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PROGRESS REPORT

TO THE

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

COVERING

RESEARCH ON HEAT-RESISTANT ALLOYS

Submitted
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PROGRESS REPORT

OF

RESEARCH ON HEAT-RESISTANT ALLOYS

JUNE 30, 1956

This report presents the progress made since the last progress report dated September 15, 1955. The major experimental work has been concentrated on the influence of melting practice and hot-working conditions using the refractory Ti + Al hardened alloy known as Udimet 500. Therefore the rest of the report will be confined to this subject. In addition there were several other investigations in progress in September.

STATUS OF INVESTIGATIONS OF ABNORMAL GRAIN GROWTH, OVERHEATING AND INFLUENCE OF HOT-WORKING CONDITIONS ON PROPERTIES OF WASPALOY

Three other programs were in progress at the time the last progress report was submitted. The status of these programs is as follows:

1. Abnormal Grain Growth in Heat-Resistant Alloys

This program has been completed and the following two final reports submitted covering the results:

(1) "Abnormal Grain Growth in Nickel-Base Heat-Resistant Alloys"

(2) "Abnormal Grain Growth in M252 and S816 Alloys"

2. Influence of Overheating on the Rupture Properties at 1500°F of S816, HS-31 and M252 Alloys.

Final reports have been submitted covering S816 and HS-31 alloys. The titles of the reports were:

(1) "Effect of Overheating on the Creep-Rupture Properties of S816 Alloy at 1500°F"

(2) "Effect of Overheating on the Creep-Rupture Properties of HS-31 Alloy at 1500°F"

The report covering M252 alloy is being prepared. The results for M252 showed that periodic exposures of 2 minutes to 1900° and 2000°F prolonged rupture life at 1500°F. Thus, this type of exposure to these temperatures does not reduce strengths at 1500°F as was the case for S816 and HS-31 alloys. Any reduction in life would require the presence of stress to accelerate creep. Some reduction in life, however, resulted from the periodic exposure to 1600° and 1800°F in a manner similar to that observed in S816 and HS-31 alloys.

3. Influence of Hot-Working Conditions on the Rupture Properties of Waspaloy.

When the budget was reduced this investigation was temporarily stopped. As soon as the report on overheating is completed, the results to date will be prepared in report form.

INFLUENCE OF MELTING PRACTICE, HOT-WORKING CONDITIONS AND HEAT TREATMENT ON THE HIGH-TEMPERATURE PROPERTIES AND HOT-WORKING CHARACTERISTICS OF A Ti + Al HARDENED ALLOY

The study of the influence of melting conditions on the properties of heat-resistant alloys using the refractory alloy known as Udimet 500 has been continued since September 1955 along the following lines:

1. Study of the influence of melting variables, particularly combinations of deoxidation and superheat practice,

2. Introduction of variations in hot-working conditions,

3. Study of the influence of heat-treating conditions on properties,

4. Development of a better understanding of the basic microstructures in the alloys with emphasis on the use of the electron microscope.

High-temperature properties were evaluated in all cases through survey rupture tests at 1600°F under a stress of 25,000 psi. Qualititive observations on hot-working characteristics have been made, although the restrictions imposed by the limited conditions of working have mainly limited this to cracking tendency.

The general objectives of the investigation have been to isolate melting, hot working, and heat treatment variables having a significant effect on creep-rupture properties. With these established, the basic metallurgical causes of the observed variations were to be determined. Melting has been carried out in a vacuum chamber. Ingots were hot rolled to bar stock and heat treated. Detailed chemical analyses including determination of O_2 and N_2 contents were used to evaluate variables. The probability that any variations in melting or hot working would have their influence through alteration of the reactions involving precipitation of Ni_3 (Al, Ti) has led to detailed analysis of microstructures.

INFLUENCE OF MELTING PRACTICE

The program on the effect of melting practice on the high-temperature properties of Udimet 500 was continued. Among the variables studied were deoxidation practice, superheat temperature, pouring temperature, ingot mold shape and refining temperature.

Melting was restricted to vacuum with no introduced gases. Induction melting was carried out in the University of Michigan vacuum furnace in stabilized zirconia crucibles. Pressures before meltdown and after pouring were less than 5 microns as measured by both Stokes and thermocouple gages. Melt temperatures were measured with a Pt - Pt + Rh immersion thermocouple.

Melting cycles for the heats melted are illustrated in figure 1.

Aim analysis, in weight percent, on all heats was as follows:

Cr	Co	Mo	Ti	Al	Mn	Si	C	Ni
20.0	15.0	4.00	3.10	3.10	0.12	0.12	0.08-0.15	Bal.

Electrolytic Ni, Cr, Co, and Mn melting stocks were used. The Mo was arc-melted low carbon stock; the Ti was Ti 55 stock and the Al was 99.99 percent purity ingot stock.

Ten-pound heats were poured into massive copper molds.

Hot-working practice was kept constant for all the heats used in the study of melting practice. This practice included:

1. Homogenizing ingot 1 hour at 2300°F, air cooling,

2. Surface grinding ingot,

3. Rolling at 2150°F to 7/8 inch bar stock using 22 passes with 21 reheats of 10 minutes between passes. The last pass was a 5 percent reduction followed by air cooling.

Chemical analyses were obtained from samples cut from the rolled bar stock. This metal originally was in the center of the as-cast ingots. The analyses are included in Table I.

Evaluation of high temperature properties of the heats was made after the as-rolled stock was solution treated at 2150°F and air cooled. Rupture samples were tested at 25,000 psi and 1600°F. The results are tabulated in Table II.

The results will be reviewed in the following sections.

Effect of Deoxidation Practice

Deoxidation was carried out using varying amounts of C in the charge and with Si and Al. One objective was to melt down with excess oxygen and then hold the melt under high vacuum for possible reaction of the oxygen with impurities to refine the melt. Final deoxidation with carbon required a prolonged time period due to the necessity of slowly adding the carbon to avoid excessive gas evolution.

Effect of a High O_2 Refining Period

The effect of a refining period in the melting cycle was studied by varying the method of carbon addition as follows:

Heat	Method of Carbon Addition
1129	100% of carbon mixed with crucible charge to deoxidize melt during meltdown.
1133	50% of carbon mixed with crucible charge to partially deoxidize melt during meltdown. 50% of carbon added to complete deoxidation after a 20 minute refining period at 2700°F.
1136	0% carbon mixed with crucible charge. 100% carbon added to deoxide melt after a 20 minute refining period at 2700°F.

Holding times for the three heats were not constant because of the difficulty of excessive gas evolution during carbon additions after meltdown. While deoxidation of Heat 1129 occurred during meltdown, deoxidation of Heat 1136 required 1.5 hours after meltdown. After deoxidation, Si, Al, Ti, and Mn additions were made. Then the melts were superheated to and poured at approximately 2900° to 3000°F.

The rupture properties obtained are listed in Table II. They were:

Heat	Rupture Time (hours)	Elongation (percent)	Reduction in Area (percent)
1129	82.3	---	4.7
1133	90.7	4.0	4.0
1136	99.5	6.0	4.0

Effect of Deoxidizing Material

Al, Si, and chunk C were used to deoxidize the melt after meltdown and a 20 minute refining period at 2700°F. Then, after additions of the remaining alloying elements, the melts were superheated to and poured at approximately 3000°F, with the exception of the Al deoxidized heat which was superheated to 3100°F. The heats and variables were:

Heat	Deoxidizing Material
1136	chunk C
1138	Al
1139	Si followed by chunk C (Si did not completely deoxidize the melt)

The rupture results were:

Heat	Rupture Time (hours)	Elongation (percent)	Reduction in Area (percent)
1136	99.5	6.0	4.0
1138	147.4	5.0	4.6
1139	75.5	4.0	3.1

Effect of Ingot Mold Shape

Heat 1137 was melted with identical procedures as those used in Heat 1136 but was cast in an ingot mold with 5° taper whereas Heat 1136 was cast in a straight mold.

The rupture results obtained were:

Heat	Rupture Time (hours)	Elongation (percent)	Reduction in Area (percent)
1136	99.5	6.0	4.0
1137	87.1	6.0	6.4

Effect of Superheat and Pouring Temperatures

Several conditions of superheating were studied to determine if purification of the melt by vaporization was a factor in properties. In addition, evaluation of the effect of pouring temperatures upon ingot structures, cracking during working and rupture properties was desired.

In order to establish the effects of superheat and pouring temperatures, Heats 1141 and 1142 were melted with identical practice as 1138 except for variations in superheat and pouring temperatures as listed below.

Heat	Superheat Temperature	Pouring Temperature
1138	3100°F	3000°F
1141	2975°F	2750°F
1142	2750°F	2750°F

It should be noted that the carbon contents obtained in Heats 1141 and 1142 were high compared to Heat 1138.

The rupture results are summarized as follows:

Heat	Rupture Time (hours)	Elongation (percent)	Reduction in Area (percent)
1138	147.4	5.0	4.6
1141	81.2	8.0	11.5
1142	50.8	8.0	12.3

As-cast macrographs and micrographs illustrating the effect of pouring temperature on structures are shown in figure 2.

Effect of Refining Temperature

It seemed possible that the temperature of the melt during the refining period might affect the purification reactions.

Since Heat 1138 had relatively good rupture properties and hot workability, a similar melting practice was used in making Heat 1143. While the melt temperature was kept at 2700°F during the 20 minute refining period with Heat 1138, in Heat 1143 this temperature was maintained at 2900°F.

In addition, a polished piece of pure Ni was suspended above the melt during this refining period in order to collect metal vapors volatilized from the melt. Chemical analysis of the condensed metal vapors yielded the following quantities of elements in the condensate:

Elements over 10%	Elements 1% to 10%	Elements 0.1% to 1%	Elements 0.01% to 0.1%	Elements 0.001% to 0.01%
Cr, Co, Mn	Ni	P	Al, Cu, Pb	B, Ca, Mg, Mo, Si

Rupture testing revealed that Heat 1143 did not reproduce the properties of Heat 1138.

Heat	Rupture Time (hours)	Elongation (percent)	Reduction in Area (percent)
1138	147.4	5.0	4.6
1143	85.6	15.0	9.3
1143 (duplicate)	86.5	8.5	10.0

Effect of Melting Practice on Cracking During Rolling

Since a constant hot-working practice was employed in rolling the ingots of the heats used to study melting practice, it was possible to compare the tendency to crack during working. Figure 3 shows photographs of the as-rolled bars.

Several differences in tendency for cracking during rolling can be observed.

1. Heat 1129, deoxidized during meltdown without a refining period, had intermediate cracking.

2. Heats 1133, 1136, and 1137, deoxidized by carbon after a refining period, underwent relatively severe cracking.

3. Heats 1138, 1141, and 1142, deoxidized by Al after refining at 2700°F cracked very little. However, it is possible that in Heats 1141 and 1142 the high carbon content contributed to the reduced tendency to crack.

4. Heat 1139, which was deoxidized incompletely with Si and then completely with C, had an intermediate tendency for crack formation. Likewise, Heat 1143, which was deoxidized by Al after refining at 2900°F, was intermediate.

INFLUENCE OF HOT-WORKING VARIABLES

Prior experience has indicated that in order to produce bar stock from the cast ingots with a minimum of cracking, the working conditions employed must remain within a rather narrow range of variables. The best procedure found to hot roll the ingots has been to reduce about 4 to 6 percent in one pass and then reheat for 10 minutes. Metal temperatures had to be kept between 2100° and 2200°F. The cracking problem thus limited the range of variables which could be considered in the study of hot working.

Effect of Homogenization of Ingots

The effect of heating to 2300°F on the workability of Heat 1129 was studied. A portion of the ingot was rolled without any exposure to 2300°F, and then the effect of exposure for one hour prior to rolling and for one hour prior to rolling followed by another hour part way through the rolling was also included. The bars so produced were rupture tested at 1600°F and 25,000 psi after solution treatment both at 1975° and 2150°F. The influence of this homogenization may be summarized as follows:

Effect on Workability

The bars from this study are shown in figure 4. Some corner cracking was evident on all three of the bars but was not severe. The cracks were not deep and apparently had no effect on the metal below them in the bars. The homogenization apparently had no effect on the workability of this heat.

Effect on Rupture Time

The rupture time for Heat 1129 (fig. 5) does not appear to have been influenced significantly by the 2300°F homogenization. When the final heat treatment was 1975°F, homogenization at 2300°F may have been slightly detrimental, while it may have been somewhat benificial when the final heat treatment was at 2150°F.

Effect on Microstructure

The microstructures of two of the bars following rolling are shown in figure 6. All three exhibited a mixed grain size which did not appear to vary appreciably from bar to bar. After solution treatment at either of the two temperatures used for this study, recrystallization resulted in a more uniform grain size (fig. 7). The same structures were retained after rupture testing at 1600°F (figs. 8 and 9) with some additional precipitation appearing in the grain boundaries. The microstructures after rupture testing also show at X1000D the presence of a fine dispersion of the Ni_3 (Al, Ti) precipitate.

