Engineering Research Institute
University of Michigan
Ann Arbor

Progress Report No. 3

(through April 30, 1953)

INVESTIGATION OF THE INFLUENCE OF TI-AL-B ON THE HIGH-TEMPERATURE PROPERTIES OF CR-NI-MO-FE AUSTENITIC ALLOYS

by

E. B. Mikus
C. L. Corey
J. W. Freeman

Project 2061

To

AERONAUTICAL RESEARCH LABORATORY (WCRRL)
RESEARCH DIVISION
WRIGHT AIR DEVELOPMENT CENTER
WRIGHT-PATTERSON AIR FORCE BASE, OHIO
Contract No. AF 33(616)-173
Expenditure Order No. R-463-8 BR-1

August, 1953

SUMMARY

This is the third quarterly progress report under Contract Number Af 33 (616)-173. The report extends the study of the mechanism by which boron increases the high-temperature properties of a lean austenitic alloy. The materials used in this phase of the investigation consisted of six laboratory induction furnace heats; they were prepared by making varying boron and titanium additions to a base composition of approximately 0.05 C, 13 Cr, 16 Ni, 2.5 Mo, 0.60 W and the balance iron. All analyses listed for the six heats in this report are "aimed," not chemical analyses.

The effect of solution treating temperature on microstructure, hardness, rupture strength, and lattice parameter was determined. Two solution treating temperatures, 1900° and 2150°F, were used.

The higher solution temperature had little effect on grain size of all heats except Heat 201, for which the grain size increased from A.S.T.M. No. 5 to No. 4. The amount of excess phases present increased with increasing amounts of boron and remained insoluble at the higher solution temperature. Higher hardnesses and greater rupture strengths generally resulted from the higher solution temperature, although there were exceptions, notably in the 0% titanium heats. In these heats the 1900°F solution treatment produced as good or better strengths than the 2150°F solution treatment.

Alloys with higher boron contents gave generally higher hardnesses and rupture strengths and smaller lattice parameters. A boron saturation effect was observed in the titanium bearing heats at 0.03% boron.

Beyond 0.03% boron the titanium heats exhibited a decrease in rupture strength, whereas the titanium free heats continued to increase in strength beyond 0.03% boron, but at a less rapid rate.

It is not possible to state at this time whether boron enters the austenitic lattice substitutionally or interstitially. While all of the consistent alloy families considered showed a decreased lattice parameter with increasing boron content, it is possible that part or all of the reduction was due to the removal from solution of known interstitial atoms.

INTRODUCTION

This is the third quarterly progress report covering work done through 30 April 1953 under Contract Number AF 33(616)-173, Expenditure Order Number R-463-8 BR-1.

It had previously been decided that alloys covering a range of boron content, both with and without titanium to act as a scavenger, would be essential to an adequate investigation of the role of boron in producing high rupture strength in an austenitic alloy. The preparation of six such alloys was reported previously, the present progress report covers the initial stages of the work done on these materials. A later report will cover the remainder of the work on the six heats and will summarize the investigation and present the general conclusions.

GENERAL PROCEDURE

The initial phase of the work on the six alloy modifications consisted of a determination of:

1. The response of the various alloys to solution treatment, treatments of four hours at 1900°F and one hour at 2150°F being employed. Brinell hardness measurements, supplemented by microstructures, were used to indicate the response.

2. The rupture strength at 1200°F of the materials as a function of boron content both with and without titanium, and as a function of solution treatment.

3. The influence of boron content on the lattice parameter both with and without titanium scavenging. It was also to be determined whether the parameters of the various alloys were affected by heat treatments, and, if so, in what manner.

TEST MATERIALS

Six 15-pound heats were made with varying boron and titanium contents in a laboratory induction furnace. Ferro-alloys were used for making additions, and, in the case of boron and titanium, additions were calculated on the basis that 70% of the alloy added would be lost during melting. This practice was based on experience with two previous induction heats made in the laboratory of a commercial producer. The titanium and boron were added in that order after final deoxidation the the heats were poured immediately. The ingots, after grinding, were forged from 2100°F into 1-1/2 inch squares and were rolled from 2050°F to 7/8 inch square bars. No difficulty was encounted in processing any of the heats.

The heat numbers and aimed percentages of boron and titanium are given below for the six alloys, together with the analyses of Heats D41 and D42 which were used as compositional patterns in the melting of these six induction heats.

Heat Number	Aimed Percentage %B	Aimed Percentage %Ti
201	0.01	0
202	0.10	0
203	0.005	0.60
204	0.10	0.60
205	0.03	0
206	0.03	1.5

Heat Number	C	Mn	Si	Cr	Ni	Mo	W	B	Ti	Fe
D41	0.045	1.33	0.32	13.69	15.03	2.02	0.61	0	0.60	Bal.
D42	0.060	1.25	0.33	13.20	15.03	2.04	0.64	0.03	0.51	Bal.

Two solution treating temperatures were employed, one to produce maximum solution of boron and titanium and the other to produce relatively incomplete solution of these alloy additions. For maximum solution, a limiting temperature of 2150°F was imposed by the formation of a low melting point eutectic at 2200°F. A temperature of 1900°F was chosen to produce a condition of incomplete solution of boron and titanium.

RESULTS

The microstructures, hardness, rupture strengths and lattice parameters of the six heats were compared after solution treating at 1900° and 2150°F.

