared for the 2150°F solution treated for 1 hour plus 24 hours age at 1350°F condition, it can be seen that much more excess phase is present in Heat D42 than the commercial heat. This condition may indicate either a dirty steel or that the differences in B and Ti contents were large enough to cause the difference in the amount of excess phase. It does not indicate differences in response to aging because the difference in the amount of excess phase also existed in the solution treated condition.

Response to Hot-cold Work

Work on the laboratory heat has shown that the high rupture strengths, considered characteristic of this alloy, are obtained by hot-cold working. For this reason work has been directed towards establishing the influence of temperature of hot-cold working on the rupture strength. Figure 9 shows the change in Brinell hardness of the Heat A-5764 due to 20 percent reductions at room temperature, 1000°, 1200°, and 1400°F. Two prior treatments before the cold or hot-cold work were used: a four-hour solution treatment at 1900°F; and a one-hour solution treatment at 2150°F. The curves show that:

(1) The higher solution temperature increased the response to working as measured by hardness; that is, the material which was solution treated at 2150°F was approximately 20 Brinell hardness points higher after either cold working or hot-cold working than the material which was prior processed at 1900°F.

(2) The increase in hardness on rolling at 1400°F, for the 1900°F solution condition, was substantially less than at the lower temperatures.

(3) Very little difference in hardness resulted from hot-cold working 20 percent at temperatures ranging from room temperature up to 1200°F after a 2150°F solution treatment.

(4) Within the conditions studied, the material solution treated at 1900°F indicated a slight maximum hardness response after a 1000°F hot-cold working treatment for 20-percent reduction.

In addition to the rolling conditions of figure 9, two specimens were subjected to treatments designed to produce minimum rupture-test strength.

This was done to obtain an indication as to where the boron might be when rupture strength was low. One specimen, after a solution treatment at 2150°F for 1 hour, followed by a reduction of 20 percent at 1200°F, was given a 100-hour age at 1500°F. The other, after the same initial treatment, was given a cyclic treatment consisting of: (1) a 5-percent reduction at 1800°F after being held at 1800°F for 1/2 hour, followed by (2) a 5-percent reduction at 1500°F after soaking at 1500°F for 4 hours. This treatment was repeated four times until a total of 40-percent reduction was obtained:

The following Brinell hardnesses were obtained:

100-hour age	-	195 Brinell
Cyclic treatment	-	181 BHN

Microstructures resulting from these treatments are presented in figure 10. The following was observed:

(1) The grain size following the 100-hour age remained normal, but the cyclic treatment produced fine grains.

(2) The precipitation resulting from the 100-hour age comes out on a definite line pattern within the grains.

(3) Particles precipitated by the cyclic treatment do not appear to follow crystallographic planes as they did after the aging treatment for 100 hours.

(4) If the microstructures of figure 10 are compared to any of the aging treatments tried at 1350°F (figs. 7 and 8), it will be noted that the precipitation in the latter case was wholly within the grain boundaries while the former exhibited precipitation within the grains also. Furthermore, the amount of precipitation shown in figure 10 is not great considering the treatment given these samples.

(5) The rupture properties of the 100-hour aged and cyclic rolled conditions are not sufficient yet to draw any conclusions.

Stress-Rupture Properties

Rupture data obtained for Heat A-5764 at 1200°F are given in Table I and figures 11, 12, and 13. Figure 11 indicates a slight superiority for the laboratory boron Heat D42 over the Heat A-5764 either as solution treated at 2150°F or as solution treated and aged.

Heat A-5764 had a 5,000 psi greater rupture strength for 100 hours at 1200°F when solution treated at 2150°F as compared to the solution treatment at 1900°F. This difference in strength at 1200°F becomes greater as hot-cold work is introduced reaching a maximum of 11,000 psi difference for a 20-percent reduction at 1000°F.

The higher solution treatment prior to hot-cold working resulted in very low rupture elongation properties as contrasted to the 1900°F treatment and the previous work done at 2150°F for the laboratory boron Heat D42:

Comparison of A-5764 and D42 Heats

Material	Treatment	100-hr. Rupture Strength, psi	Elongation, %
Lab. D42	S.T. 2150°F, 1 hr., W.Q.	44,000	33
"	S.T. 2150°F, 1 hr., W.Q. + 24 hr. age 1350°F	37,000	47
"	S.T. 2150°F, 1 hr., W.Q. + 40% reduction at 1300°F	(61,000)*	(20)*
"	S.T. 2150°F, 1 hr., W.Q. + 20% reduction at 1400°F	54,000	21
"	S.T. 2150°F, 1 hr., W.Q. + 40% reduction at 1400°F	56,000	16
Comm. Heat A-5764	S.T. 1900°F, 4 hr., W.Q. + 20% reduction at 1000°F	50,000	11
"	S.T. 2150°F, 1 hr. W.Q.	40,000	(30)
"	S.T. 2150°F, 1 hr. W.Q. + 24 hr. age 1350°F	34,000	(58)

Continued on next page

Comparison of A-5764 and D42 Heats (continued)

Material	Treatment	100-hr. Rupture Strength, psi	Elongation, %
Comm. Heat A-5764	S.T. 2150°F, 1 hr. W.Q. + 20% reduction at 1000°F	63,000	1
"	S.T. 2150°F, 1 hr. W.Q. + 20% reduction at 1200°F	56,000	3

* Values in parentheses are estimated

During the next period, the data will be analyzed more completely from the viewpoint of the reasons for the influence of composition and treatment on rupture properties.

X-Ray Studies

Lattice parameter measurements on several differently solution-treated samples are presented below. The measurements were made using a precision back reflection camera and utilizing Cohen's method of graphical extrapolation. The surfaces of the X-ray samples were given an electrolytic polish in a solution of 1/3 HCL to 2/3 glycerine at a current density of 8 amps/square inch for 1-1/2 hours to remove all traces of any cold work that might broaden the resulting X-ray lines. The following table summarizes the lattice parameter values obtained:

Material	Treatment	Lattice Parameter, A°
Non-boron Lab. Heat D41	1 hr., 2150°F, W.Q.	3.5867
0.03% Boron Lab. Heat D42	1 hr., 2150°F, W.Q.	3.5875
0.027% B, Heat A-5764	4 hrs., 1900°F, W.Q.	3.5897
0.027% B, Heat A-5764	1 hr., 2150°F, W.Q.	3.5877

These data are the averages of duplicate determinations and are believed to be reproducible. The first two entries in the above table show an increasing lattice parameter with the addition of boron; the two latter entries show a decrease in parameter with increasing solution temperature. If the boron atoms were going into solution interestitially, the lattice should expand with the addition of boron and with increased solution temperature; conversely, if the boron atoms are substitutional, the presence of the small boron atoms should result in a contraction of the lattice. The laboratory heats in the above table of parameters indicate that boron is interstitial, while the two conditions of heat treatment of the commercial heat suggest that boron is substitutional. However, the data are not conclusive in that the boron laboratory heat D42 contained 0.015 percent more carbon that the non-boron laboratory heat D41; it is possible that the carbon caused a lattice expansion which overshadowed the contraction effect of substitional boron. Furthermore, if boron is interstitial one would expect a considerable lattice expansion, even more per atom than for carbon, whereas substitutional boron might result in very little overall lattice contraction. Thus, further work is underway to resolve the problem.

FUTURE WORK

There are two general divisions to the work now in progress and planned for the immediate future. One concerns the fundamental role of boron in enhancing the properties of austenitic alloys and the other is a determination of the effect of processing variables on high temperature strength.