Effect of Amount of Finishing Reduction

Two heats, 1141 and 1142, were used in the study of the effect on rupture properties of the amount of reduction given in the last passes of the rolling. The

ingots were homogenized one hour at 2300°F and surface ground prior to the start of rolling. They were then rolled from the 2-1/2 inch ingot size down to 1 inch broken-cornered squares using the usual procedure of single pass reductions of about 4-6 percent with reheats for ten minutes between passes. The bars were then cut in half and one half finished to 7/8 inch squares using the usual practice of three passes with two reheats. The remaining two halves were then reduced to 7/8 inch squares in two passes with no reheats. The samples obtained from these bars were then heat treated and tested at 1600°F and 25,000 psi. Although there was some scatter in the data obtained for the two heats and the two working conditions (table II and fig. 10), there do not appear to be any differences in rupture properties as a result of the heavier reduction in the final passes on these two heats.

EFFECT OF HEAT TREATMENT AFTER ROLLING

In all of the prior studies discussed in this report the material has been rupture tested as solution treated at 2150°F and air cooled. Several other heat treatments also were studied in order to determine the effect on properties of the various steps of normal heat treating. The following conditions were evaluated on many of the heats produced in this investigation:

1. Solution treat 4 hours at 1975°F, air cooled,
2. Solution treat 1 or 2 hours at 2150°F, air cooled,
3. Solution treat 2 hours at 2150°F, air cooled, plus solution treat 4 hours at 1975°F, air cooled, followed by aging 24 hours at 1550°F plus 16 hours at 1400°F,
4. Solution treat 2 hours at 2150°F, air cooled plus the same double age as in (3).

The results of this study on most of the heats prepared indicate the following general trends as a result of the heat treating conditions (fig. 11):

1. With only one exception, solution treating at 2150°F resulted in higher strengths than solution treating at 1975°F. Although the strength levels were different from heat to heat, the magnitude of this increase appeared to be about the same for all heats.

2. The effect of aging on the strength of the material after solution treating at 2150°F was only studied on two heats. The results are not conclusive and show an improvement in life for one the heats and a decrease in life for the other one when compared to the strength as solution treated at 2150°F with no aging.

3. The effect of a double solution treatment followed by a double age (condition 3 above) was also difficult to evaluate. In general, the strength appears to be about the same as it was after solution treating at 2150°F.

Since cooling rate after solution treatment was found to influence the precipitation reaction, a limited investigation was carried out to find the effect of cooling rate before aging and rupture testing.

Specimens of Heats 1138 and 1139 were heat treated by 2 hours at 2150°F, ice-brine quenched; plus 4 hours at 1975°F, ice-brine quenched; plus 24 hours at 1550°F; plus 16 hours at 1400°F.

The specimen from Heat 1139 fractured intergranularly during machining. Other cracks were visible on the machined surface. The specimen from Heat 1138 had no visible cracks after machining, but ruptured upon loading at 1600°F.

Microstructural investigations showed the presence of a discontinuous precipitate at the grain boundaries after aging in both samples. However, specimens which had been air cooled after solution treatment did not exhibit discontinuous precipitation and were not embrittled.

Effect of Testing Procedures

Testing of all samples prepared was conducted at 1600°F and 25,000 psi. Loading the specimens into the furnace was done by a standard practice of placing the test in a hot furnace, adjusting temperature and loading in four hours. In order to know the effect of variations from this standard procedure,deliberate differences in the preheat time were evaluated. The results of these tests were as follows as conducted on Heat 1130:

Time at 1600°F before applying stress(hours)	Rupture Time (hours)
2	80.0
4	102.2
24	23.4

These results show that there is a definite influence on rupture time of the time at temperature prior to loading. These rupture times, plotted together with hardnesses after aging at 1600°F (fig. 12) indicate a similar trend for both effects.

STRUCTURAL STUDIES OF UDIMET 500

The variations in properties may be controlled by the distribution of the Ni_3 (Al, Ti) precipitates and other secondary phases. Electron micrographic studies offer a means of studying this possibility. Accordingly, a program is in progress to provide familiarity with the fine structures revealed by the electron microscope. The initial phase of this study involves determination of the size and distribution of the secondary phases in one heat of the alloy as influenced by exposure to several temperatures for varying times. Ultimately it is expected

that sufficient background information will be developed so that it will be possible to determine if melting practice and processing variables have their influence on properties at high temperature through the secondary phase structures.

The structural studies included work on solution of the secondary phases and precipitation of the secondary phases as described in the following sections.

Effect of Solution Treatment

Specimens from rolled bar stock of Heat 1138 were homogenized 2 hours at 2150°F and ice-brine quenched.

These specimens were treated 4 hours at 1700°, 1800°, 1900°, and 2000°F and ice-brine quenched.

Vickers penetration hardnesses obtained on these samples are presented in figure 13. In addition, hardness of a sample aged at 1600°F is included.

While treating at 1600° and 1700°F increased the hardness above the original value obtained by homogenization at 2150°F followed by ice-brine quenching, treating at 1800° and 1900°F decreased the hardness significantly. Treating at 2000°F gave a slightly lower hardness than the homogenized and quenched value.

Figures 14 and 15 include micrographs of the samples. A uniform precipitate was present in the matrix after treatments at 1700°, 1800° and 1900°F. This precipitate increased in size and spacing as the treatment temperature increased to 1900°F. The precipitate is assumed to be undissolved Ni_3 (Al, Ti).

Treating at 2000°F maintained the precipitate in solution so that none is visible in the micrographs (figs. 14d, 15d). In addition, it is apparent that considerable amounts of secondary phases agglomerated at the grain boundaries at temperatures up to 1900°F. These were not present after treatment at 2000°F.

It is evident from this study that the solution temperature for Ni_3 (Al, Ti) in Udimet 500 lies between 1900° and 2000°F for a 4 hour heat treatment. The

massiveness of the precipitate particles after treatment at 1900°F suggests that the solution temperature could be as high as 1950°F in Heat 1138.

Microstructures of several of the heats after solution treatment at 1975°F have shown presence of undissolved Ni_3 (Al, Ti). In some cases the precipitate was uniform throughout the structure, while in other cases the precipitate appeared in bands. It is believed that this condition arises because the solution temperature of Ni_3 (Al, Ti) is close to 1975°F. The presence of the precipitate after the 1975°F treatment seems to depend somewhat upon prior history.

It is probable that some of the erratic rupture test results obtained after the 1975°F treatment can be attributed to failure to completely dissolve the Ni_3 (Al, Ti). In cases where the precipitate was visible after the 1975°F treatment, increasing the solution treating temperature to 2150°F increased rupture life.

The hardness data in figure 13, showing low hardness after the 1800° and 1900°F treatments, indicate that these treatments can lead to severe overaging of the Ni_3 (Al, Ti) and matrix depletion. In fact, the hardnesses after these treatments were lower than those obtained after complete solutioning and ice-brine quenching.

Precipitation Effects

Samples of rolled bar stock of Heat 1138 were solution treated 2 hours at 2150°F. One set of samples was ice-brine quenched, one set was air cooled and one sample was water quenched after the treatment.

Effect of Cooling Rate after Solution Treatment

Comparison of hardnesses of samples ice-brine quenched, water quenched, and air cooled after the treatment at 2150°F is included in figure 16.

Figure 16 also contains optical and electron micrographs of the three conditions.

Review of these data reveals that considerable precipitation of Ni_3 (Al, Ti) occurs during air cooling after solution treating. Although the optical micrograph contains no evidence of Ni_3 (Al, Ti) in the air cooled sample, the high hardness level and the electron micrograph show its presence. While the micrographs of the ice-brine quenched and water cooled samples do not show a precipitate, the higher hardness level of the latter sample indicates that some precipitation occurred even during water quenching.

Effect of Aging Time and Temperature

Two sets of samples, one set ice-brine quenched after solution treatment and the other air cooled, were aged 1, 10 and 100 hours at 1000°, 1200°, 1400°, and 1600°F.

Vickers penetration hardnesses for these samples are summarized in figures 17 and 18. Observations of these figures allows several general conclusions to be drawn.

1. Aging at 1000°F increases hardness little.

2. Hardness increases progressively up to 100 hours at 1200° and 1400°F. No overaging was evident at these temperatures in 100 hours.

3. Overaging occurred at 1600°F during the first 100 hours.

In addition several comparisons can be made between figure 17 and figure 18 to show the effect of initial treatment before aging.

1. The difference in the initial precipitate present in the air cooled and ice-brine quenched conditions affects the aging curves at 1000° and 1200°F. Longer aging times are needed to reach a given hardness level when the initial condition was ice-brine quenched.

2. At 1400°F, although the hardness after 1 hour is lower in the ice-brine quenched sample, longer aging times give approximately the same hardness level with both starting conditions.

3. During aging at 1600°F, the samples initially ice-brine quenched resisted overaging to a greater extent.

Representative optical micrographs of the aged samples are included in figures 19 and 20.

Electron microscopic studies in the aging program have not been completed as yet. However, two representative micrographs are shown in figure 21.

It was observed from the micrographs that:

1. The Ni_3 (Al, Ti) precipitate was not clearly resolvable by optical microscopy after any of the aging treatments of the study. Aging did develop the precipitate so that evidence of it was seen in optical micrographs as a general greying or spotting of the background.

2. Preliminary electron micrograph studies have shown that the precipitate is resolvable after aging at or above 1000°F.

3. Significant changes in the grain boundaries occurred during aging. In general, the grain boundaries broadened with increasing temperature and time.

4. A particularly striking phenomenon occurred at the grain boundaries during aging after ice-brine quenching. This has been tentatively identified as a discontinous precipitation; it appeared to be most pronounced during aging at 1200° and 1400°F. It should be noted that microstructural evidence of this was obtained in the weak and brittle ice-brine quenched rupture specimens mentioned in the section on heat treating variables.

5. Electron micrographs of specimens aged at 1600°F showed two agglomerated phases at the grain boundaries. One of these phases eteched like Ni_3 (Al, Ti).

Future Structural Studies

Electron micrography will be completed for the aged specimens and for specimens after rupture testing. This will serve as a basis for relating properties at high temperatures to possible structural differences resulting from variations in melting practice and prior history. The latter studies are the main objective of this part of the investigation. At present it seems as if the size and distribution of the Ni_3 (Al, Ti) particles may be the main cause of variation. The grain boundary precipitation observed during aging suggest that attention should also be directed to this factor. The available information also suggests that stability of precipitates may be a factor and should be evident in both microstructures and hardness values.

TABLE I

Results of Chemical Analyses of Heats (weight percent)

Heat	C	Vacuum Fusion O_2	Vacuum Fusion N_2	Kjeldahl N_2	Ti	Al	Mo	Cr	Co	Si	Mn	S	P
1129	.05	.0006	.0003	.013	2.82	3.30	4.00	21.2	14.9	.12	.13	.021	.003
1130	.04	.0007	.0004	.010	3.17	3.45	4.00	21.7	14.6	.14	<.10	.025	.004
1133	.06	.0004	.0008	.005	3.22	3.35	4.00	20.7	14.8	.11	.16	.025	.005
1136	.05	.0003	.0005	.007	2.98	3.10	4.10	18.1	15.2	.10	.10	.021	.006
1137	.09	.0003	.0006	.005	3.18	3.15	4.20	18.2	15.1	.10	.10	.025	.008
1138	.08	.0012	.0004	.008	3.14	3.14	4.15	18.8	15.1	.10	<.10	.026 .008 (1)	.008
1139	.08	----	----	.006	3.30	3.35	4.10	19.4	15.0	.14	.15	.026	.006
1141	.20	----	----	.006	2.98	3.00	4.20	19.2	14.5	.22	.15	.020	.007
1142	.19	----	----	.006	2.93	2.85	4.10	19.8	14.5	.23	.13	.018	.004
1143	.13	----	----	.005	3.05	3.15	4.10	19.8	15.2	.12	.10	.008	.005

(1) Check analysis at second laboratory.