Microstructures

The following observations were made from the photomicrographs of these alloys:

1. Grain sizes for the six alloy modifications ranged from A.S.T.M. No. 4 to A.S.T.M. No. 6. Heat 201, containing very little boron and no titanium, exhibited the largest grain size; whereas the grain sizes of the alloys containing greater boron contents were smaller. The grain size of all the alloys except Heat 201 remained unaffected when the solution temperature was raised from 1900° to 2150°F. For Heat 201 a solution temperature of 2150°F increased the grain size from A.S.T.M. No. 5 to A.S.T.M. No. 4. This slight increase in grain size is not in keeping with that of standard 18-8 types of austenitic alloys; A.I.S.I. Type 321, for example, would coarsen noticeably at 2150°F. The slight increase in grain size observed for Heat 201 may be due either to a very strong grain growth inhibiting effect of small amounts of boron or to the fact that induction heats are often quite fine grained compared to electric furnace heats.

2. The addition of titanium did not appear to result in nearly as much secondary phase as did the addition of boron. The 0.10% boron

and the 0.10% boron - 0.60% titanium alloys contain, for example, about the same amount of secondary phase and both contain considerably more than the 0.03% boron modification (see figures 2, 3 and 4).

3. Secondary phases appeared to be unaffected by the high temperature solution treatment.

4. Micro-segregation was evident in heats containing high boron and high boron and titanium (see figures 2, 3 and 4).

Hardness Response

A comparison of the hardness of these heats in the 1900° and 2150°F solution treated conditions is shown in figure 5 as a function of boron content; values for D41 and D42 were included for completeness. Nearly parallel results were obtained from the two solution treatments. An increase in hardness was obtained with increasing boron contents with and without titanium. The increase in hardness obtained by the boron additions may be due to:

1. The strengthening of the matrix due to the increasing amount of boron in solution, or

2. The formation of increasing amounts of secondary phase or phases resulted in an apparent hardness increase.

Rupture Properties

Rupture data at 1200°F obtained for these six heats in two solution treated conditions are presented in Table I and figures 6 and 7. The heats increased in 100-hour strengths from 23,000 to 44,000 psi due to a combination of alloying and heat treatment. Up to 0.03%

boron a contribution to strength was obtained either in the presence or absence of titanium; the strength level for the titanium-bearing modifications was, however, higher than for the straight boron-alloys. Larger boron additions in the presence of titanium appeared to contribute no further increase in rupture strength; whereas, in the absence of titanium higher boron additions continued to increase the rupture strength. Further verification of this trend is needed to establish whether or not this apparent saturation value of boron is real.

The titanium-boron alloys were all improved by the 2150°F solution treatment, the 100-hour strength being improved by as much as 5,000 psi over that obtained with a 1900°F solution treatment. Thus, the 0.10% B - 0% Ti alloy and the 0.03% B - 1.5% Ti alloy had nearly the same 100-hour strengths, 37,500 and 39,000 psi respectively, solution treated at 1900°F; but these two values became 38,000 and 44,000 psi respectively for material solution treated at 2150°F. The straight boron alloy of 0.03% boron was improved by the higher solution treatment also; but, the other straight boron alloys were either not improved or reduced in strength.

The rupture elongations for all the alloys were very good. As the boron and or boron and titanium contents increased, the elongations generally tended to increase also.

Lattice Parameters

Lattice parameters were determined on solution treated samples. The same technique was employed as outlined in the Second Progress Report. Table II lists the measured parameters; the data are graphically presented in figure 8.

An evaluation of the reproducibility of the parameter values has been in progress throughout the investigation. The general technique and camera characteristics were checked using a very pure aluminum standard; the results indicated a precision of ± 0.0003 kX. However, checks on the alloy samples are less reproducible, being ± 0.0006 kX, because of:

(a) absence of a good set of back reflection lines, such as are obtainable with aluminum; and

(b) non-homogeneity of sample material.

A unique answer to the question of the direction of the effect, contraction or exapnsion, of boron on the austenitic lattice is not permissible from an evaluation of the data. The addition of boron resulted in a contraction of the lattice for any of the groupings of the various alloys, indicating substitutional boron. However, when the values of Heats D41 and D42 are considered (indicated by a dashed line in figure 8) this consistency apparently no longer holds true. The discrepancy may be due to the following:

1. Sufficient chemical variation exists between Heats D41, D42 and the 200 series heats to account for the lattice parameter variations.

2. The melting practice of the heats is different, which would influence the amounts of small sized atoms in solution.

Considering just the 200 series heats, a tendency towards substitutional boron exists. But, there are many unanswered questions:

1. Why do two of the three points for the 0% Ti, solution treated at the lower temperature, fall below the line for the higher treatment temperature? If substitutional boron is producing a contraction of

the lattice, one would expect the line for samples solution treated less completely (at 1900°F) to fall above that for samples treated at a temperature (2150°F) permitting more complete solution of soluble boron.

2. How much of the lattice parameter reduction is due to removal of interstitial carbon and nitrogen atoms from solution or large size atoms such as molybdenum or tungsten?

3. To what extent does the solution of titanium account for the very high parameter values obtained for the 1.5% Ti alloy?

These and other questions must be answered before a complete understanding of the role of boron is possible. Further, as was pointed out previously, the compositions given in this report are "aimed" analyses and not chemical analyses.

FUTURE WORK

Work covered by the next progress report will conclude the data which was originally outlined for these six tests. This work will consider the effect of aging and hot-cold working on the properties of the alloy modifications. Complete chemical analyses will be presented at that time.

Arrangements are being made for vacuum melting equipment which will be available for this work. It is expected that better control of composition by such melting will enable development of clearer answers to the role of boron.

CONCLUSIONS

Microstructural studies indicate the possibility of boron acting as a grain growth restrainer. The grain sizes of these heats were unaltered by solution treatments of 1900° and 2150°F. Increasing boron contents in the presence or absence of titanium resulted in large amounts of excess phases which exhibited no solubility even at 2150°F. In heats containing high boron or high boron and titanium, micro-segregation was evident.

