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PROGRESS REPORT

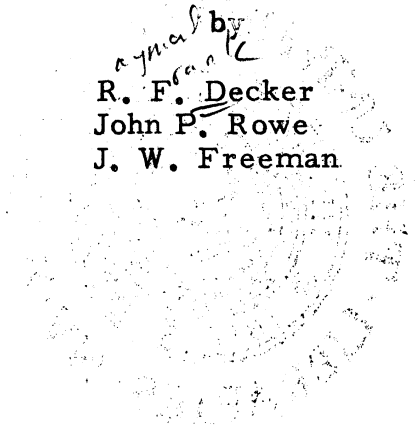
TO THE

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

COVERING

RESEARCH ON HEAT-RESISTANT ALLOYS

Submitted



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The examination of structures with the electron microscope has been a joint endeavor with Professor Wilbur Bigelow and his associate, Mr. J. Amy. Professor Bigelow's work is sponsored by the Metallurgy Research Branch of the Aeronautical Research Laboratory, WADC. Samples were prepared, etched and examined under guidance of Professor Bigelow. In addition, he will use the samples to improve techniques for phase identification and the results will become available to the NACA research. The excellent techniques developed by Professor Bigelow are now being standardized and adopted by metallurgists for more routine studies. Particular acknowledgments are due to Professor Bigelow for his excellent work in developing the techniques and phase identification procedures.

The contribution of a number of people working on this project is acknowledged. Mr. Karl Kienholz contributed greatly to operating the vacuum furnace. Mr. Alex Dano did excellent optical and electron micrographic work. His development of etching procedures for macrostructures and optical micrography aided greatly in the study. Miss Christine Sadler has ably prepared metallographic samples and photographs. Mr. Jerry White assisted with much of the routine heat treating and testing. Mr. Ted Spence performed much of the overheat testing.

alloy as the experimental material. Original limited objectives were to define the role of O_2 and N_2 with some work on minor additions such as B.

The investigation has developed along the following lines:

1. Various conditions of "deoxidation" with varying superheat and pouring temperatures have been studied, mainly in the vacuum furnace.

2. Variations in melting practice resulted in rupture times ranging from 21.9 to 147.4 hours at 1600°F and 25,000 psi. Generally the ductility was low. It had been originally indicated that the alloy ought to have rupture times between 175 and 500 hours. Considerable effort was expended to try to produce these values. At present the characteristic values for the alloy are not clear and recent information indicates that apparently the values of 175 to 500 hours are high and the characteristic values ought to be 30 to 80 hours at 1650°F and 25,000 psi which converts to 100 to 290 hours for our test conditions of 1600°F and 25,000 psi. Considerable effort was expended attempting to produce rupture times in excess of 175 hours at 1600°F and 25,000 psi. From this research certain patterns have emerged:

(a) The variations in rupture time obtained by melting practice correlate better with the amount of Zr picked up through the use of a zirconia crucible than any other factor. The high Zr heats gave rupture times in excess of 100 hours.

(b) Rupture times in excess of 600 hours were obtained by a small B addition. B plus Zr increase both strength and ductility and compliment each other.

(c) There may be considerable benefit from the use of a magnesia crucible.

(d) The lowest strength and ductility have been obtained by the use of an alumina crucible with no pickup of Zr, B or Mg.

3. The study of hot workability has been restricted to observations during rolling of the ingots. The following comments are not fixed and merely represent inconclusive observations to date. High C content improves hot workability. Either B or Zr reduced surface cracking. The limited experience to date indicated that the presence of both B and Zr leads to internal rupture during ingot breakdown. The B heats were also subject to center breaks.

4. A study of solution treating and aging of the alloy has been carried along so that the observed melting effects could be correlated with basic structural conditions. This has included optical and electron metallographic studies of the structures, particularly the $Ni_3(Al\ Ti)$ precipitate dispersion, and hardness changes. This work is still inconclusive. The general precipitate effects have not been as pronounced as might be expected. In addition, a structural condition in the grain boundary has been found which seems to always be present when weak, brittle heats are produced.

Introduction

The study of melting-practice influences on properties at 1600°F under 25,000 psi stress and hot-working characteristics has been continued since June 1956 when the last previous progress report was submitted. At the same time a study of the structures of the alloy has been continued to provide a basis for explanation of the melting-practice variables. Heat-treatment effects are one of the variables included in the study.

Except for one air-melted heat, melting was restricted to vacuum with no introduced gases. Vacuum melting was carried out in the University of Michigan vacuum furnace where pressures before meltdown and after pouring were less than 5 microns as measured by both Stokes and thermocouple gages. The air

heat was melted in an induction furnace. Melt temperatures were measured with a Pt - Pt + Rh immersion thermocouple.

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

Cr	Co	Mo	Ti	Al	Mn	Si	C	Ni	B and Zr
20.0	15.0	4.00	3.10	3.10	0.12	0.12	0.08-0.15	Bal	Varied

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

Melting cycles for the heats are pictured in figure 1. Table I lists the details of melting practices.

Ten-pound heats were poured into a massive copper mold.

Hot-working practice was kept constant for all the heats used in the study of melting practice except for the air heat. This constant 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.

The air-heat ingot was not homogenized or ground and was rolled at 2150°F to 5/8-inch bar stock.

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 II.

Evaluation of high-temperature properties of the heats was made after the as-rolled stock was heat treated. Rupture samples were preheated 4 hours at 1600°F and tested at 25,000 psi and 1600°F. The results are tabulated in Table III.