TABLE II

Stress-Rupture Data at 25,000 psi and 1600°F

Heat	Carbon (Weight Percent)	Solution Treating Time (hr)	Solution Treating Temperature (°F)	Aged 24 hrs at 1550°F+ 16 hrs at 1400°F	Rupture Time (hr)	Elongation (percent)	Reduction of Area (percent)	Hot Working Procedure (1)
1129	0.05	4	1975	none	116.7	--	6.3	(2)
		1	2150	none	80.7	5.0	6.3	(2)
		4	1975	none	63.9	5.0	8.0	(3)
		1	2150	none	82.3	--	4.7	(3)
		4	1975	none	73.6	8.0	5.6	(4)
		1	2150	none	124.1	4.0	8.5	(4)
1130	0.04	4	1975	none	80.0	5.9	6.9	Standard
				2 hr (5)	102.2	2.0	5.6	Standard
				4 hr (5)	23.4	13.0	13.0	Standard
				24 hr (5)				
		2	2150	none	117.7	8.0	3.2	Standard
1133	0.06	4	1975	none	62.1	6.0	7.9	Standard
		2	2150	none	90.7	4.0	4.0	Standard
1136	0.05	4	1975	none	79.0	12.0	16.7	Standard
		1	2150	none	99.5	6.0	4.0	Standard
1137	0.09	4	1975	none	29.3	13.0	18.4	Standard
		1	2150	none	87.1	6.0	6.4	Standard

TABLE II (continued)

Heat	Carbon (Weight Percent)	Solution Treating Time (hr)	Solution Treating Temperature (°F)	Aged 24 hrs at 1550°F+ 16 hrs at 1400°F	Rupture Time (hr)	Elongation (percent)	Reduction of Area (percent)	Hot Working Procedure (1)
1138	0.08	4	1975	none	71.7	7.0	7.9	Standard
		2	2150	none	147.4	5.0	4.6	Standard
		2 4	2150+ 1975	aged	152.9	3.0	5.6	Standard
		2 4	2150 (5) + 1975 (5)	aged	Broke on Loading			Standard
1139	0.08	4	1975	none	126.9	5.3	[illegible]7	Standard
		2	2150	none	75.5	4.0	3.1	Standard
		2 4	2150+ 1975	aged	91.9	3.0	4.0	Standard
		2 4	2150 (6) + 1975	aged	Broke during Machining			Standard
1141	0.20	4	1975	none	28.2	7.0	9.0	Standard
		2	2150	none	81.2	8.0	11.5	Standard
		2 4	2150 + 1975	aged	42.1	9.7	11.7	Standard
		2	2150	aged	66.9	8.5	10.0	Standard
		4	1975	none	55.9	10.0	11.0	(7)
		2	2150	none	53.4	8.0	9.5	(7)

TABLE II (continued)

Heat	Carbon (Weight Percent)	Solution Treating Time (hr)	Solution Treating Temperature (°F)	Aged 24 hrs at 1550°F+ 16 hrs at 1400°F	Rupture Time (hr)	Elongation (percent)	Reduction of Area (percent)	Hot Working Procedure (1)
1141	0.20	2 4	2150 + 1975	aged	36.5	8.8	11.7	(7)
1142	0.19	4	1975	none	37.7	9.0	10.1	Standard
		2	2150	none	50.8	8.0	12.3	Standard
		2 4	2150 + 1975	aged	52.5	10.0	10.1	Standard
		2	2150	aged	85.4	8.0	8.8	Standard
		4	1975	none	37.3	10.0	13.8	(7)
		2	2150	none	56.2	9.0	11.7	(7)
		2 4	2150 + 1975	aged	57.6	12.0	14.8	(7)
1143	0.13	4	1975	none	82.5	5.7	11.5	Standard
		2	2150	none	85.5 86.5	15.0 8.5	9.3 10.0	Standard
		2 4	2150 + 1975	aged	80.6	11.5	13.0	Standard

Notes 1 through 7 on following page.

TABLE II (continued)

(1) "Standard" procedure as follows: 1 hour at 2300°F, surface ground, rolled at 2150°F to 7/8 inch bar in 22 passes with 21 reheats with 5% in last pass.

(2) Same as standard procedure with omission of surface grinding and homogenization at 2300°F.

(3) Same as standard procedure with omission of surface grinding.

(4) Same as standard procedure with omission of surface grinding, plus 1 hour at 2300°F after completion of first 10 passes (bar size 1-3/16 inches).

(5) Preheat time before applying load in rupture testing.

(6) Ice-brine quenched after solution treating.

(7) Same as standard procedure through first 19 passes (bar size 1 inch). Then rolled to 7/8 inch at 2150°F in 2 passes with no reheats (15 percent reduction).

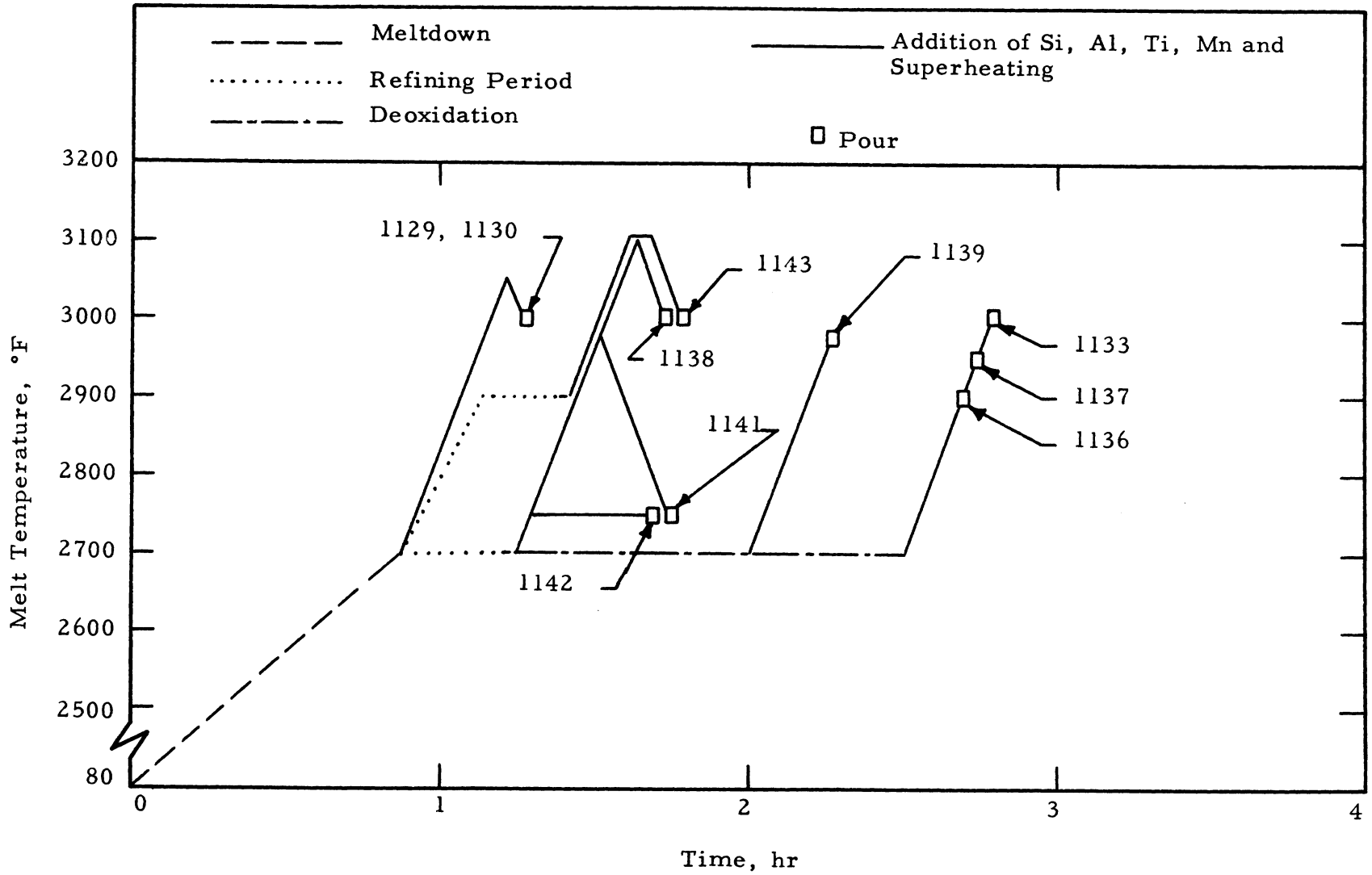
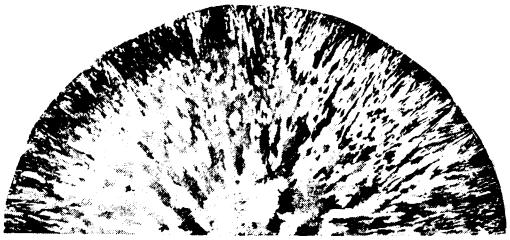
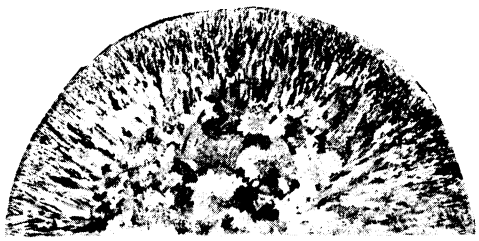


Figure 1. - Melting cycles used in study of effect of melting variables on high temperature properties of UDIMET 500.

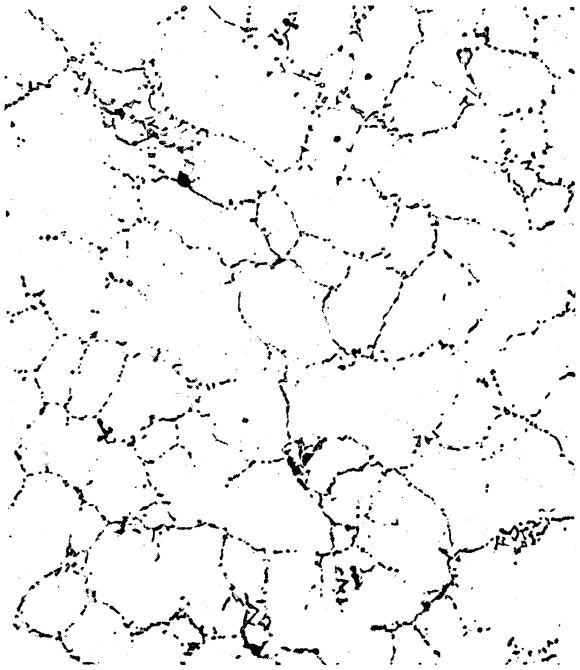


Actual Size
(a) Macrograph of ingot section of Heat 1138 poured at 3000°F



Actual Size
(c) Macrograph of ingot section of Heat 1141 poured at 2750°F

X100D
(b) Microstructure of center of as-cast ingot of Heat 1138 poured at 3000°F



X100D
(d) Microstructure of center of as-cast ingot of Heat 1141 poured at 2750°F

Figure 2. - Effect of pouring temperature on as-cast structures of Heat 1138 (0.08C) and Heat 1141 (0.20C).

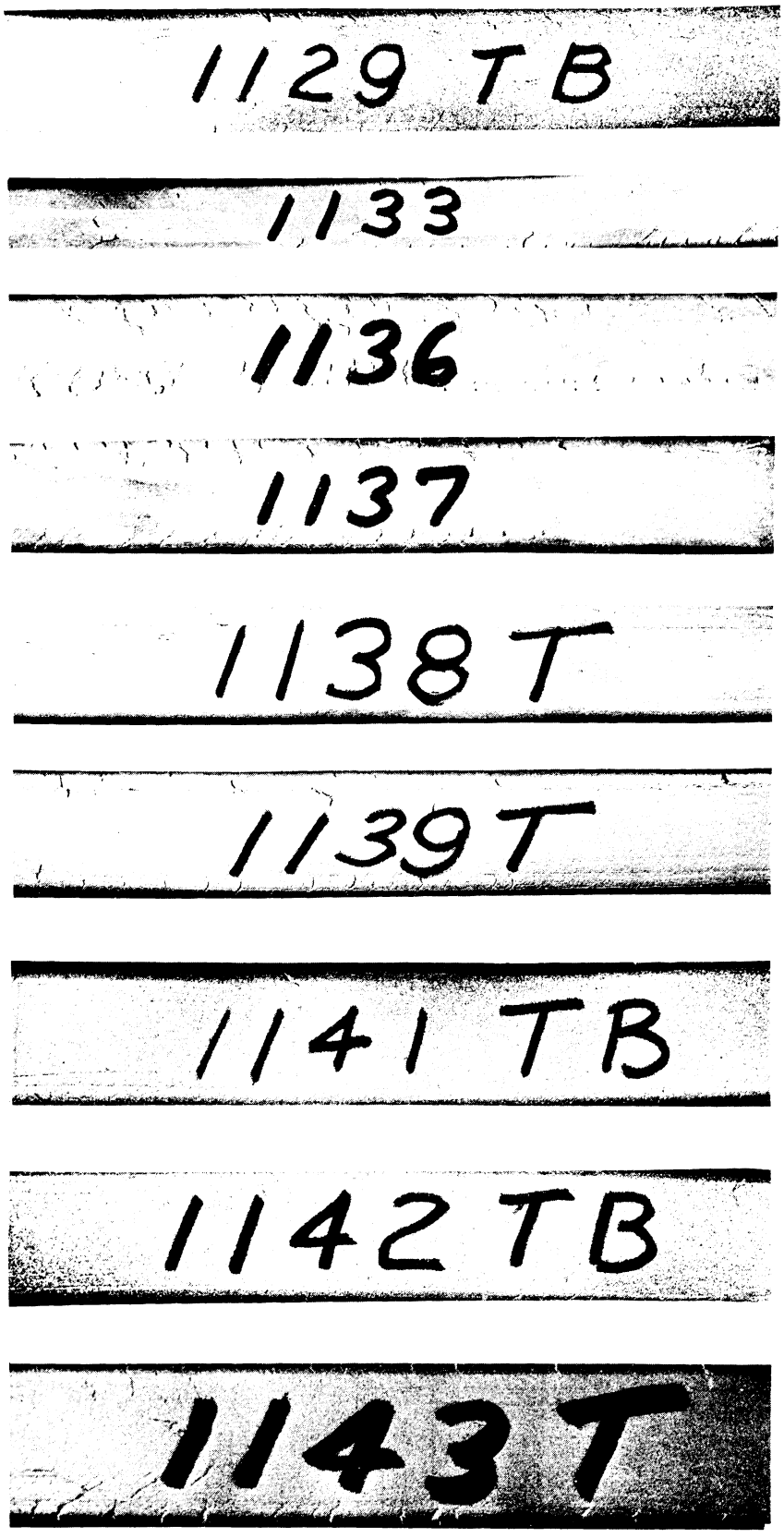
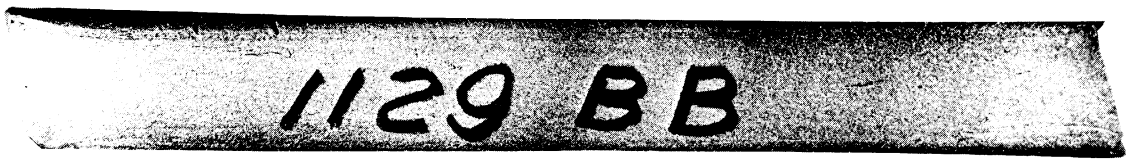


Figure 3. - Surfaces of 7/8 inch bars rolled from heats melted in study of effect of melting practice. Bars were rolled with constant hot-working practice.