The hardness response to two solution treatments of 1900° and 2150°F was nearly parallel. Hardness increased as the boron was increased either in the presence or absence of titanium, although a greater rate of hardening was evident up to 0.03% boron.

Rupture data have shown that up to 0.03%, boron has a beneficial effect on the high temperature strength of austenitic alloys either in the presence or absence of titanium. Higher boron additions in the presence of titanium did not contribute to further increases in strength; whereas, in the absence of titanium further boron additions did increase strength. Solution treating at 2150°F improved the strength of boron-titanium alloys over that obtained from the 1900°F solution treatment. However, in the absence of titanium, little or no increase in strength resulted from the higher solution temperature. Rupture elongation for all the alloys was remarkably good.

Lattice parameter studies have suggested that boron exerts its influence on the strength of these austenitic alloys through a solid solution mechanism. However, further work needs to be done before anything definite can be concluded. If the reduction in lattice parameter which was observed was not due to substitutional boron, but rather to the removal of odd sized atoms from solution by boron, then the small amount of boron which may be dissolved interstitially must be very effective in promoting high temperature strength. It would have to account for the observed increase in strength, as well as to counteract the weakening effect which would result from the removal of odd size atoms from solution.

TABLE I

RUPTURE TEST DATA FROM TESTS AT 1200°F FOR SIX LABORATORY INDUCTION HEATS 201-206 WITH VARYING BORON AND TITANIUM CONTENTS

Heat Number and Heat Treatment	Aimed Per Cent		Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)	Estimated 100-hr Rupture Strength (psi)	Est. 100-hr Rupture Elong (%)
	B	Ti						
201 - S.T. 1900°F- 4 hrs W.Q.	0.01	0	24,000 27,000	130.4 54.8	20.8 20.0	15 20	25,000	20
201 - S.T. 2150°F- 1 hr W.Q.	0.01	0	21,000 25,000	227.9 49.5	19 19	18.5 12	23,000	19
202 - S.T. 1900°F- 4 hrs W.Q.	0.10	0	35,000 41,000	341 18.1	43 40	65 38	38,000	41
202 - S.T. 2150°F- 1 hr W.Q.	0.10	0	33,000 40,000	427 56.4	42 36	67 46	38,000	37
203- S.T. 1900°F- 4 hrs W.Q.	0.005	0.60	31,500 35,000	139.7 65.0	49 51	65 67	33,000	50
203 - S.T. 2150°F- 1 hr W.Q.	0.005	0.60	30,000 40,000	769 33.4	40 65	45 68	36,000	60
204 - S.T. 1900°F- 4 hrs W.Q.	0.10	0.60	35,000 40,000	227* 69.8	-- 43.6	-- 50	39,000	--
204 - S.T. 2150°F- 1 hr W.Q.	0.10	0.60	40,000 45,000	139.9 68.0	33 42	64 51	42,000	38
205 - S.T. 1900°F- 4 hrs W.Q.	0.03	0	28,000 32,000	233 54.9	49 32	45 37	30,000	38

TABLE I, Continued

Heat Number and Heat Treatment	Aimed Per Cent		Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)	Estimated 100-hr Rupture Strength (psi)	Est. 100-hr Rupture Elong. (%)
	B	Ti						
205 - S.T. 2150°F-1 hr W.Q.	0.03	0	35,000	58.2	33	32	33,000	31
			40,000	13.3	35	29		
206 - S.T. 1900°F - 4 hrs W.Q.	0.03	1.5	37,000	330	35	36	39,000	47
			45,000	3.4	52	61		
206 - S.T. 2150°F - 1 hr W.Q.	0.03	1.5	40,000	862.5	23.6	23	44,000	52
			50,000	6.3	56.7	55		

* Specimen in third-stage creep when furnace failed.

TABLE II

LATTICE PARAMETER VALUES OBTAINED ON SIX INDUCTION HEATS WITH VARYING BORON AND TITANIUM CONTENTS IN TWO SOLUTION TREATED CONDITIONS

Heat Number	Heat Treatment	Aimed Per Cent		Lattice Parameter (kx Units)
		B	Ti	
201	S.T. 1900°F - 4 hrs W.Q.	0.01	0	3.5857
201	S.T. 2150°F - 1 hr W.Q.	0.01	0	3.5871
202	S.T. 1900°F - 4 hrs W.Q.	0.10	0	3.5840
202	S.T. 2150°F - 1 hr W.Q.	0.10	0	3.5836
203	S.T. 1900°F - 4 hrs W.Q.	0.005	0.60	3.5863
203	S.T. 2150°F - 1 hr W.Q.	0.005	0.60	3.5838
204	S.T. 1900°F - 4 hrs W.Q.	0.10	0.60	3.5817
204	S.T. 2150°F - 1 hr W.Q.	0.10	0.60	3.5833
205	S.T. 1900°F - 4 hrs W.Q.	0.03	0	3.5849
205	S.T. 2150°F - 1 hr W.Q.	0.03	0	3.5861
206	S.T. 1900°F - 4 hrs W.Q.	0.03	1.5	3.5890
206	S.T. 2150°F - 1 hr W.Q.	0.03	1.5	3.5898