To determine the role of boron it is necessary to establish definitely whether boron enters the lattice interstitially or substitutionally. Six laboratory induction heats with different boron and titanium contents have been processed. The effect of different boron contents, both in the presence and absence of titanium, on the lattice parameter is being determined. Furthermore, heats are to be made with controlled nitrogen and

and oxygen contents. These heats will be used for further lattice parameter work and also for a determination of the effectiveness of boron additions, the maxiumum useful boron addition, and an evaluation of the character of minor phases present in the air melted material.

The determination of a suitable commercial heat treatment is in progress. Particular attention is being paid to the effect of the temperature of hot-cold working and of solution treating.

CONCLUSIONS

The data submitted in this report have been used to verify the trends observed on the laboratory boron heat. As yet the role of boron in enhancing the high-temperature properties of austenitic alloys is still obscure. However, the following statements can be made:

(1) Melting occurs in the grain boundaries of heats containing approximately 0.03 percent boron during a four hour treatment at 2200°F. This conclusion is based on both the laboratory heat D42 and the arc-furnace heat A-5764. Consequently the maximum safe solution temperature appears to be 2150°F. The arc-furnace heat contained somewhat less non-metallic stringers than did the induction heat; this is of importance since the boron melting point constituent formed preferentially near the stringers.

(2) Aging the boron heats results in only a slight increase in hardness. Rupture strengths at 1200°F are reduced and elongations in the rupture test increased by aging. It does not appear as if boron contributes to increased strength at high temperatures through a precipitation hardening reaction.

(3) Boron additions of approximately 0.03 percent increase the 100-hour rupture strength at 1200°F approximately 10,000 psi in the

solution treated condition. The differential is increased to as much as 17,500 psi by hot-cold working.

(4) Temperature of hot-cold working (for 20 percent reduction) has little effect on hardness from room temperature to 1200°F. Solution treating at 2150°F prior to hot-cold working results in somewhat higher hardness than solution treating at 1900°F. The hardness of solution-treated stock after hot-cold working falls off when the temperature of reduction is increased from 1200° to 1400°F.

(5) Maximum rupture strength at 1200°F resulted from hot-cold work at 1000°F for the conditions studied. Solution treating at 2150°F results in approximately 10,000 psi higher rupture strength for 100 hours at 1200°F than solution treating at 1900°F.

(6) Unlike the induction furnace heat D42, the arc-furnace heat A-5764 had low ductility in the rupture tests at 1200°F when hot-cold worked after a solution treatment at 2150°F.

(7) It has not yet been shown whether boron enters the austenitic lattice interstitially or substitutionally.

TABLE I

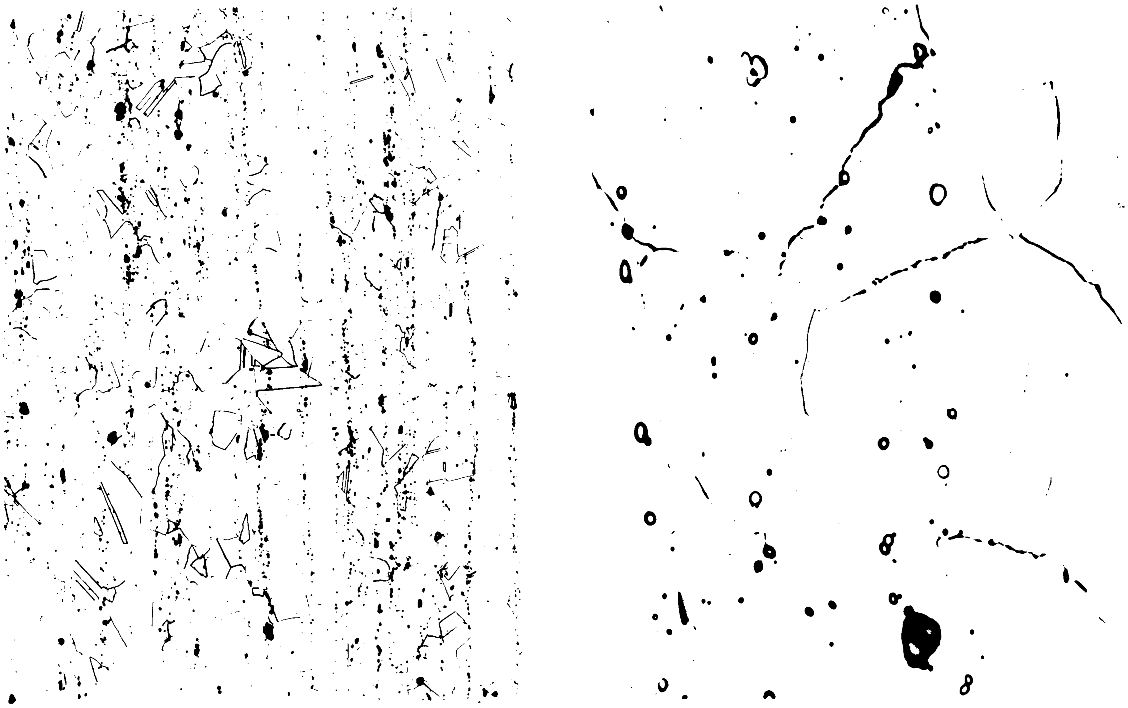
RUPTURE TEST DATA FROM TESTS AT 1200°F FOR ARC-FURNACE HEAT A-5764 CONTAINING 0.027% BORON

Heat Treatment	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)	Estimated 100-hr Rupture Strength (psi)	Estimated 100-hr Rupture ~~Strength~~ (%) Elong.
S.T. 2150°F 1 hr. W.Q.	40,000	83.1	38	53.7	40,000	-
S.T. 2150°F 1 hr. W.Q. Aged 12 hrs. at 1350°F	34,000	124.3	46	74.5	35,000	-
S.T. 2150°F 1 hr. W.Q. Aged 24 hrs. at 1350°F	34,000 40,000	111.6 ± 4.5 14.4 ± 12	57.6 61	75 73	34,000	58
S.T. 2150°F 1 hr. W.Q. Aged 48 hrs. at 1350°F	40,000 34,000	14.4 ± 12 86.5 ± 4.5	57.6 81	74.6 75	33,000	86
S.T. 2200°F 1 hr. W.Q. Aged 24 hrs. at 1350°F	40,000 34,000	26.2 229.5	55 44	75 72	36,000	51
S.T. 1900°F 4 hrs. W.Q.	35,000 40,000	76.5 ± 6.5 19.6	46 36	46 58.1	35,000	52
S.T. 1900°F 4 hrs. W.Q. Red. 20% at room temp.	50,000	33.9	3.9	30.2	43,000	3
S.T. 1900°F 4 hrs. W.Q. Red. 20% at 1000°F	47,000 54,000	233.3 40.7	5 14	15 31	50,000	11
S.T. 1900°F 4 hrs. W.Q. Red. 20% at 1200°F	47,000 54,000	133.9 23.5	6.9 23	25.6 29	48,000	12
S.T. 1900°F 4 hrs. W.Q. Red. 20% at 1400°F	44,000 47,000	98 59	17 20	38.6 41.6	44,000	17
S.T. 2150°F 1 hr. W.Q.	40,000	141.6	37	50	40,000	30
S.T. 2150°F 1 hr. W.Q. Red. 20% at room temp.	55,000 60,000	145.9 62.3	3 5	8 22	57,000	4

(concluded on the following page)