(a) No homogenization

(b) Homogenized 1 hour at 2300°F before rolling

(c) Homogenized 1 hour at 2300°F before rolling
plus 1 hour during the rolling

Figure 4. - Photographs of as-rolled 7/8 inch bars from Heat 1129 showing the influence of homogeniztion at 2300°F on the workability.

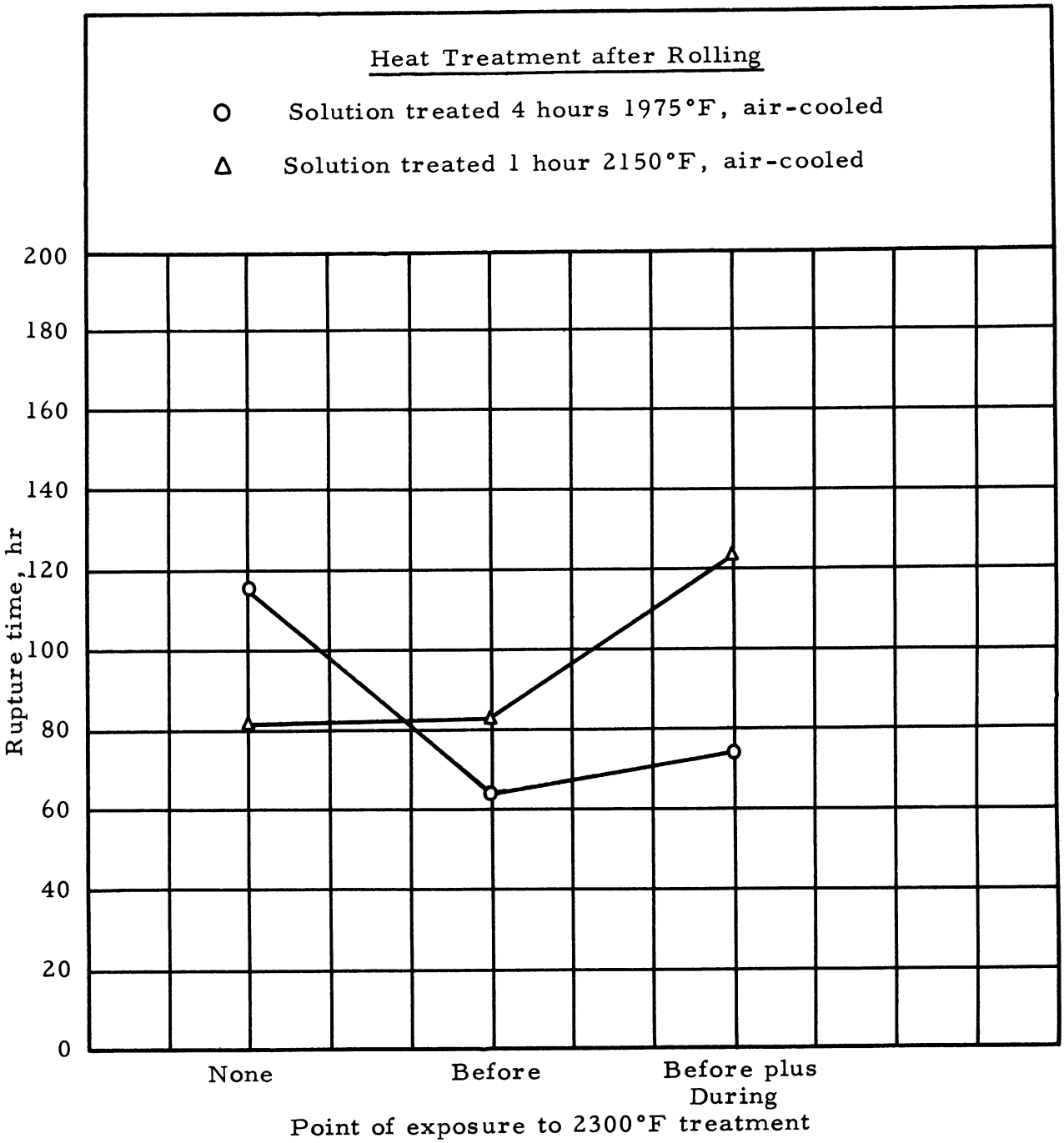
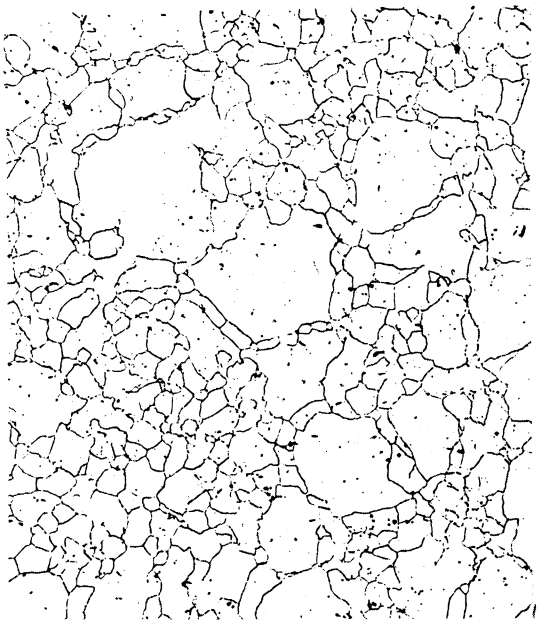
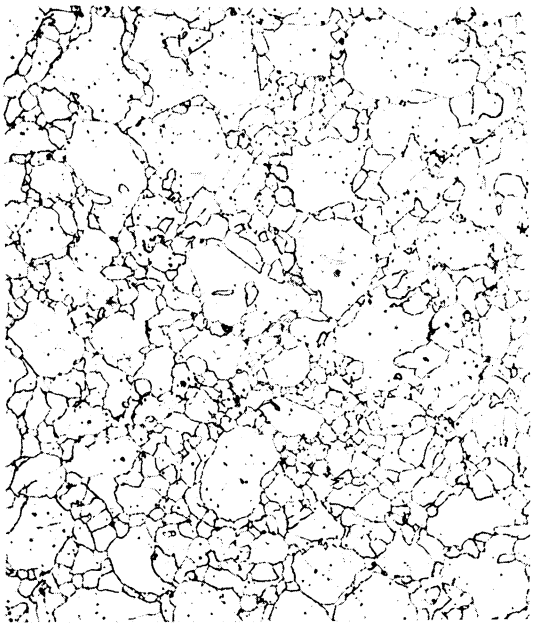


Figure 5. - Influence of exposure to 2300°F for 1 hour before and during the rolling of Heat 1129 on its rupture time at 1600°F and 25,000 psi.



X100D

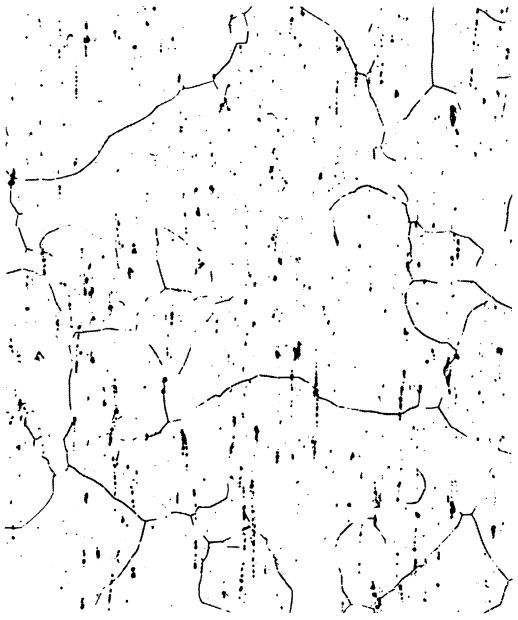
(a) No homogenization



X100D

(b) Homogenized 1 hour at 2300°F before rolling plus 1 hour during rolling

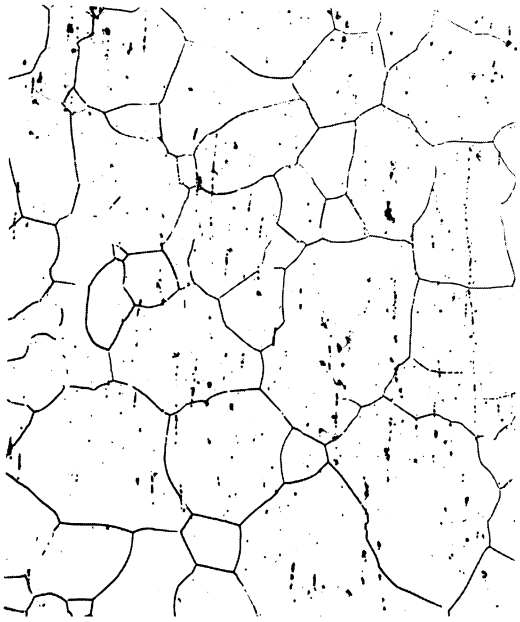
Figure 6. - Influence of homogenization at 2300°F on the microstructure of Heat 1129 in the as-rolled condition.



X100D
No homogenization

X100D
Homogenized 1 hour before rolling
plus 1 hour during rolling

(a) Bars solution treated 4 hours 1975°F, air-cooled



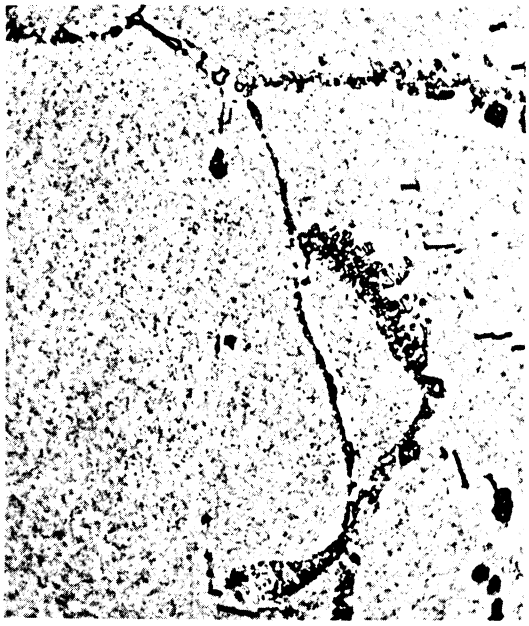
X100D
No homogenization

X100D
Homogenized 1 hour before rolling
plus 1 hour during rolling

(b)Bars solution treated 1 hour 2150°F, air-cooled

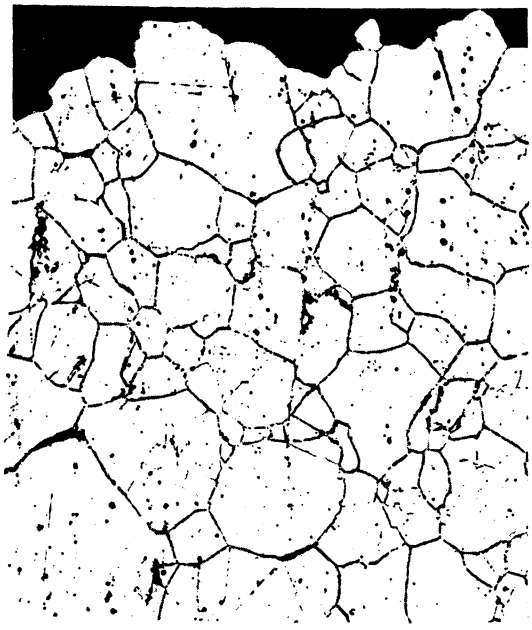
Figure 7. - Influence of homogenization at 2300°F on the microstructure of Heat 1129 after solution treatment.