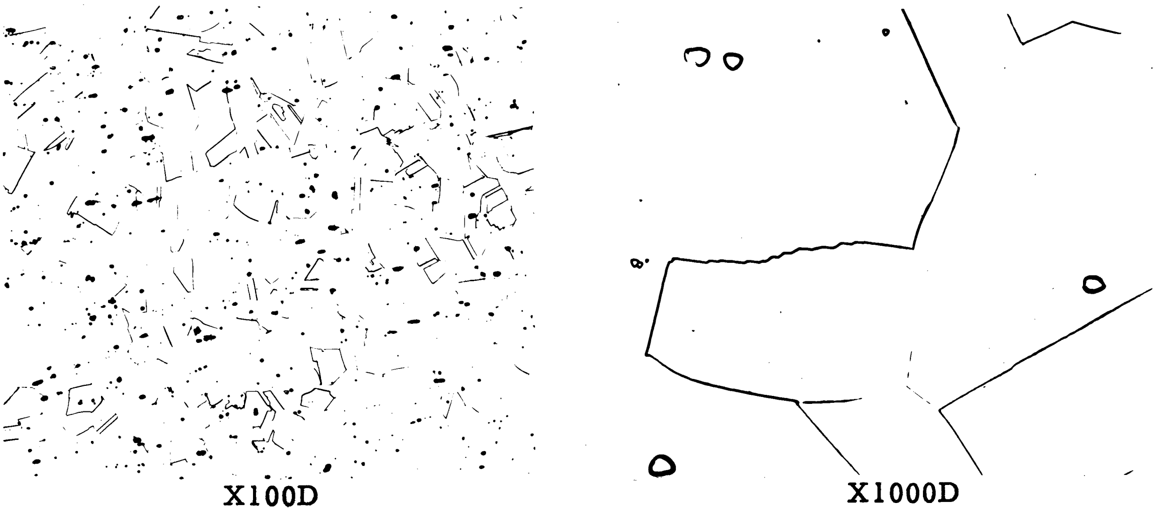
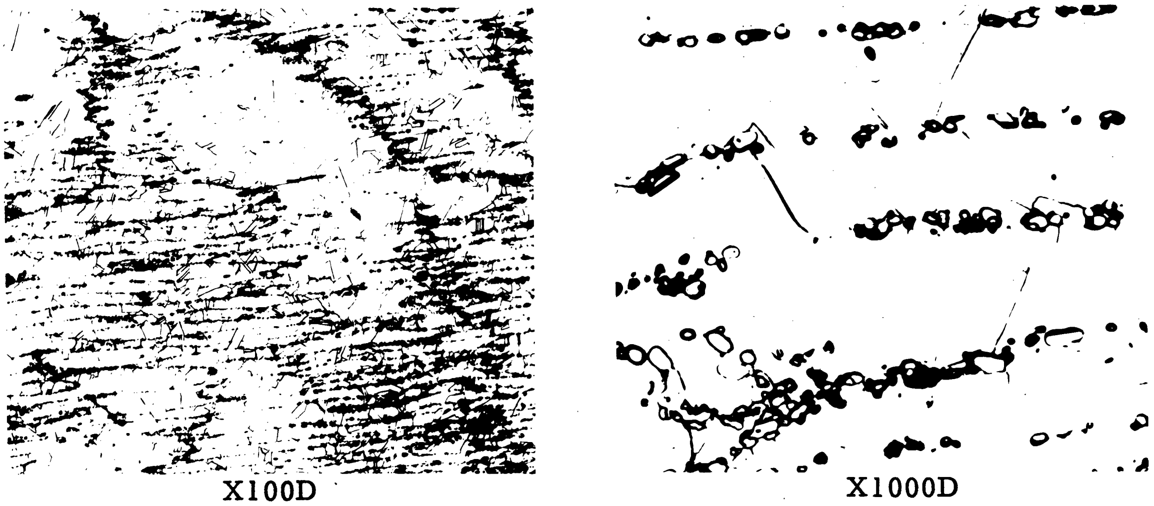
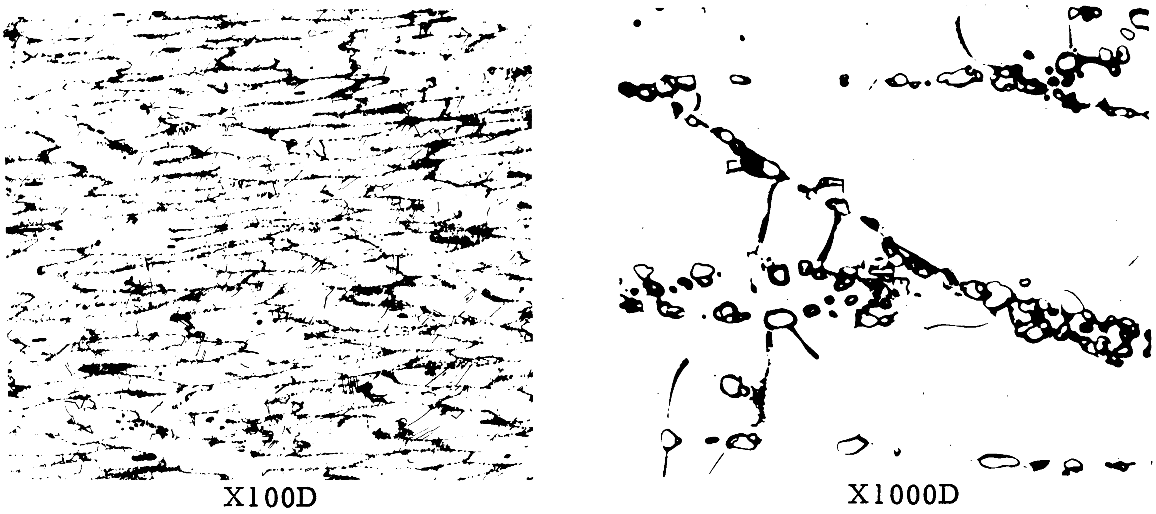


Figure 1 - Microstructure of Heat 201 Solution Treated 4 Hours at 1900°F, Aim Composition 0.01% B, 0% Ti.