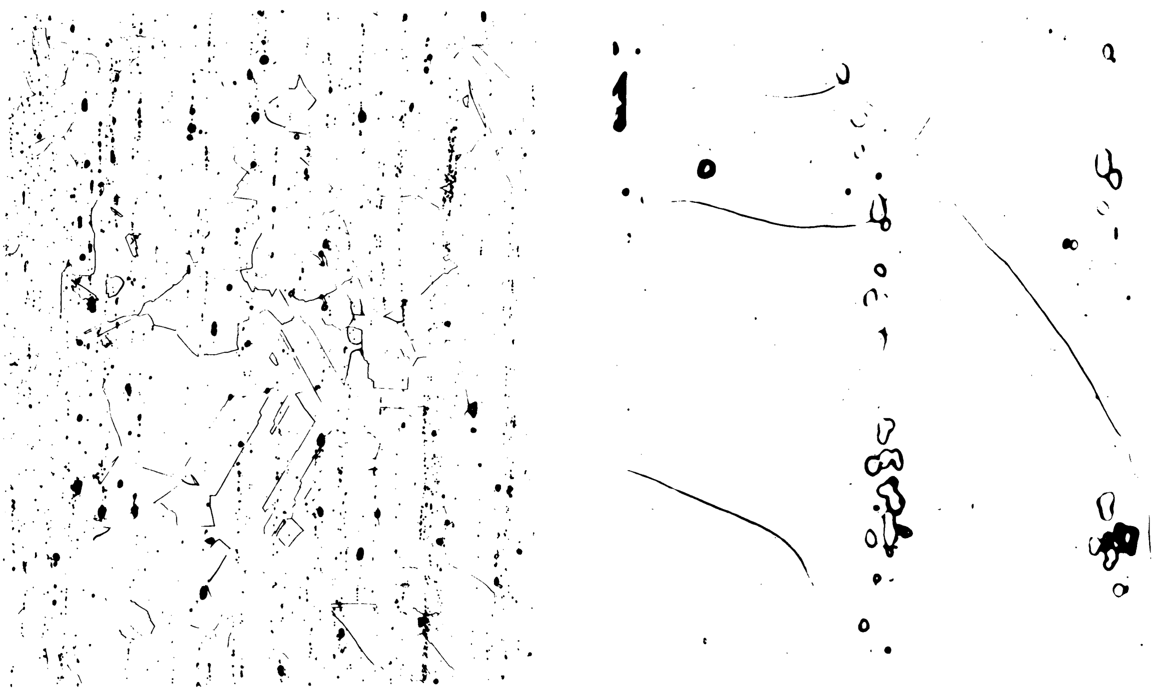
Table I - concluded

Heat Treatment	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)	Estimated 100-hr Rupture Strength (psi)	Estimated 100-hr Rupture Strength (%)
S.T. 2150°F 1 hr. W.Q. Red. 20% at 1000°F	55,000 60,000	156.6 119.7	10 2.9	11 8	63,000	1
S.T. 2150°F 1 hr. W.Q. Red. 20% at 1200°F	55,000 60,000	126.9 58	3 3	8.65 11	56,000	3
S.T. 2150°F 1 hr. W.Q. Red. 20% at 1200°F; Aged 100 hrs. at 1500°F	34,000	332.8	20	66	-	-



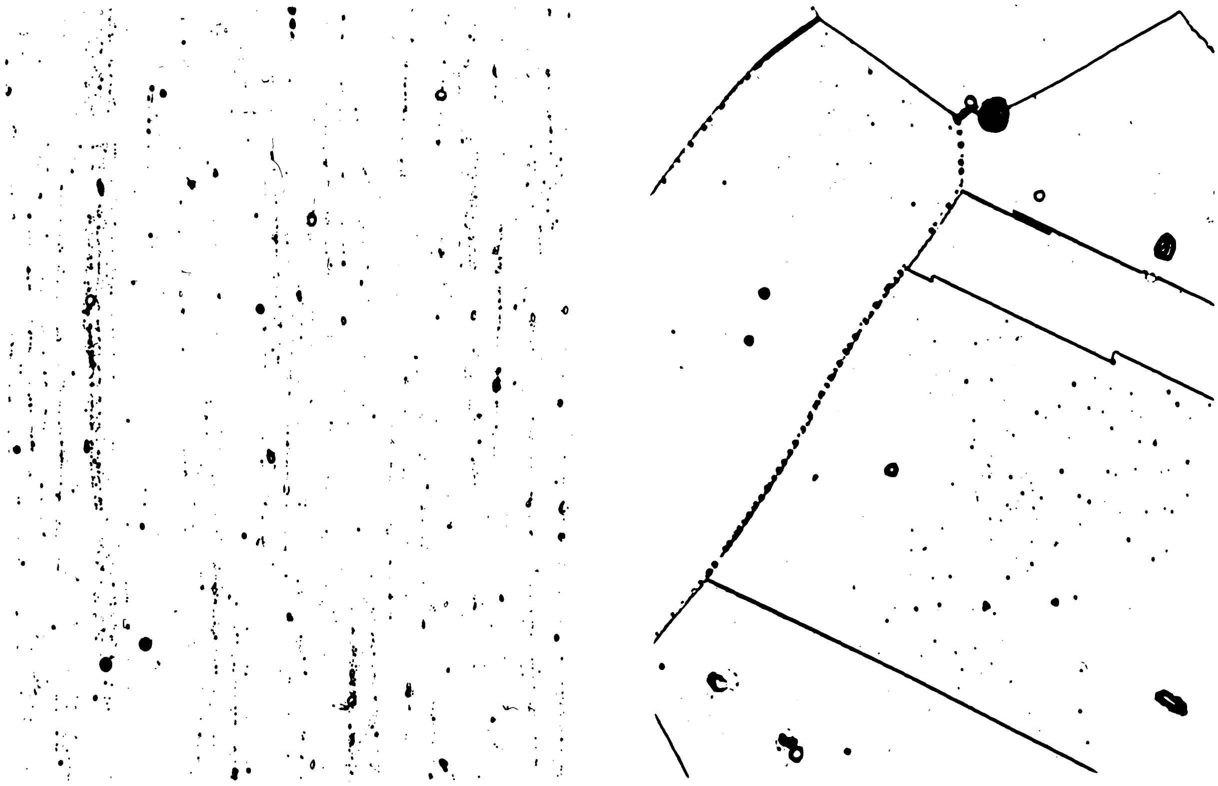
X100D X1000D

Figure 1 - Microstructure of Heat A-5764 Containing 0.027% Boron After Rolling at 2050°F to 7/8-inch Square Bars.



X100D X1000D

Figure 2 - Microstructure of Heat A-5764 After Solution Treatment at 2150°F for 1 Hour Followed by Water Quench.



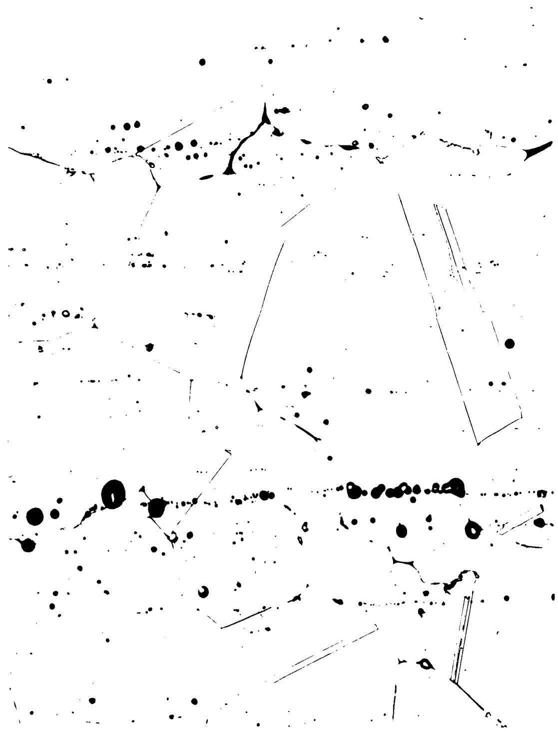
X100D X1000D

(a) Solution treated 1 hour at 2200°F, water quenched.