X100D

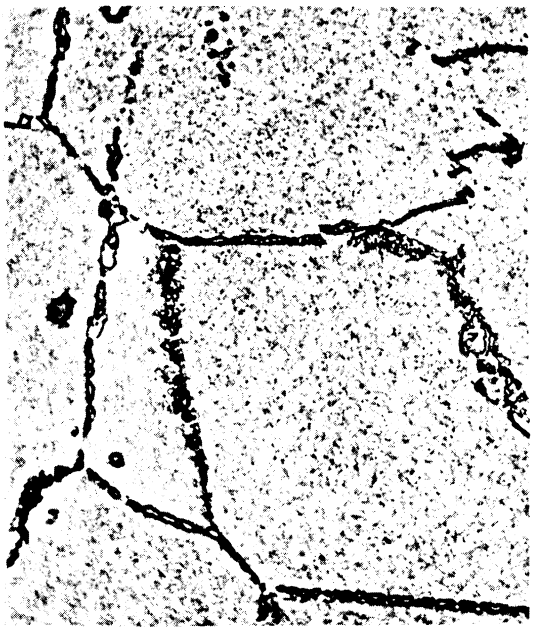


X1000D

(a) Solution treated 4 hours 1975°F, air-cooled, Rupture 116.7 hr



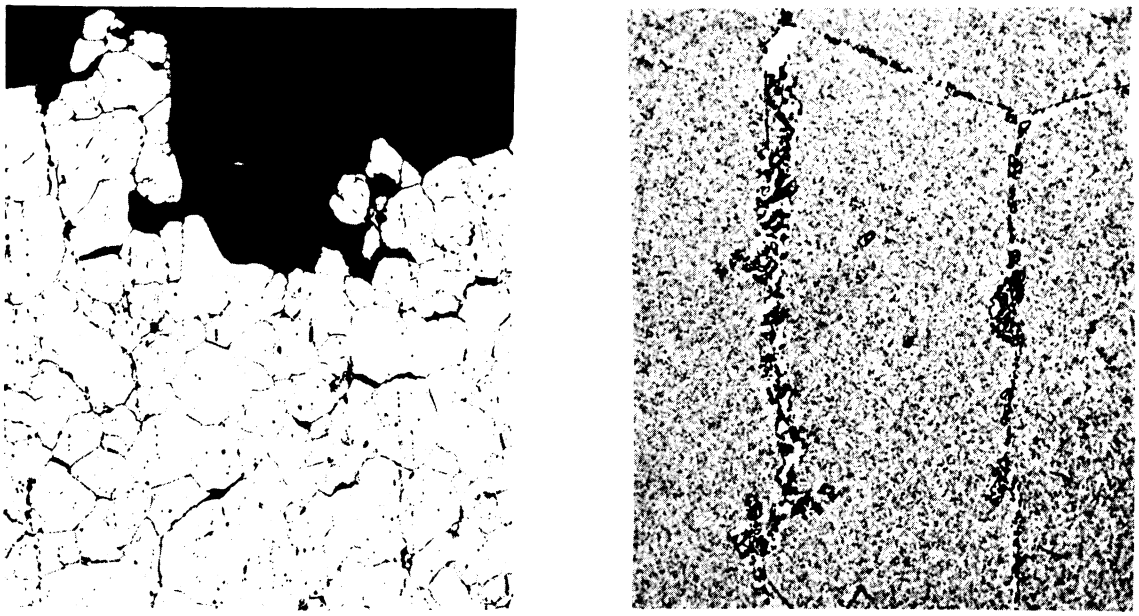
X100D



X1000D

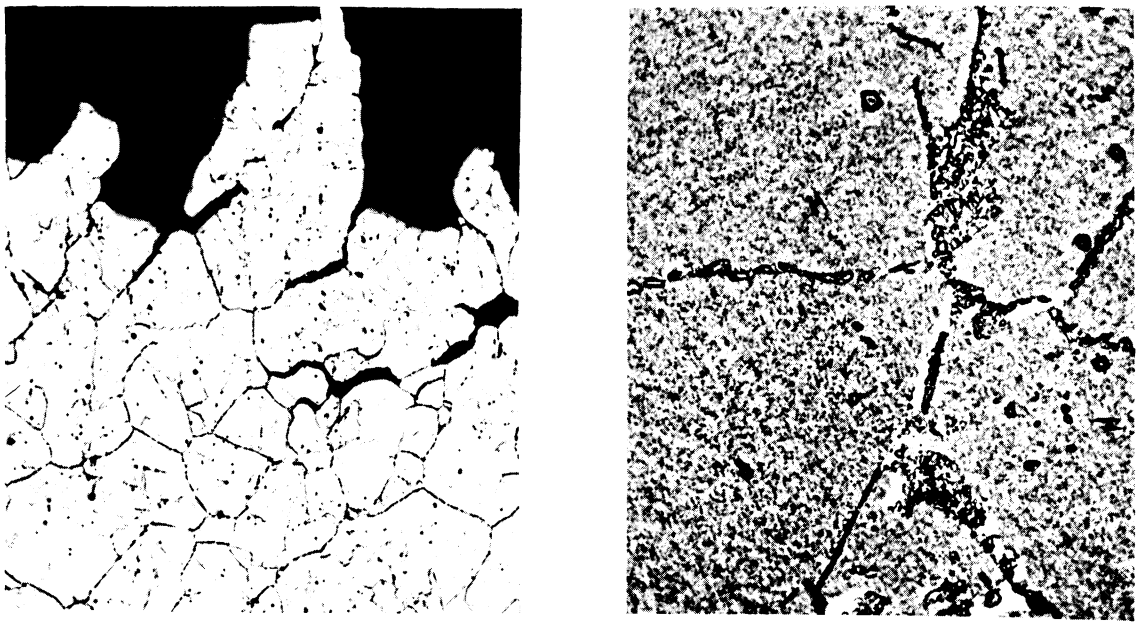
(b) Solution treated 1 hour 2150°F, air-cooled, Rupture 80.7 hr

Figure 8. - Microstructures of Heat 1129 rolled without homogenization and rupture tested at 1600°F and 25,000 psi after the indicated solution treatment.



X100D X1000D

(a) Solution treated 4 hours 1975°F, air-cooled, Rupture 73.6 hr



X100D X1000D

(b) Solution treated 1 hour 2150°F, air-cooled, Rupture 124.1 hr

Figure 9. - Microstructures of Heat 1129 homogenized 1 hour at 2300°F before rolling and again during rolling and rupture tested at 1600°F and 25,000 psi after the indicated solution treatment.

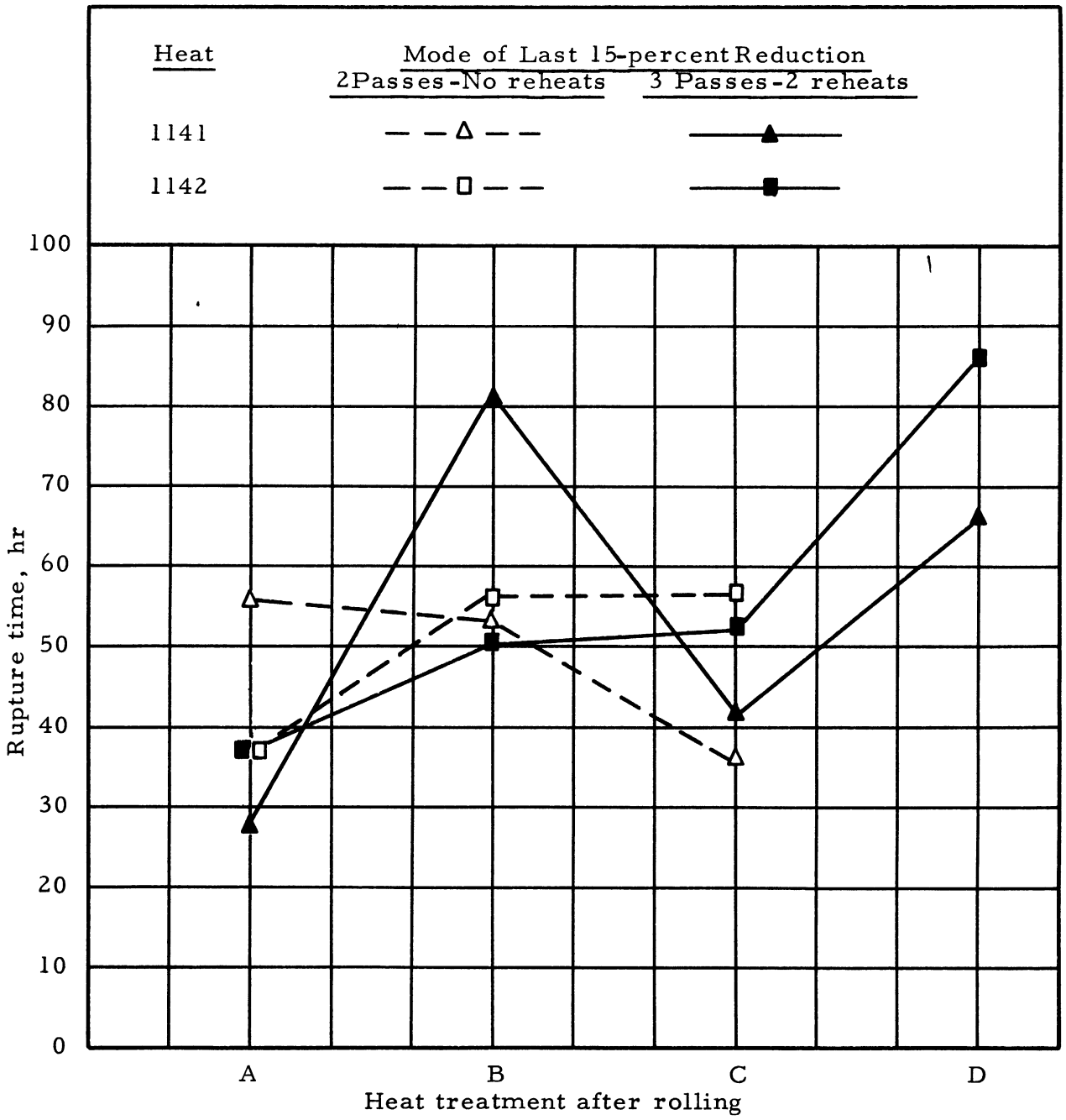


Figure 10. - Influence of mode of last 15-percent reduction by rolling on the response of two heats to the following subsequent heat treatments:
A - 4 hours 1975°F, air-cooled
B - 2 hours 2150°F, air-cooled
C - B+A+ age 24 hours 1550°F, plus 16 hours 1400°F
D - B+ age as in C.
Rupture test at 1600°F and 25,000 psi.

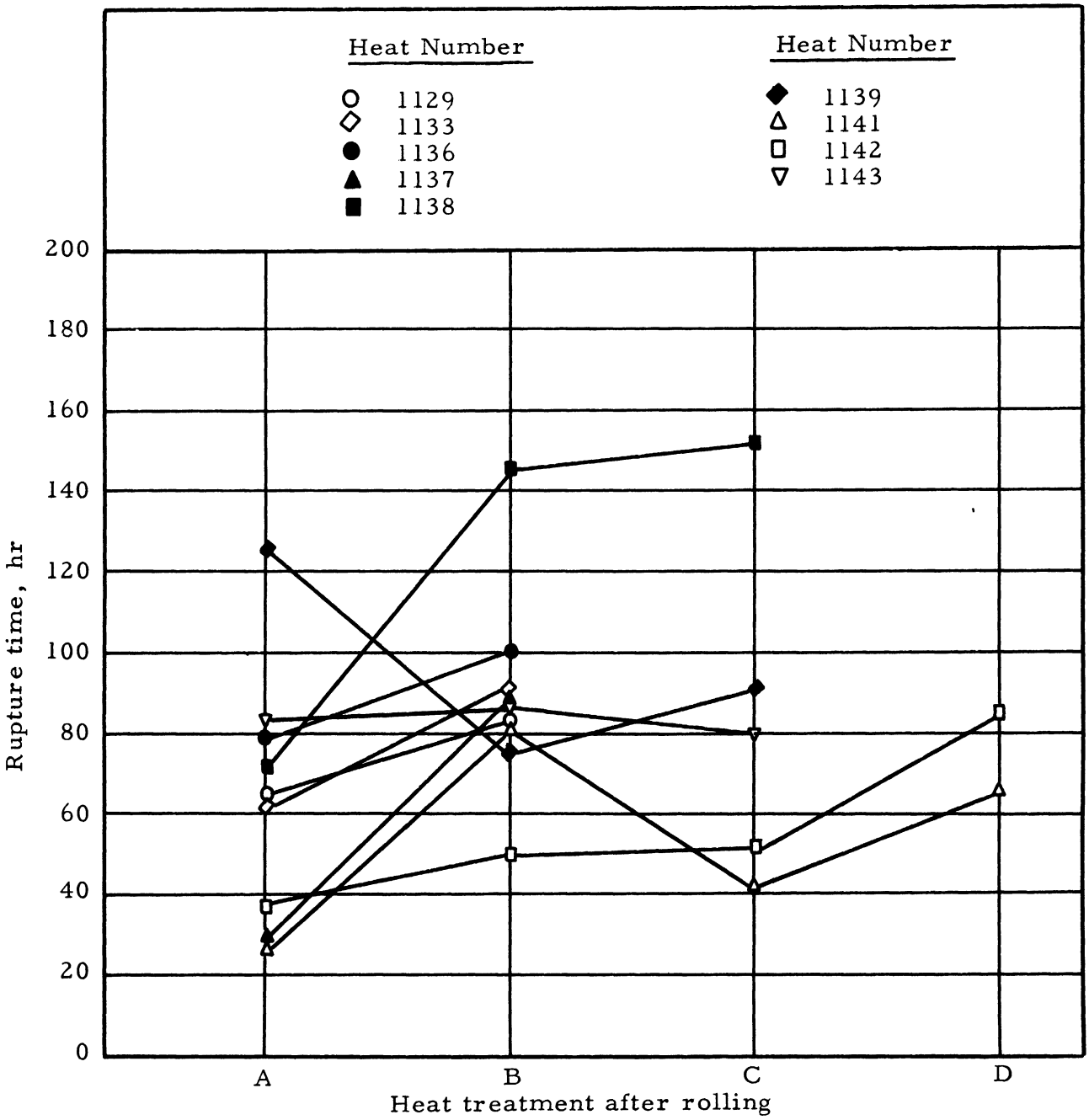


Figure 11.- Effect of heat treatment after rolling on the rupture time at 1600°F and 25,000 psi for several heats. All heats processed by "standard" rolling procedure. Heat treatments as follows:

A- 4 hours 1975°F, air-cooled
B- 2 hours 2150°F, air-cooled
C- B+A+ age 24 hours 1550°F, plus 16 hours 1400°F
D- B+ age as in C.