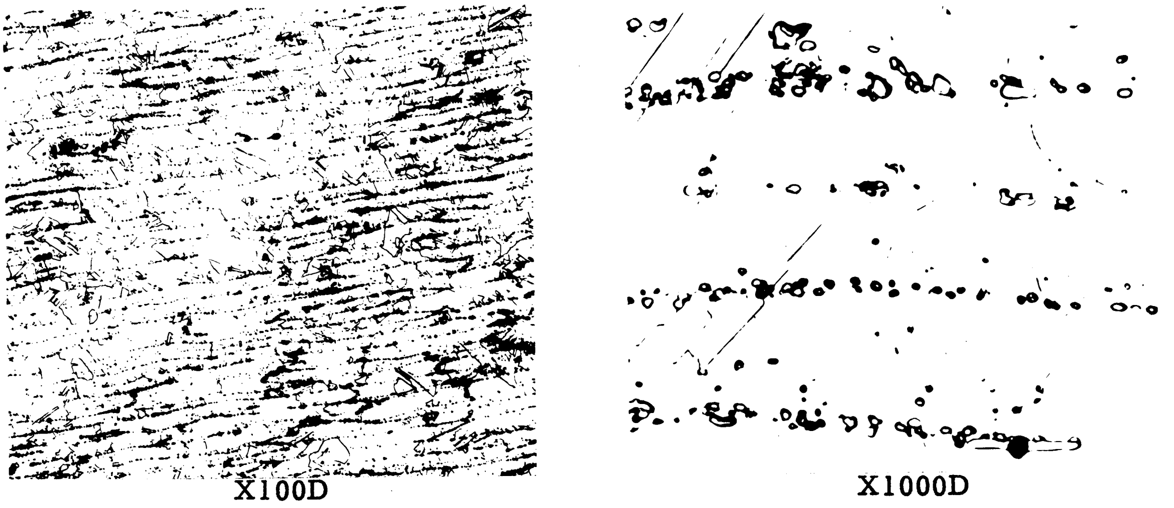


(a) Solution treated 4 hours at 1900°F, water quenched.

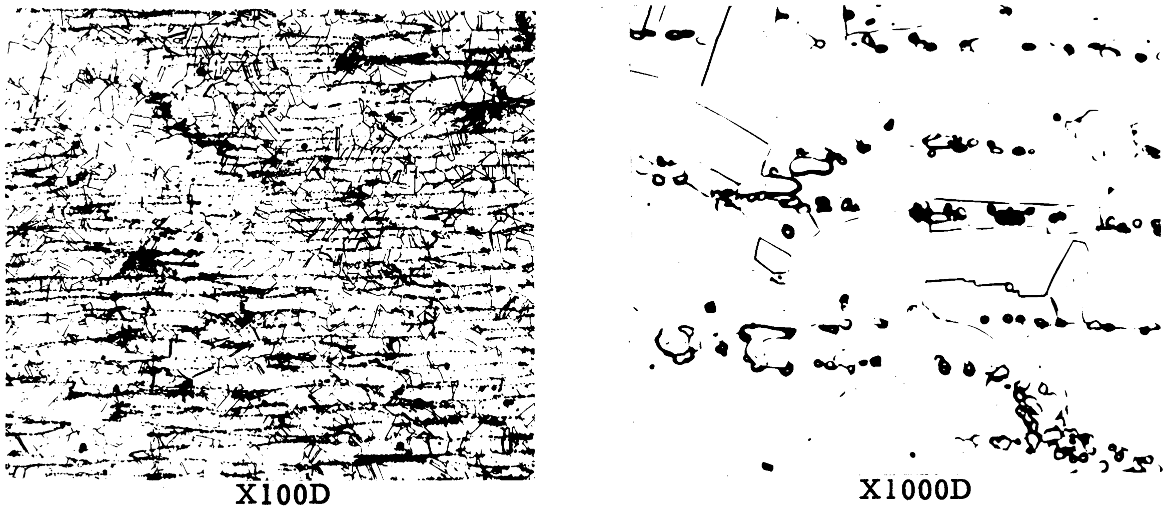


(b) Solution treated 1 hour at 2150°F, water quenched.

Figure 2 - Microstructure of Heat 202, Aim Composition 0.10% B, 0% Ti, in Two Solution-Treated Conditions.



(a) Solution treated 4 hours at 1900°F, water quenched.



(b) Solution treated 1 hour at 2150°F, water quenched.

Figure 3 - Microstructure of Heat 204, Aim Composition 0.10% B, 0.60% Ti, in Two Solution-Treated Conditions.