X100D X1000D

(b) Solution treated 4 hours at 2200°F, water quenched.

Figure 3 - Microstructure of Heat A-5764 After Solution Treatment at 2200°F.



X100

Figure 4 - Microstructure of Heat A-5764 Solution Treated 4 Hours at 2200°F, W.Q. Shows Tendency of Eutectic to Form in Regions of Stringers.

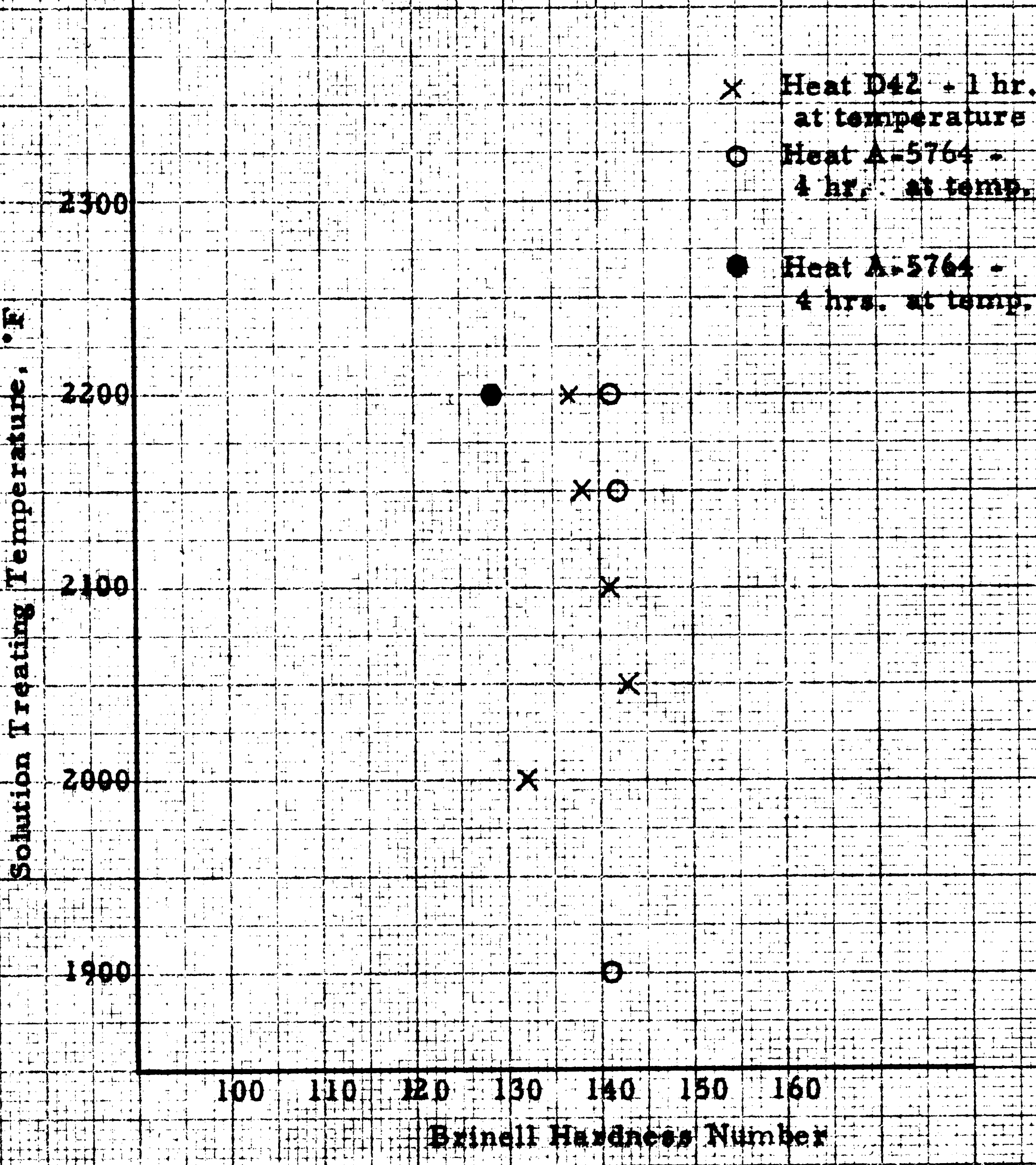


Figure 5 - Comparison of the Effect of Solution Treating Temperatures on Hardness of Laboratory Boron Heat D42 and Arc-Furnace Boron Heat A-5764. Samples were Water Quenched after being Held at the Indicated Temperature.