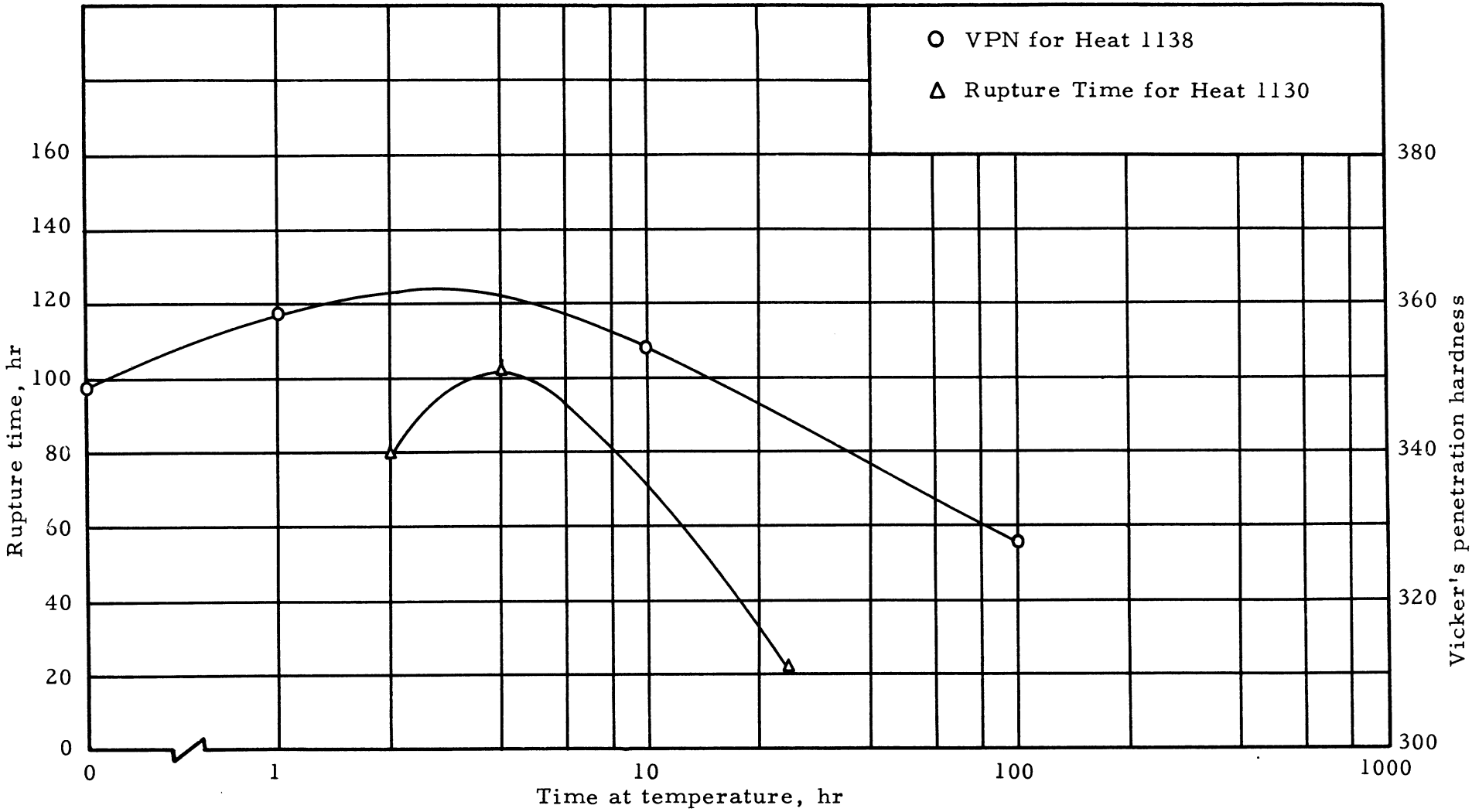


Figure 12. - Influence of time at temperature before applying stress on the rupture time at 1600°F and 25,000 psi for Heat 1130 compared with the time-hardness curve of Heat 1138 at 1600°F.

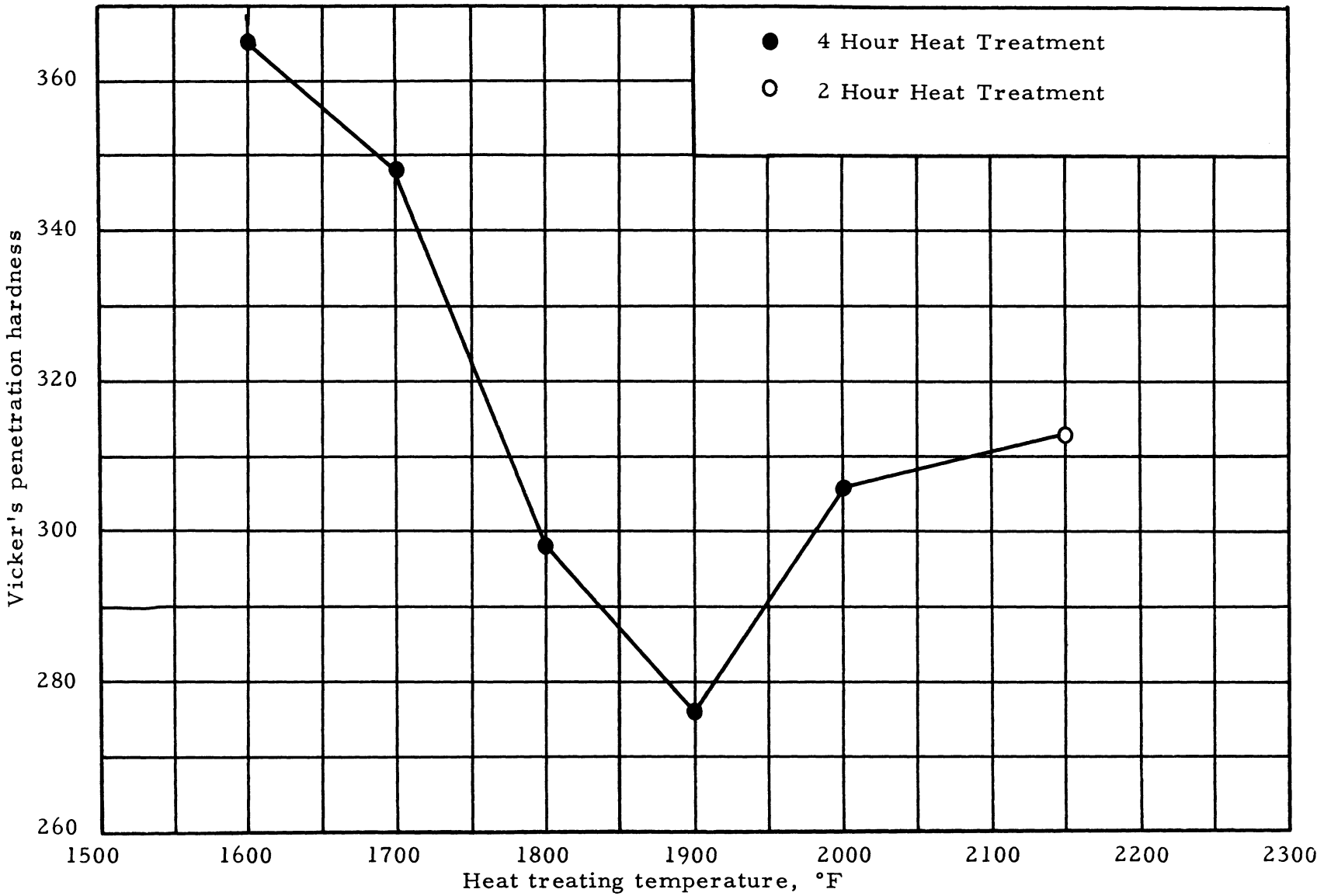
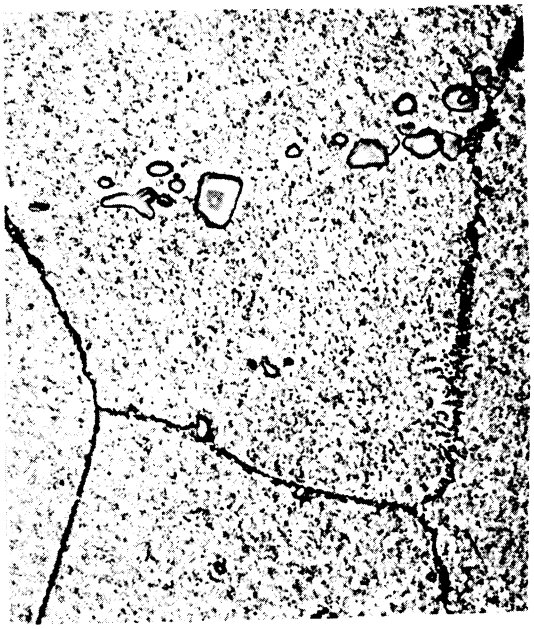


Figure 13. - Effect of heat treating temperature on hardness of Heat 1138. Rolled bar stock was annealed 2 hours at 2150°F and ice-brine quenched prior to heat treatment. Samples were ice-brine quenched after heat treatment.