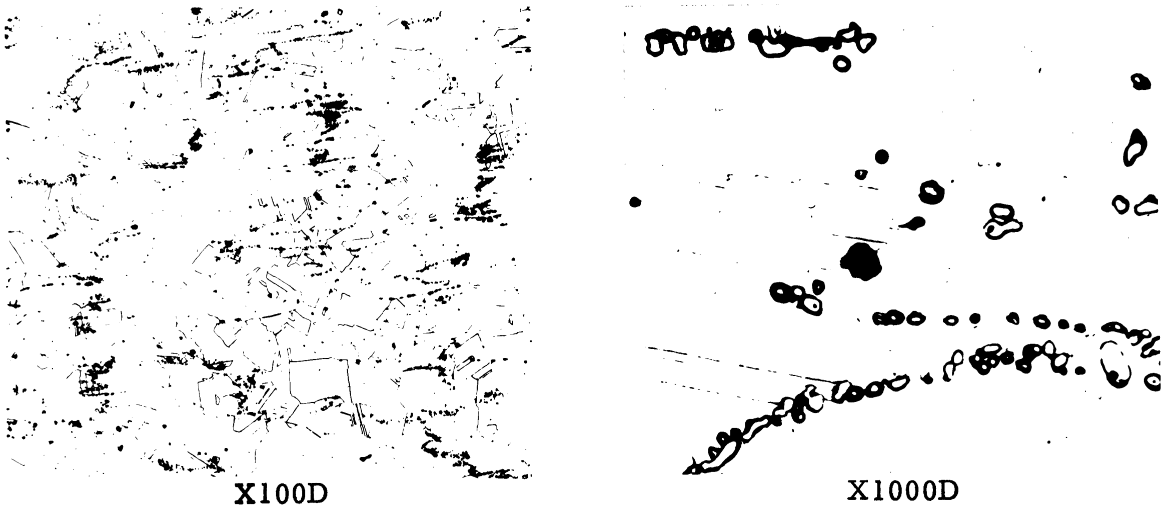


Figure 4 - Microstructure of Heat 206 Solution Treated 1 Hour at 2150°F, Aim Composition 0.03% B, 1.5% Ti.

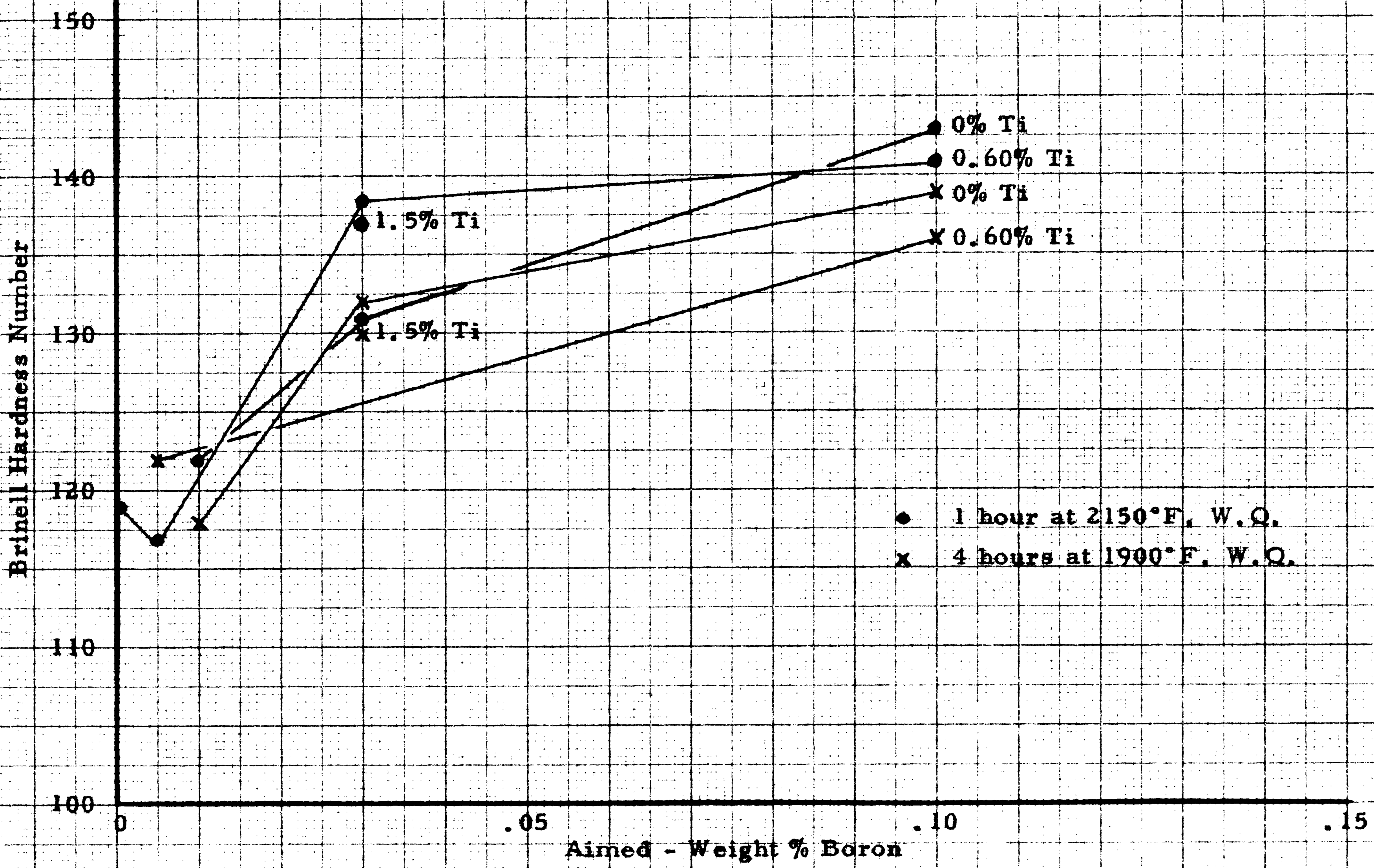


Figure 5. - Effect of Increasing Boron Content on the Brinell Hardness of Induction Heats at Constant Titanium Percentages in Two Solution Treated Conditions.