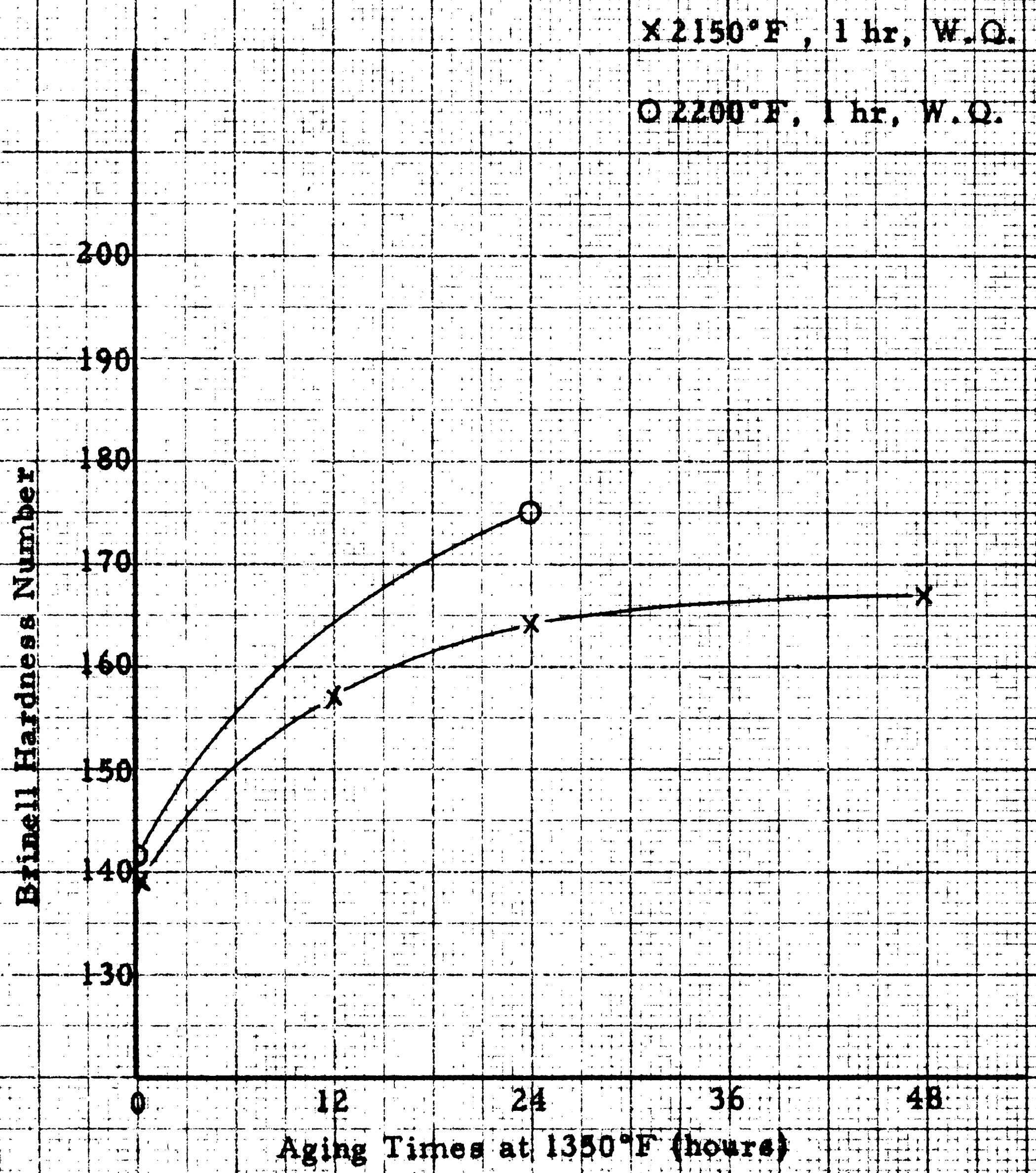
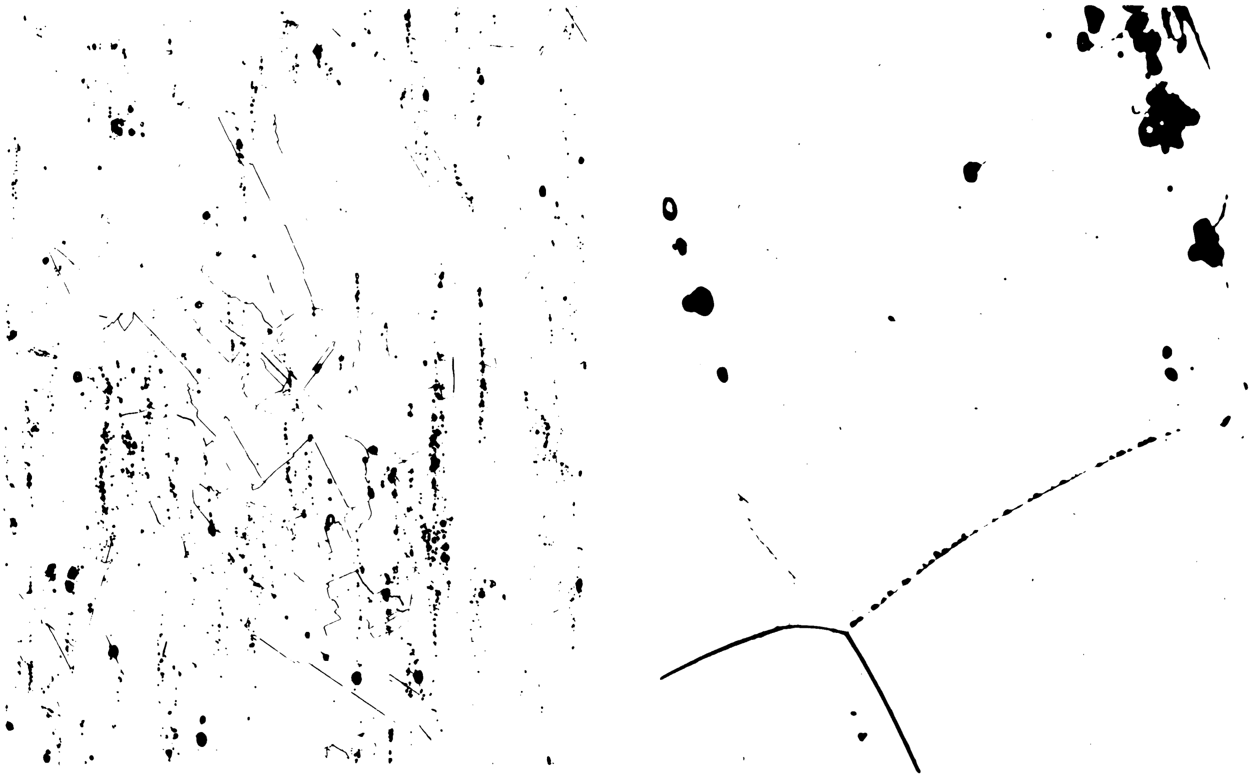
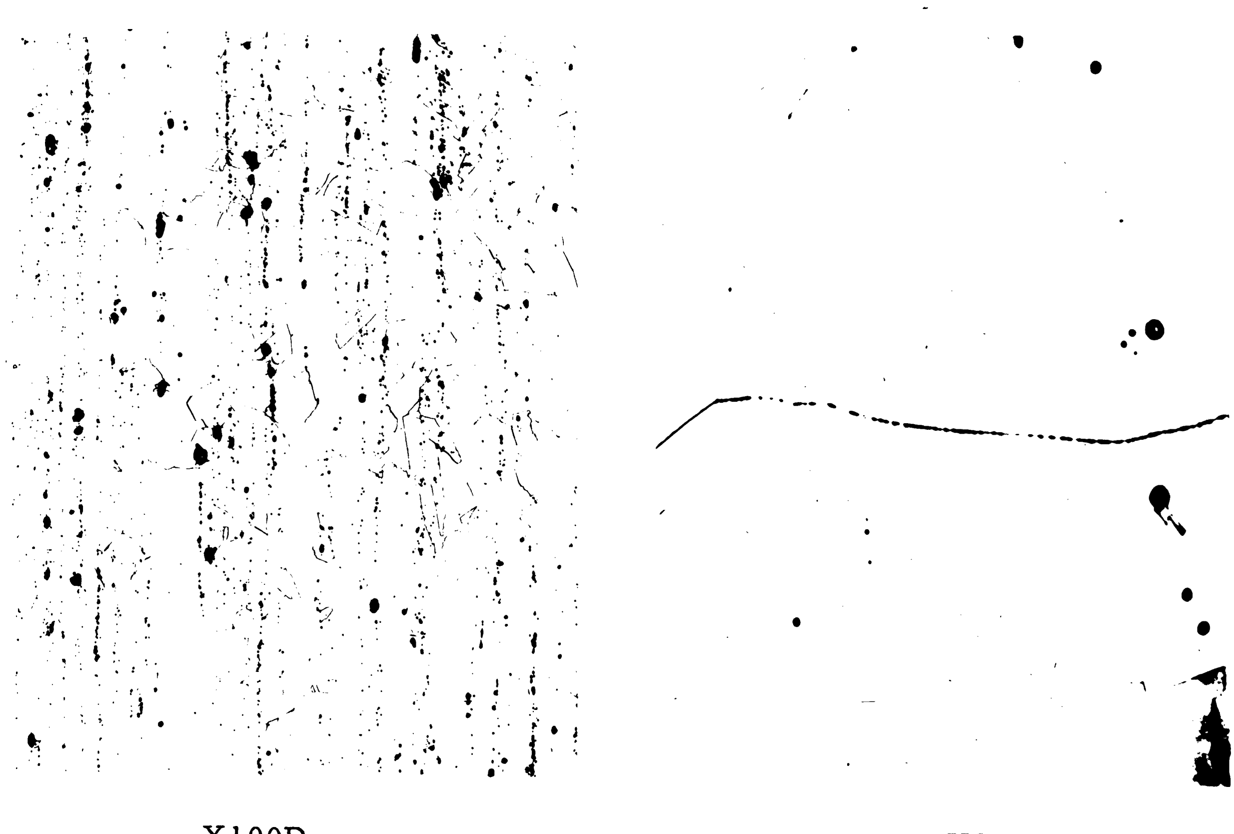


Figure 6 - The Effect of Aging Time at 1350°F on the Hardness of Heat A-5764.