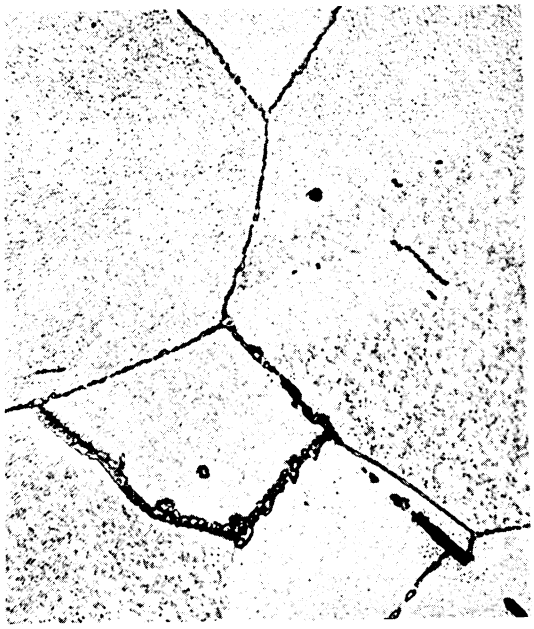
X1000D

(a) 1700°F



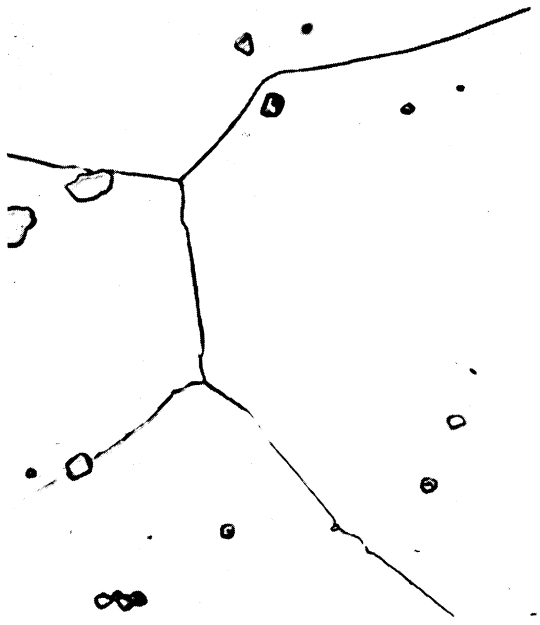
X1000D

(b) 1800°F



X1000D

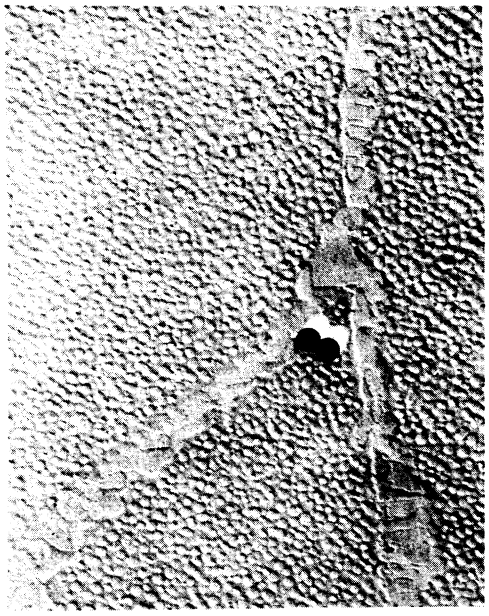
(c) 1900°F



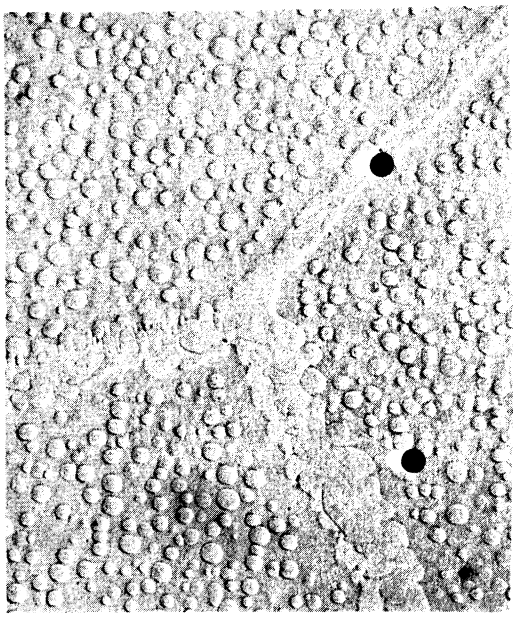
X1000D

(d) 2000°F

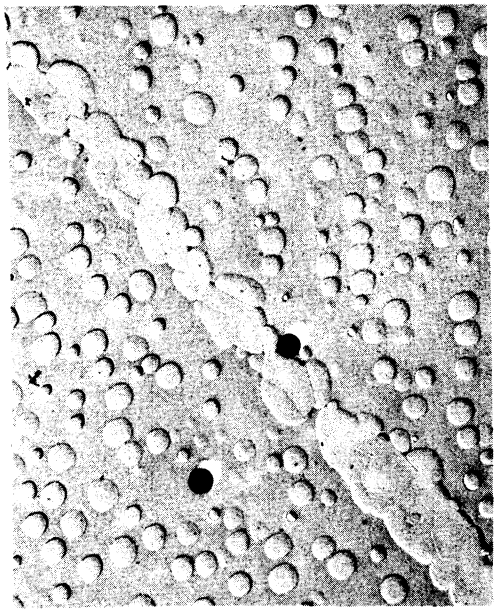
Figure 14. - Effect of 4 hour heat treatments at designated temperatures on microstructure of Heat 1138. Rolled bar stock was annealed 2 hours at 2150°F prior to heat treatment. Samples were ice-brine quenched after heat treatment.



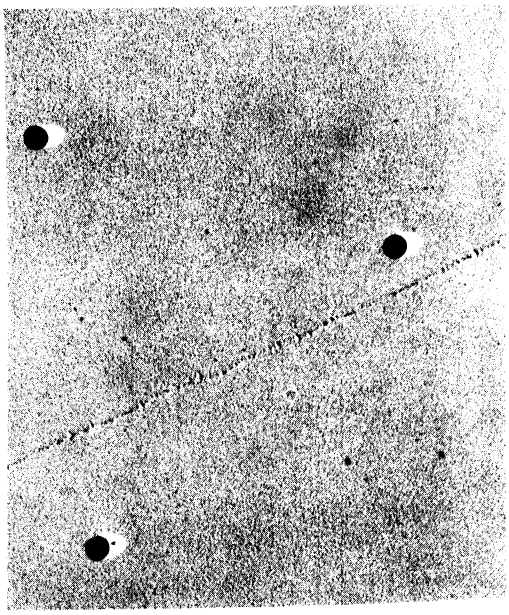
X13,000D
(a) 1700°F



X13,000D
(b) 1800°F



X13,000D
(c) 1900°F



X13,000D
(d) 2000°F

Figure 15.- Effect of 4 hour heat treatments at designated temperatures on microstructure of Heat 1138. Rolled bar stock was annealed 2 hours at 2150°F prior to heat treatment. Samples were ice-brine quenched after heat treatment. Electron micrographs.

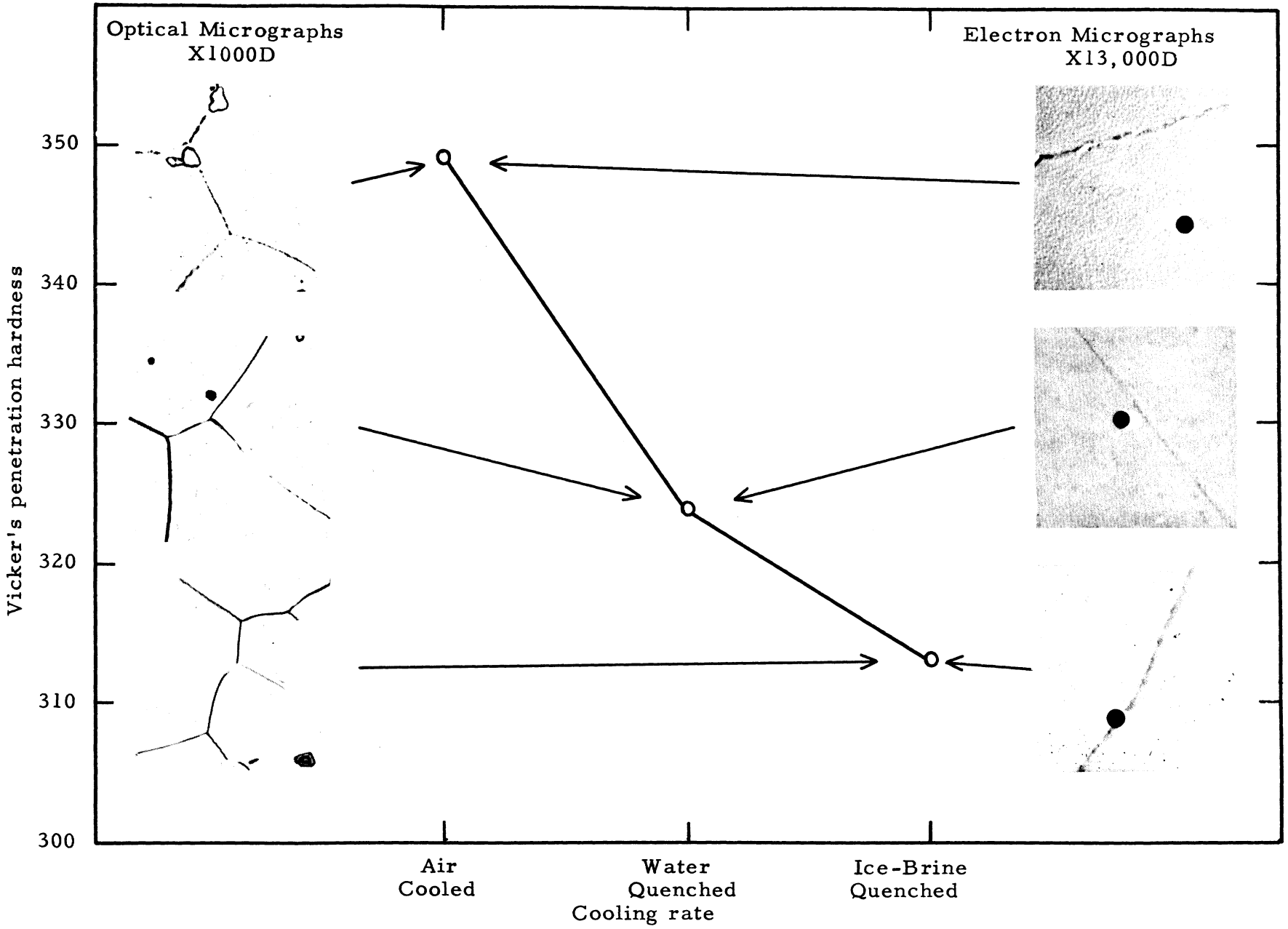


Figure 16. - Effect of cooling rate after 2 hours at 2150°F on hardness and microstructures of Heat 1138.

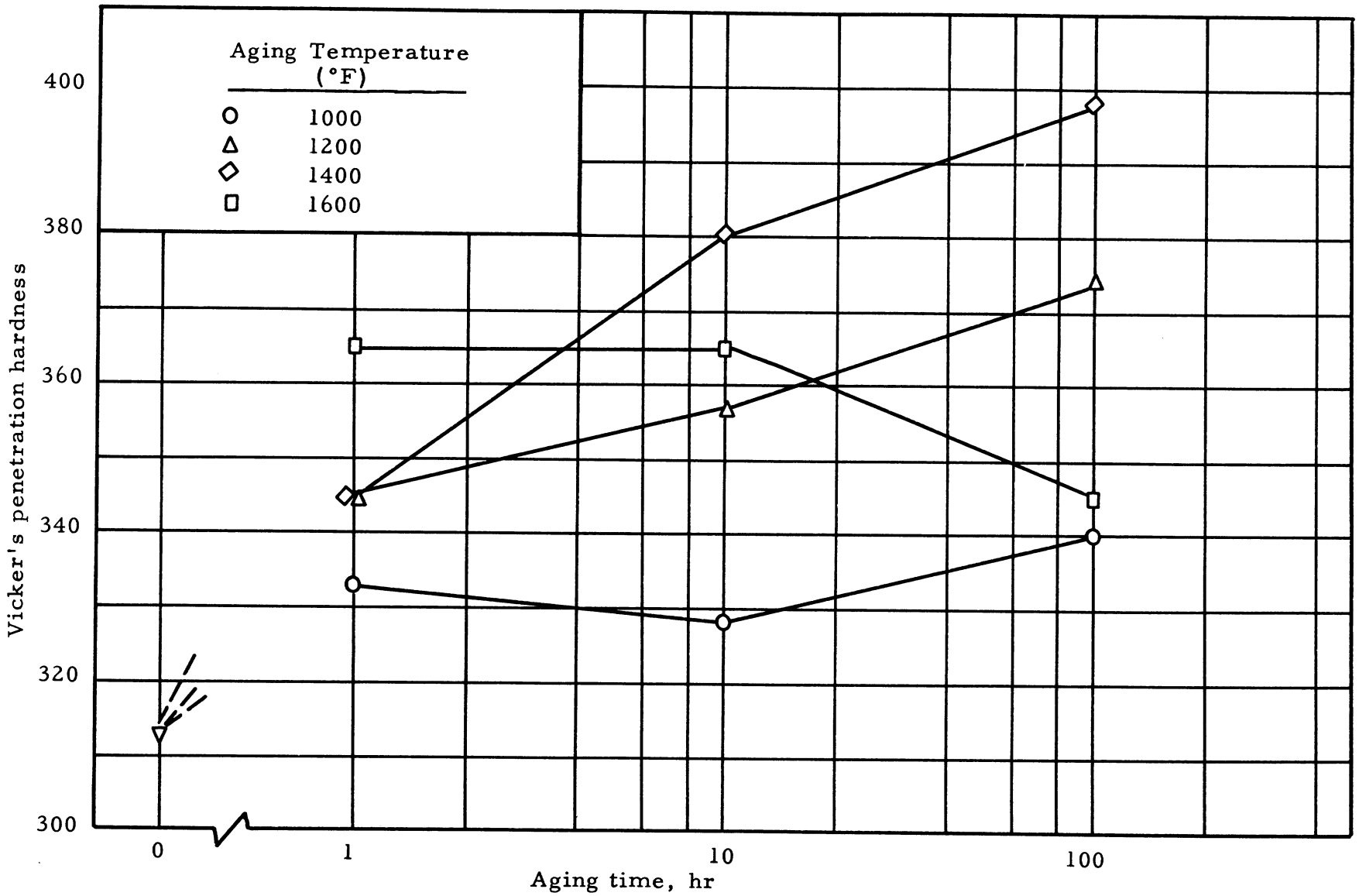


Figure 17. - Effect of aging temperature and time on the hardness of Heat 1138. Treatment prior to aging was 2 hours at 2150°F, ice-brine quenched.