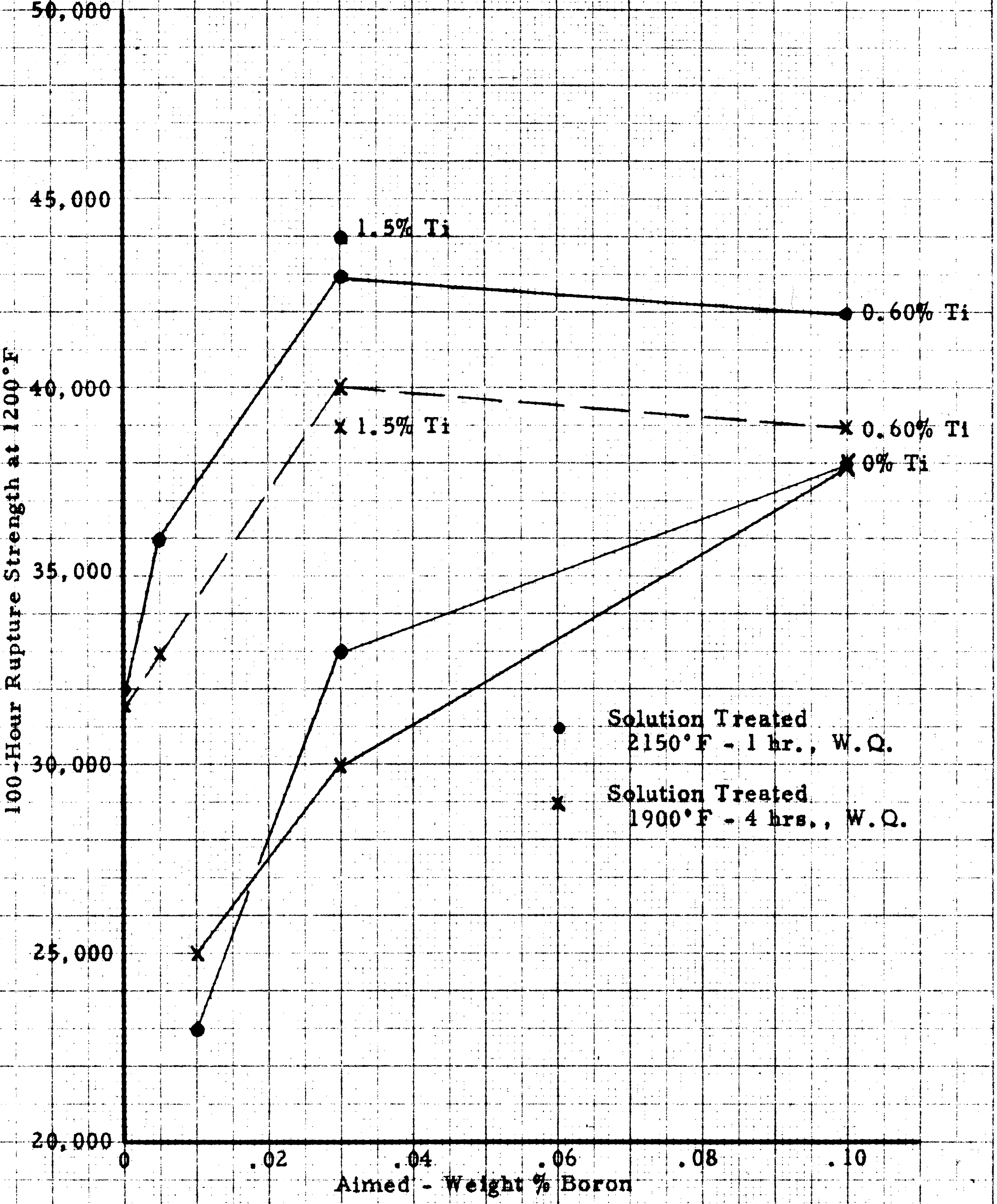


Figure 6. - Influence of Boron Content on the 100-Hour Rupture Strength at 1200°F for Induction Heats in Two Solution Treated Conditions.