X100D X1000D

Figure 7 - Microstructure of Heat A-5764 After Aging at 1350°F. Solution treated 1 hour at 2150°F, water quenched + aged 12 hours at 1350°F



X100D X1000D

Figure 8 - Microstructure of Heat A-5764 After Aging at 1350°F. Solution treated 1 hour at 2150°F, water quenched + aged 48 hours at 1350°F

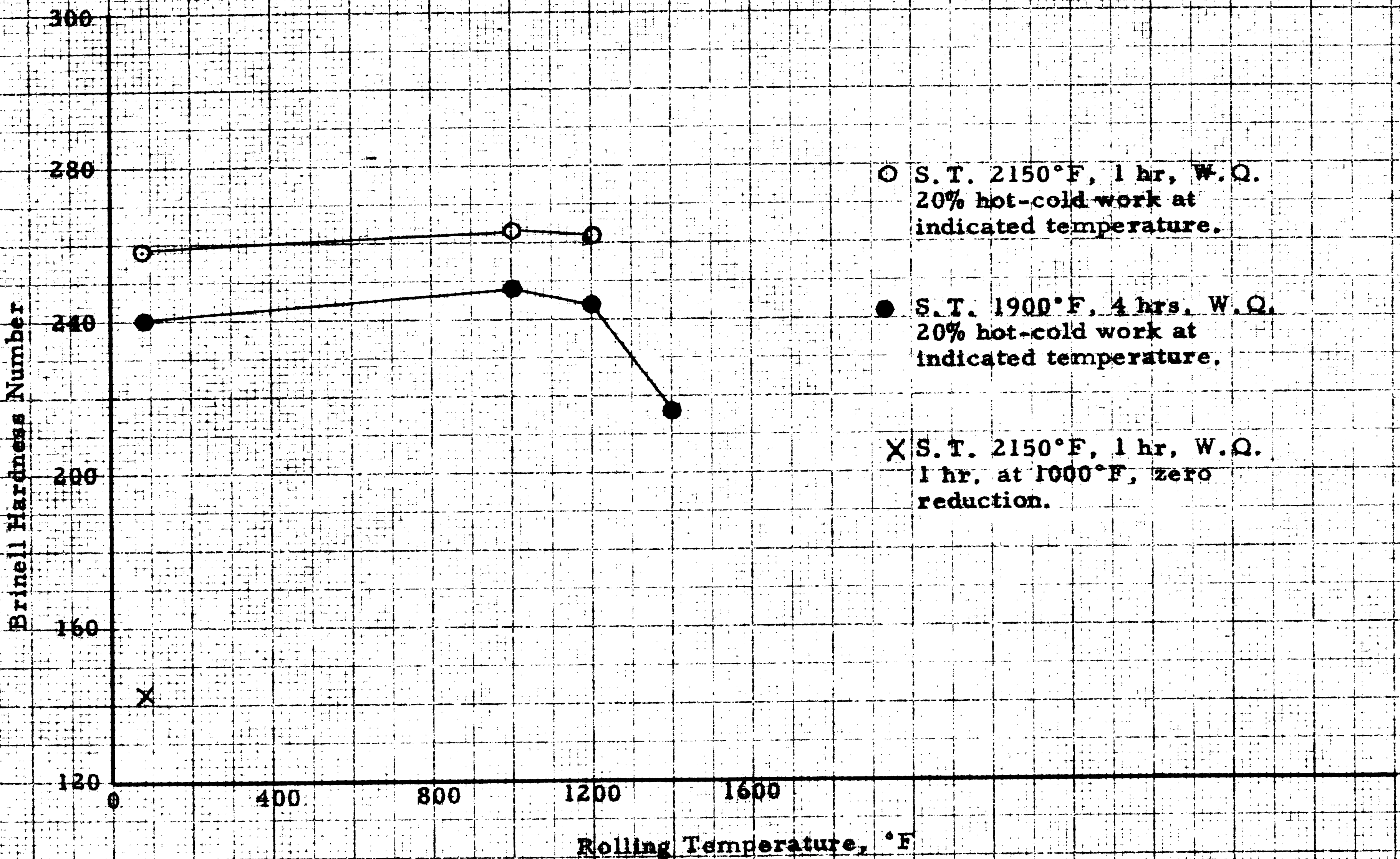
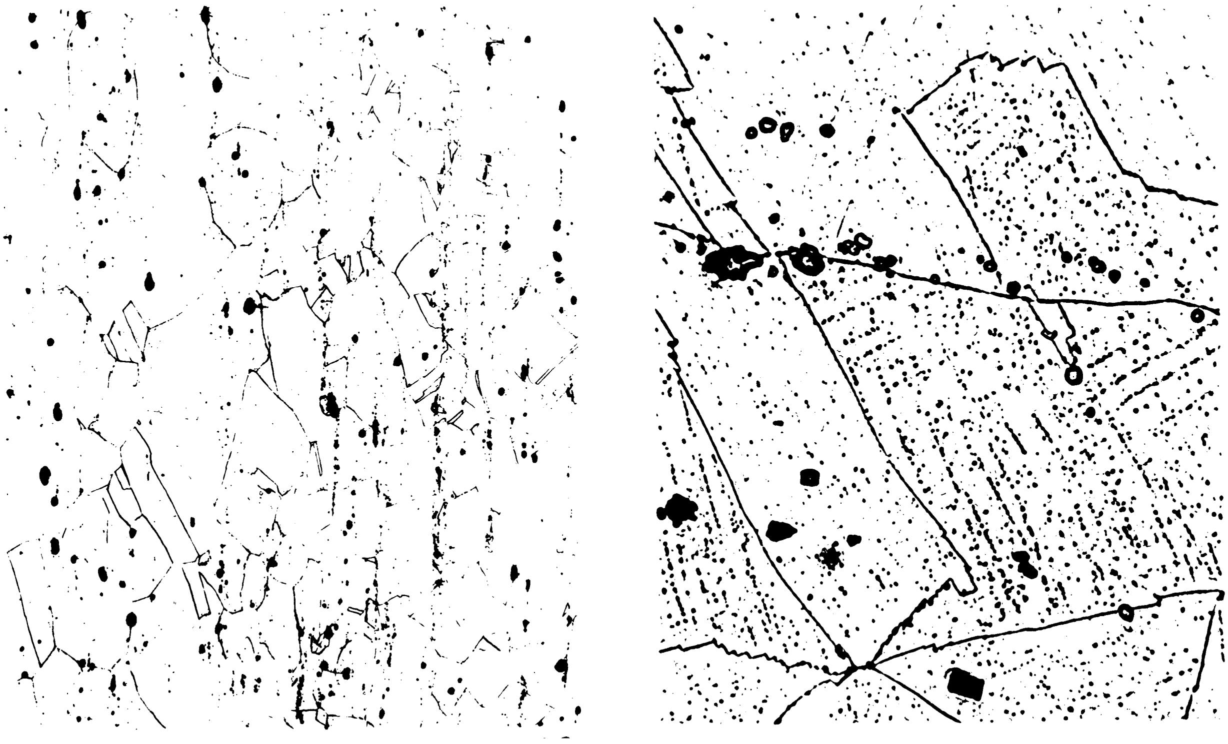
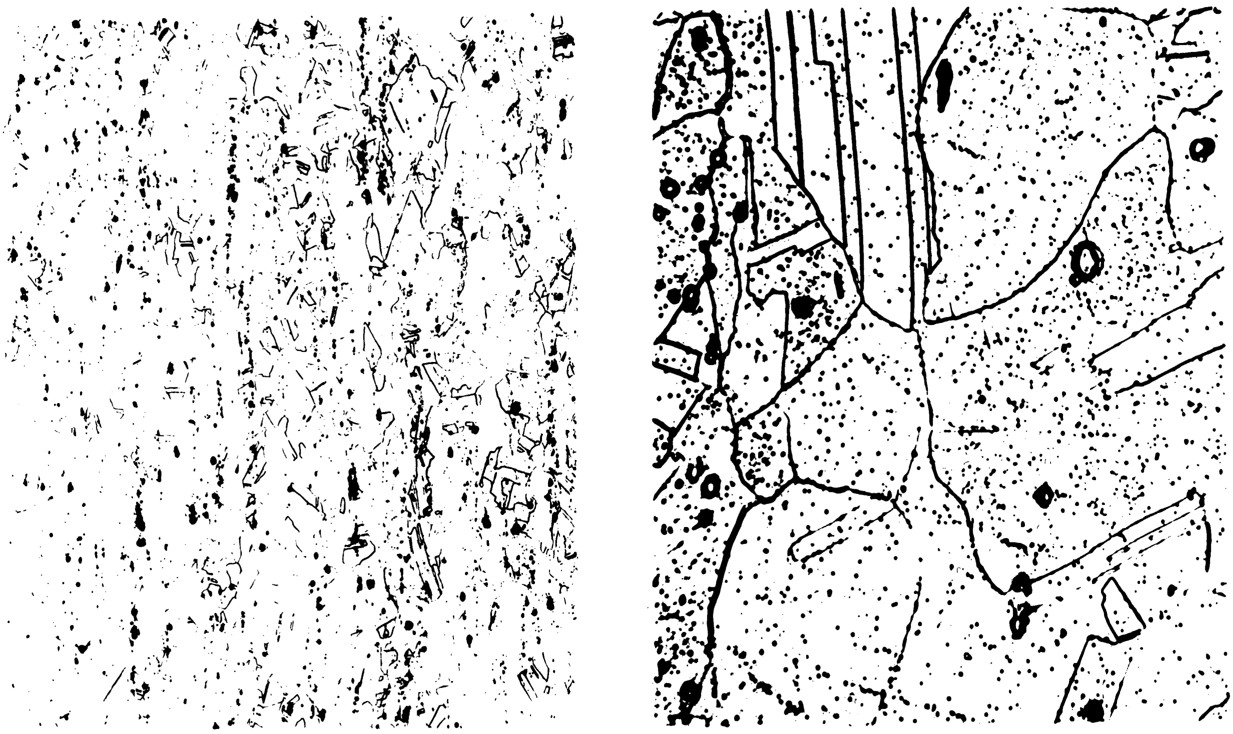