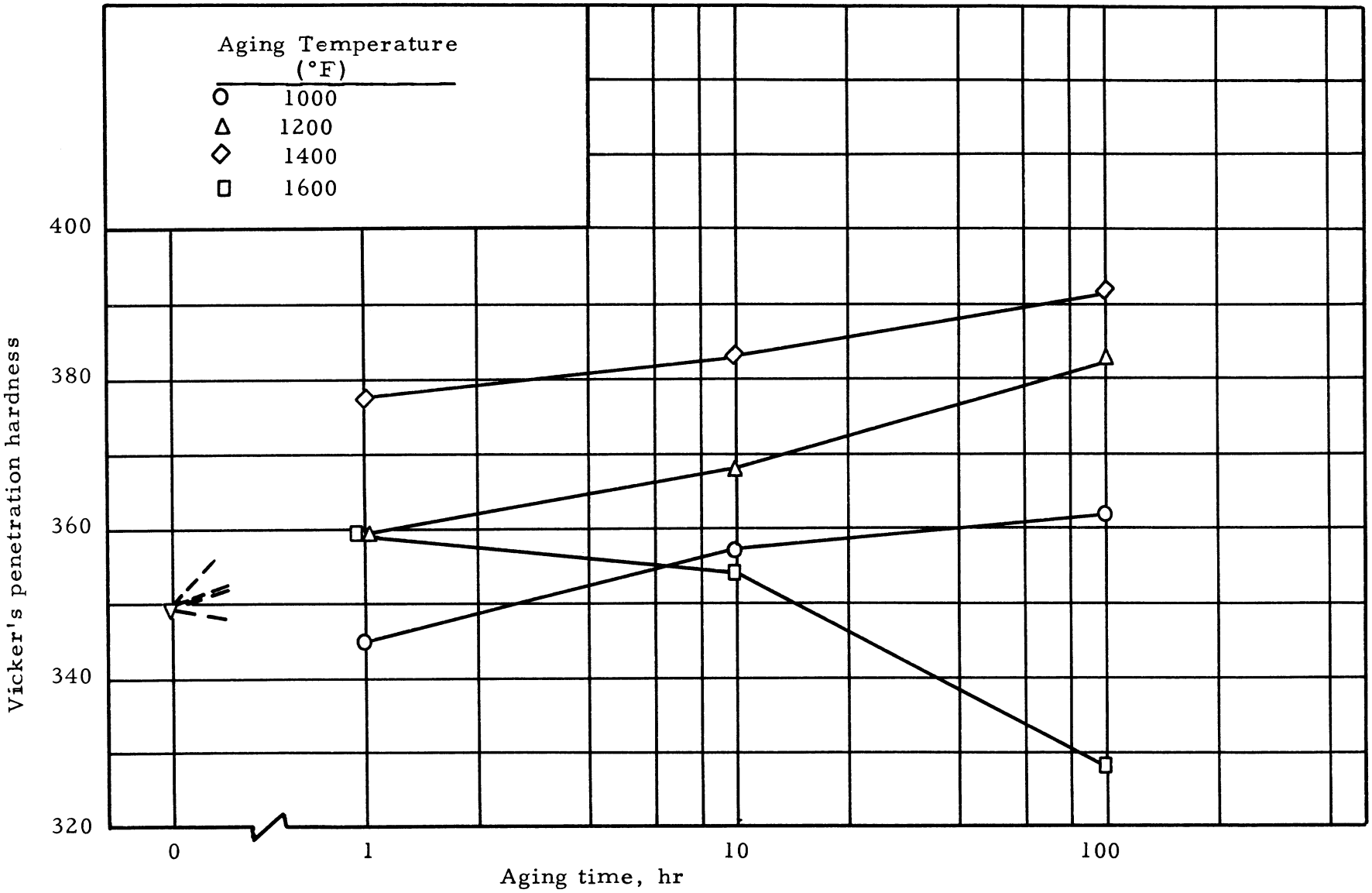
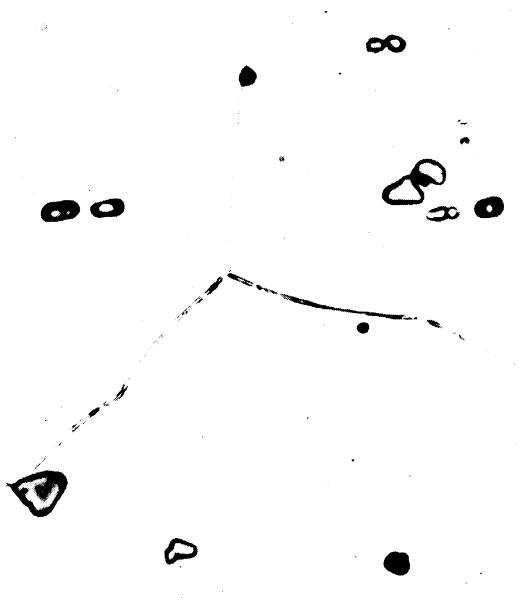


Figure 18. - Effect of aging temperature and time on hardness of Heat 1138. Treatment prior to aging was 2 hours at 2150°F, air-cooled.

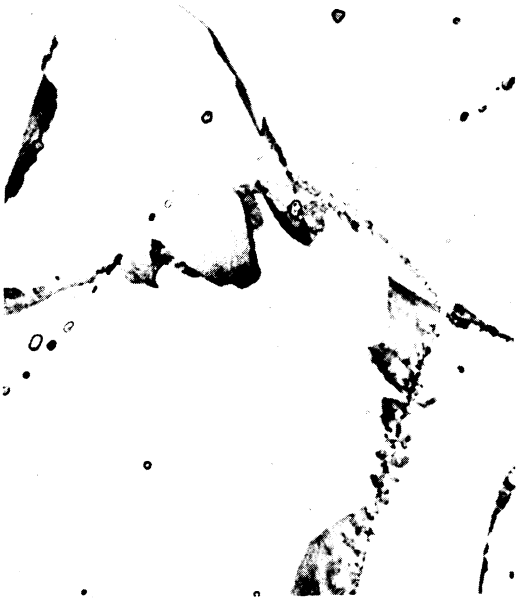


X1000D

(a) 1000°F

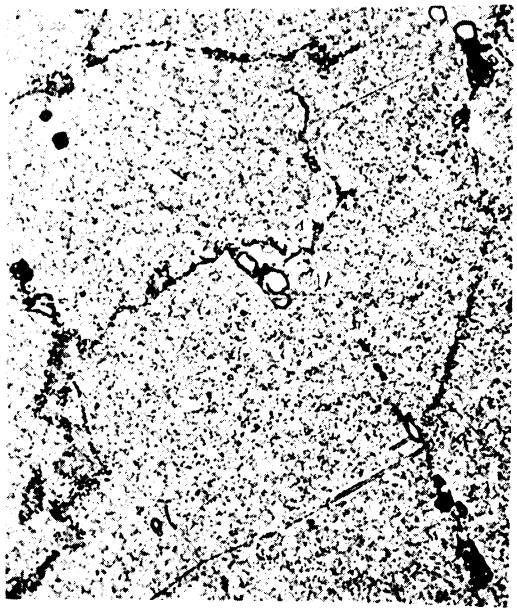
X1000D

(b) 1200°F



X1000D

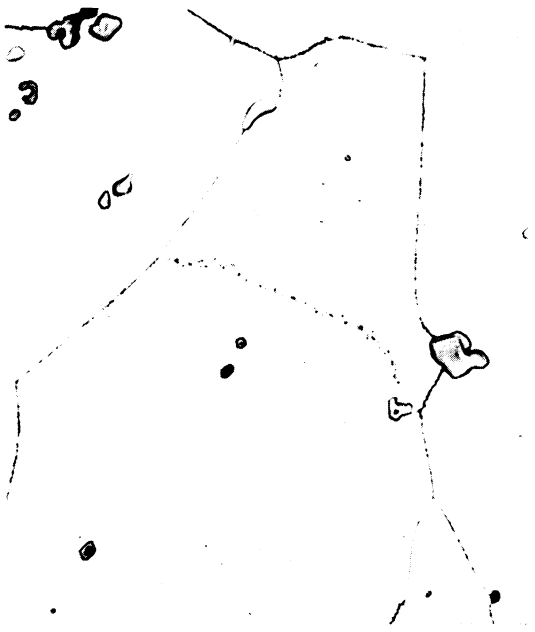
(c) 1400°F



X1000D

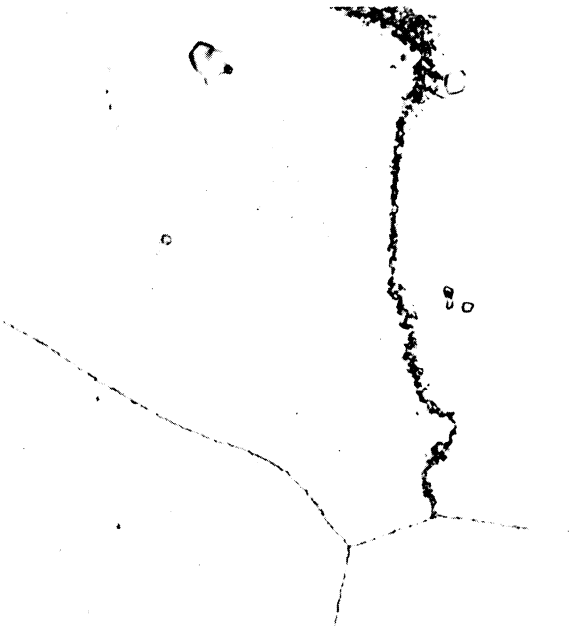
(d) 1600°F

Figure 19. - Effect of 100 hour aging at designated temperature on the microstructure of Heat 1138. Initial treatment was 2 hours at 2150°F, ice-brine quenched.



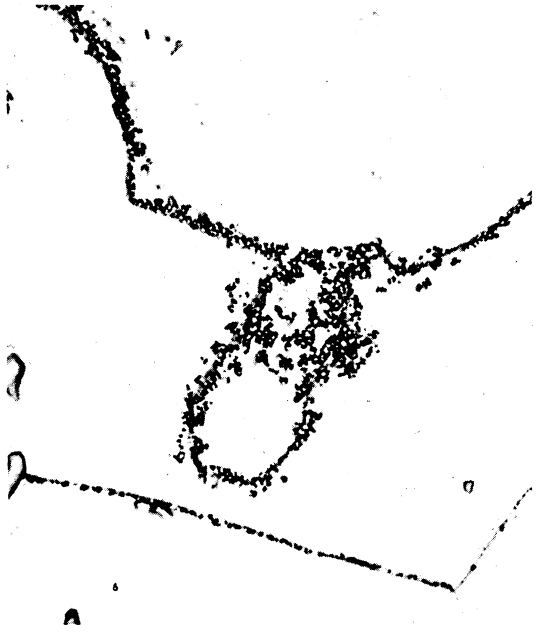
X1000D

(a) 1000°F



X1000D

(b) 1200°F



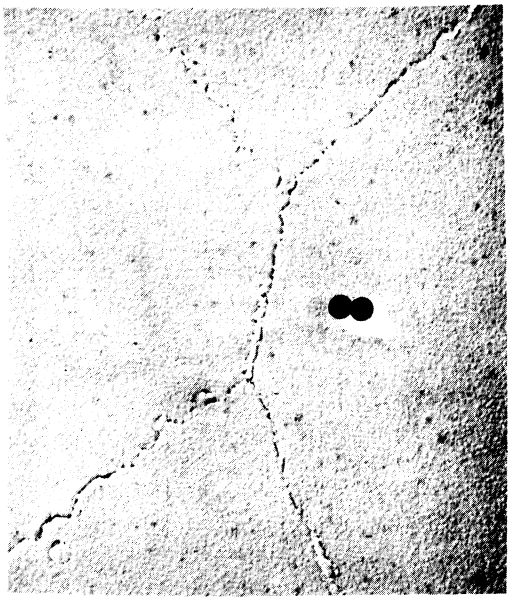
X1000D

(c) 1400°F

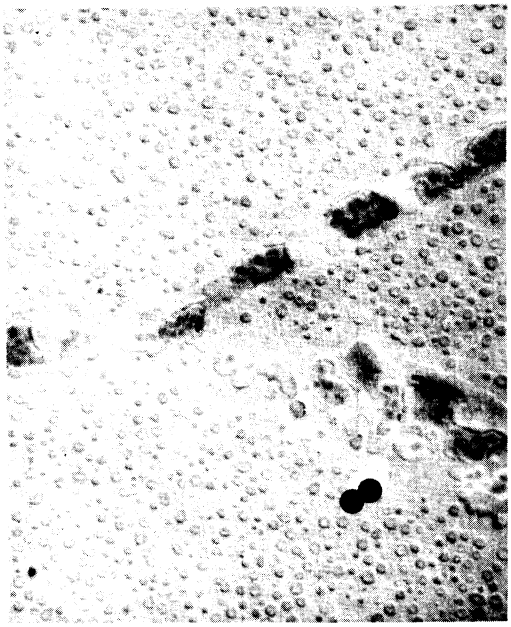
X1000D

(d) 1600°F

Figure 20. - Effect of 100 hour aging at the designated temperature on the microstructure of Heat 1138. Initial treatment was 2 hours at 2150°F, air-cooled.



X13,000D
(a) Aged 1 hour at 1200°F



X13,000D
(b) Aged 100 hours at 1600°F

Figure 21. - Effect of aging on the microstructure of Heat 1138. Initial treatment was 2 hours at 2150°F, air-cooled. Electron micrographs.