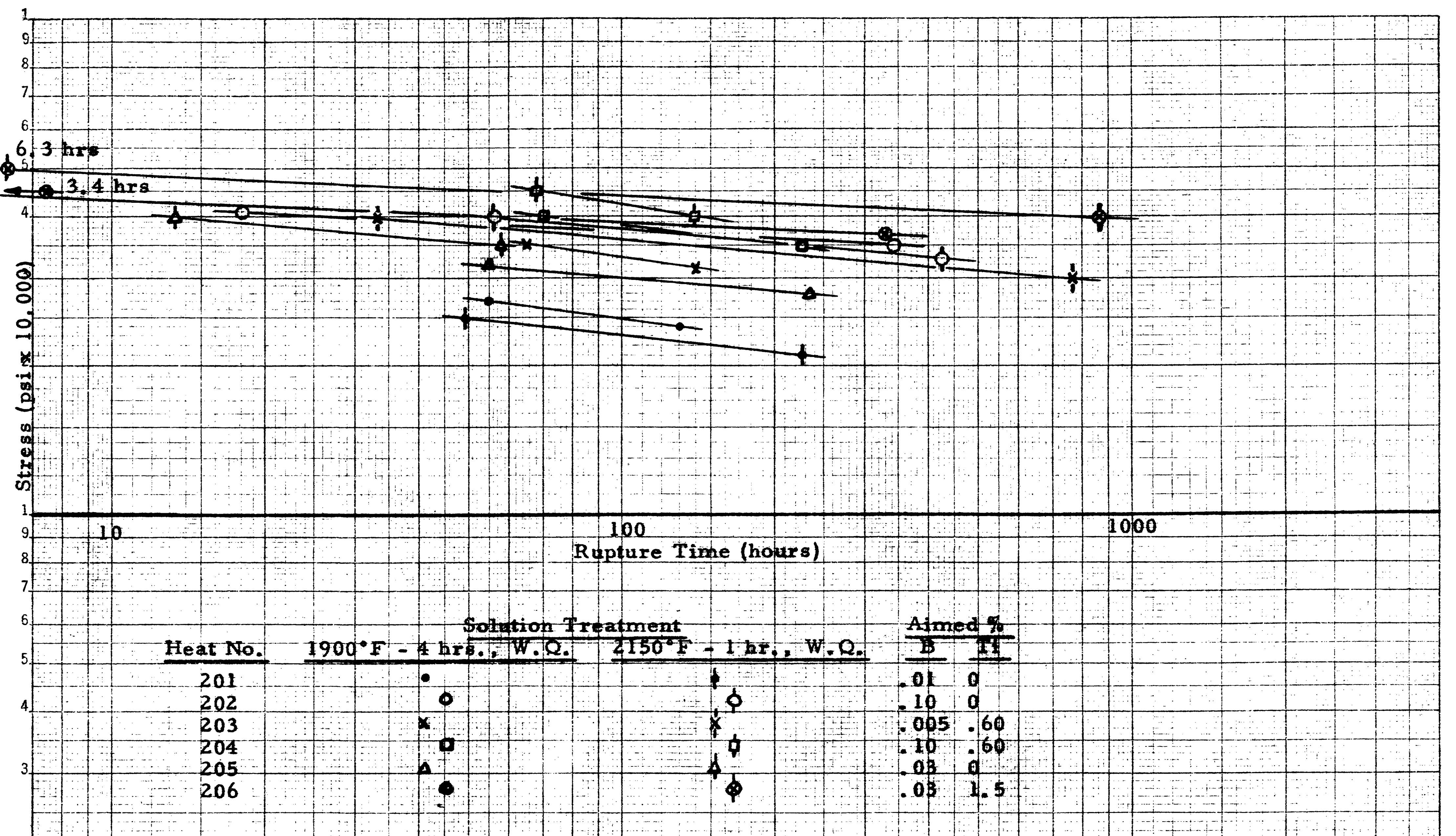


Figure 7. - Comparative Stress-Rupture Properties at 1200°F for Six Induction Heats 201 - 206 in Two Solution Treated Conditions.

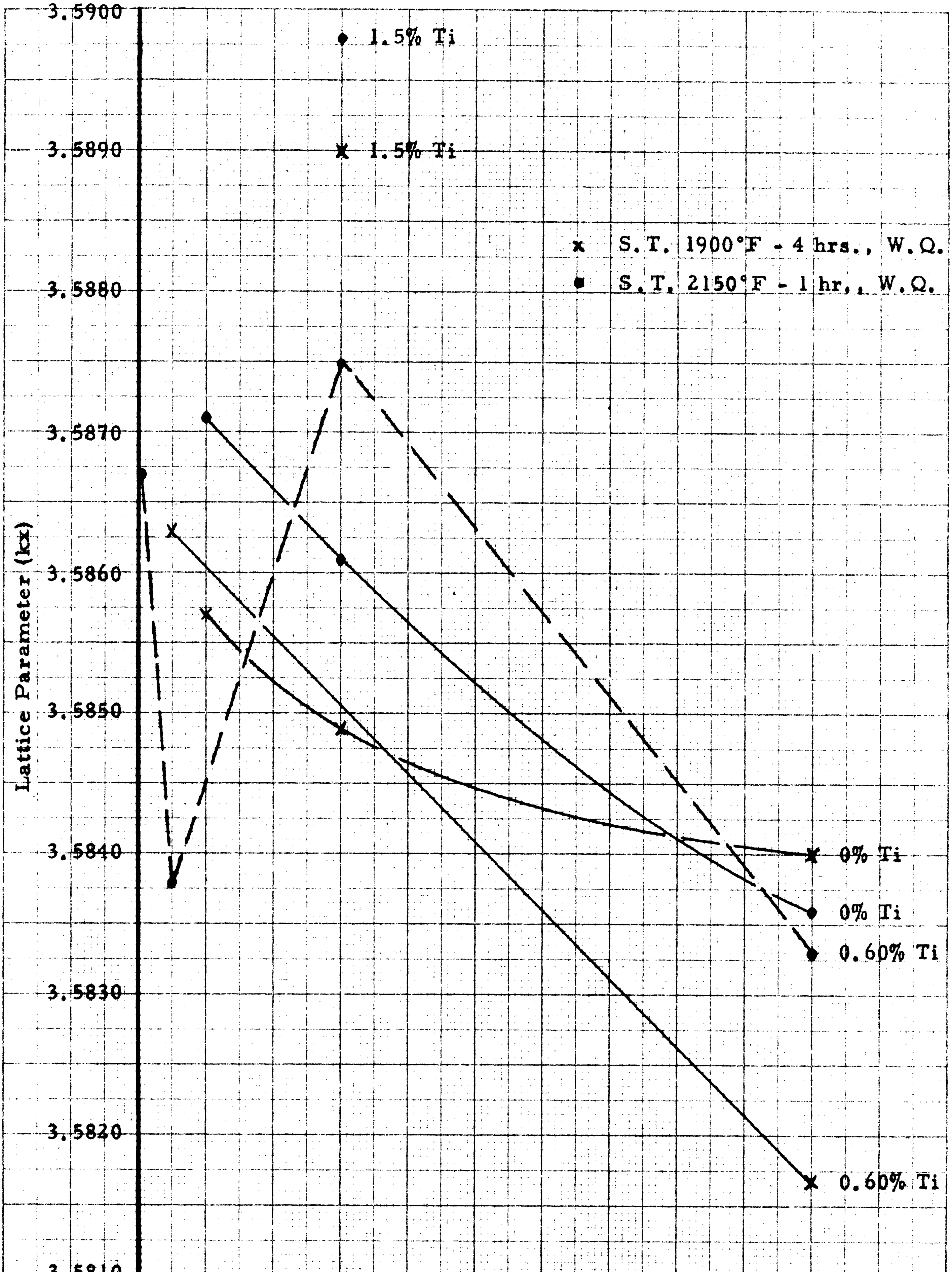


Figure 8. - Effect of Boron Content on the Lattice Parameter of Induction Heats in Two Solution Treated Conditions.