Figure 9 - Effect of Reductions of 20 Percent by Rolling at Room Temperature to 1400°F on Hardness of Heat A-5764.



X100D X1000D

(a) Solution treated 2150°F 1 hour, water quenched + 20 percent reduction at 1200°F + 100 hours age at 1500°F.



X100D X1000D

(b) Solution treated 2150°F 1 hour, water quenched, + 20 percent reduction at 1200°F + 1/2 hour at 1800°F and 5 percent reduction at 1800°F followed by 4 hours at 1500°F and 5 percent reduction at 1500°F; repeated four times for total of 40 percent reduction in the cycle treatment.

Figure 10 - Microstructure of Heat A-5764 After Two Treatments Designed to Produce Minimum Properties.

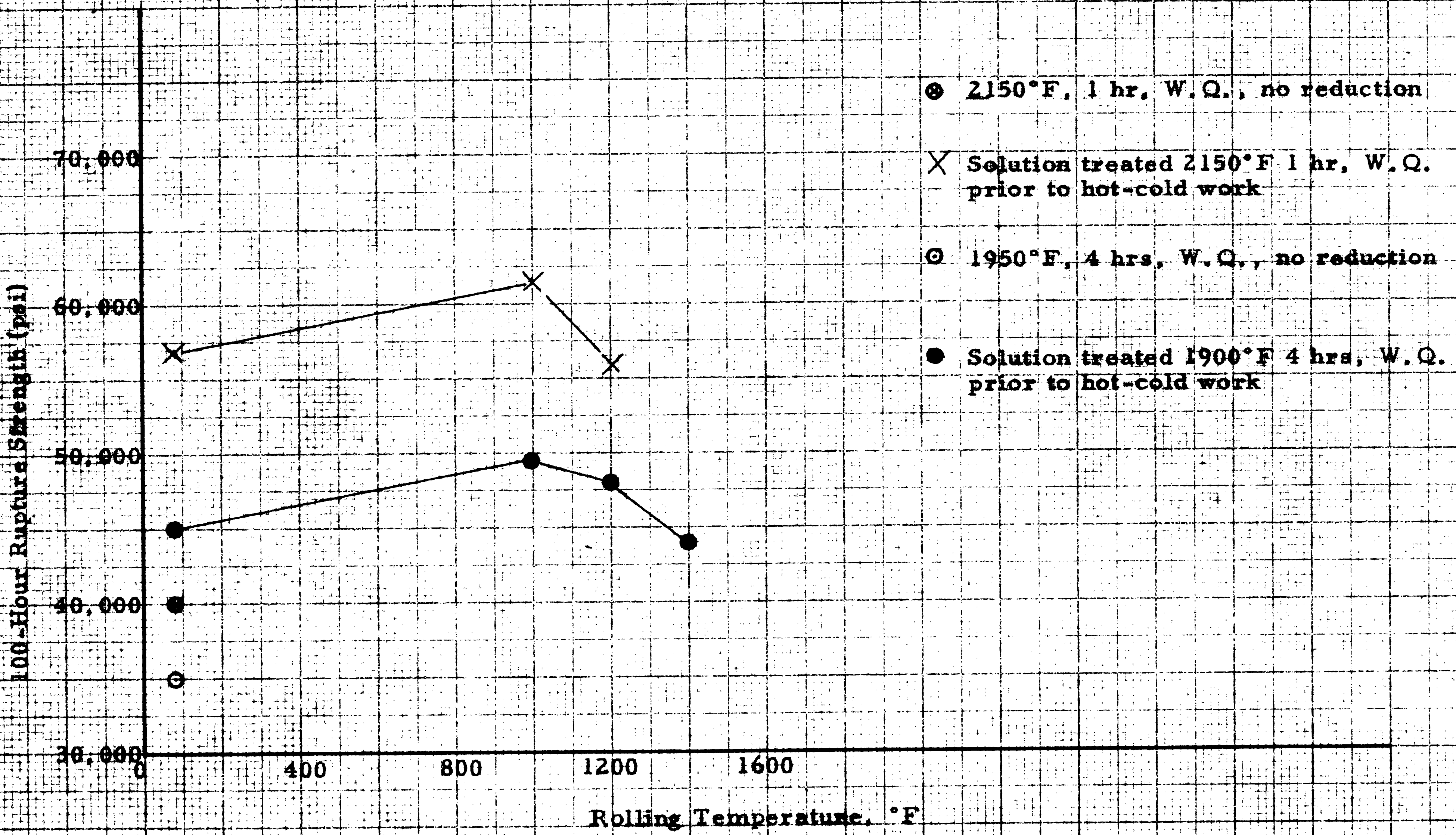


Figure 11 - Effect of Temperature of Hot-Cold Work by Rolling to 20 Percent Reduction, on the 100-Hour Rupture Strength at 1200°F of Heat A-5764

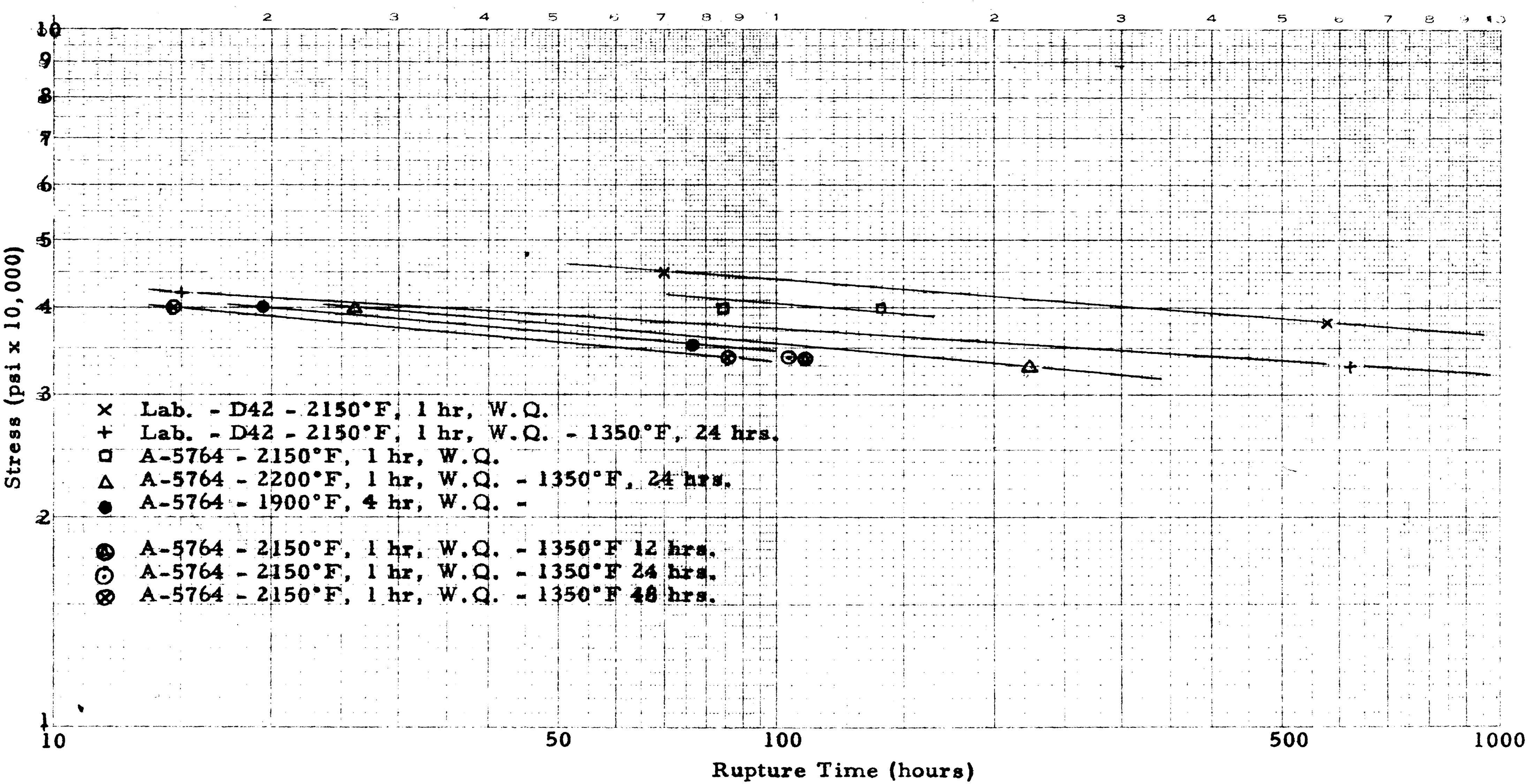


Figure 12 - Comparative Stress-Rupture Time Data at 1200°F for Boron Heat D42 and Haynes 88 Alloy in the Solution Treated and Aged Condition.

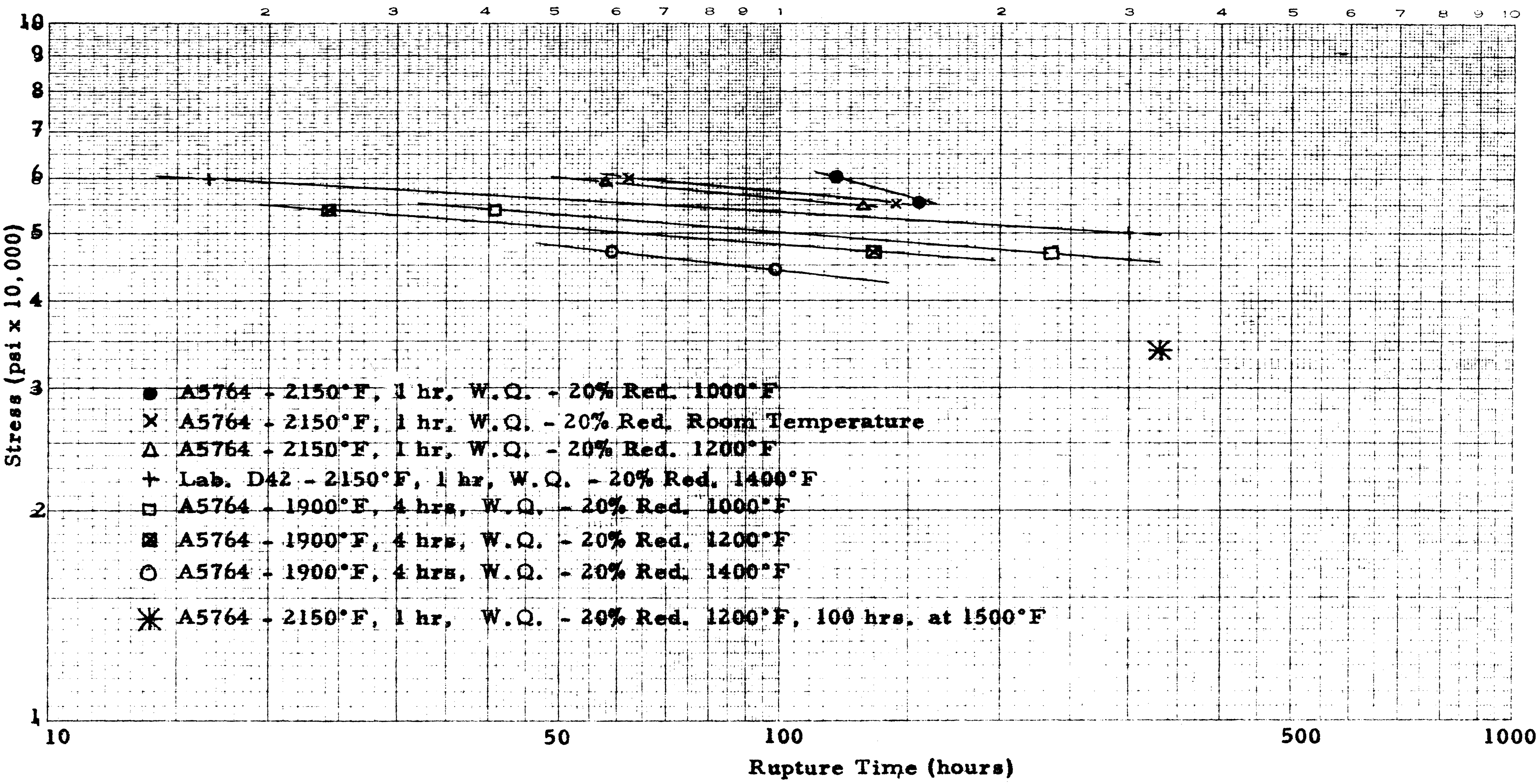


Figure 13 - Rupture Strengths at 1200°F of Titanium-Boron Heats A-5764 and D42 in Hot-Cold Worked Condition.