Variation of High-Temperature Properties with Melting Practice

Several melting variables were investigated. These were melting atmosphere, time and temperature of refining period, deoxidant, superheat temperature, pouring temperature, crucible material and ingot mold shape.

Comparisons of high-temperature properties to evaluate the melting effects were based on the materials with a heat treatment at 2150°F with air cooling.

The following comparisons are valid:

1. Effect of refining time.
   Crucible: Zirconia
   Superheat temperature: 2900° - 3050°F
   Pouring temperature: 2900° - 3000°F
   Atmosphere: vacuum

Heat	Zr (%)	Refining Time	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1129	.09	0 (C in charge)	82.3	--	4.7
1130	.08	0 (C in charge)	117.7	8.0	3.2
1133	.04	20 minutes at 2700°F (50% C in charge)	90.7	4.0	4.0
1136	.06	20 minutes at 2700°F (no C in charge)	99.5	6.0	4.0

The change in refining time did not result in any significant variations.

2. Effect of refining temperature.
Crucible: Zirconia Pouring temperature: 3000°F
Superheat temperature: 3100°F Atmosphere: vacuum
Deoxidation: Al after refining, no carbon in charge.

Heat	Zr (%)	Refining Time	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1138	.19	20 minutes at 2700°F	147.4	5.0	4.6
			133.7	6.1	7.9
1143	.06	20 minutes at 2900°F	85.5	---	9.3
			86.5	8.5	10.0

As will be shown later, the high Zr pickup of heat 1138 was the significant variation.

3. Effect of deoxidant.
Crucible: Zirconia Pouring temperature: 2900° - 3000°F
Superheat temperature: 2900° - 3100°F Atmosphere: vacuum
Refining treatment: 20 minutes at 2700°F, no carbon in charge.

Heat	Zr (%)	Deoxidant	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1136	.06	Chunk C	99.5	6.0	4.0
1138	.19	Al	147.4	5.0	4.6
			133.7	6.1	7.9
1139	.03	Si	75.5	4.0	3.1

Again the variation can be attributed to Zr pickup.

4. Effect of superheat and pouring temperature.
Crucible: Zirconia Atmosphere: vacuum
Refining treatment: 20 minutes at 2700°F, no carbon in charge.
Deoxidant: Al

Heat	Zr (%)	Superheat Temperature (°F)	Pouring Temperature (°F)	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1138	.19	3100	3000	147.4	5.0	4.6
				133.7	6.1	7.9
1141	<.03	2975	2750	81.2	8.0	11.5
1142	<.03	2750	2750	50.8	8.0	12.3

The variation can be assigned to differences in Zr pickup.

5. Effect of melting atmosphere (rolling practice was varied).
Superheat temperature: 2980 - 3000°F Pouring temperature: 2980° - 3000°F
Refining treatment and deoxidation: C in charge

Heat	Zr (%)	Crucible	Atmosphere	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1146	<.01	Alumina	Vacuum	56.9	1.9	1.6
UA3	<.01	Zirconia	Air	21.9	1.0	1.1
				24.0	4.9	<1.0

Although the rupture time of heat 1146 is higher, the difference is not clearly significant.

6. Effect of crucible material.
Superheat temperature: 3000° - 3050°F Pouring temperature: 3000°F
Refining treatment and deoxidation: C in charge

Heat	Zr (%)	Crucible	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1129	.09	Zirconia	82.3	--	4.7
1130	.08	Zirconia	117.7	8.0	3.2
1144	<.01	Magnesia	185.2	3.0	2.0
1146	<.01	Alumina	56.9	1.9	1.6

Zr pickup from the zirconia crucibles increased rupture time and ductility.

There is some evidence that the magnesia crucible was beneficial, although the effect might be caused by the high Al of heat 1144.

7. Effect of ingot mold shape.
Superheat temperature: 2900° - 2950°F Pouring temperature: 2900° - 2950°F
Refining treatment: 20 minutes at 2700°F, no carbon in charge.
Deoxidation: Chunk C Crucible: Zirconia

Heat	Zr (%)	Ingot Mold	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1136	.06	Straight	99.5	6.0	4.0
1137	.09	Tapered	87.1	6.0	6.4

No significant effect of ingot mold shape is evident.

Chemical analyses of the heats used in the study of melting variables indicated that variable pickup of Zr from the crucible occurred (see Table II). Figure 2 shows that the correlation of Zr pickup with rupture life removes much of the variance from the data.

Random heat-to-heat variations of rupture life with constant melting practice and random variations during testing of several samples from one heat are being thoroughly analyzed. Preliminary data indicate that these two variations might account for the scatter of values around the curves of correlation.

This information reveals that the melting variables studied affected rupture life mainly by virtue of their effect on Zr pickup from the crucible.

Because of the random variations, further testing on existing heats and duplication of existing heats would be required to establish the less pronounced effects of the melting variables other than Zr pickup.

The relatively high-rupture life of heat 1144, which was melted in a magnesia crucible, suggests a beneficial effect of the crucible. However, chemical analysis did not show Mg pickup and indicated that the Al content was high. Therefore, further experimental work will be required for confirmation of the beneficial effect.

Two significant effects are apparent in the ductility results. First, high carbon levels in heats 1141 and 1142 apparently resulted in higher ductility. Secondly, the ductility increased with Zr content (see figure 3).

Influence of Additions of B and Zr

Test Results

A series of three heats were melted to obtain varying amounts of B and Zr as follows:

Heat	Crucible	B added (percent)	Zr pickup (percent)	Rupture Life (hours)	Elongation (percent)	Reduction of Area (percent)
1145	Magnesia	.01	<.01	428.8	10.0	11.3
1146	Alumina	none	<.01	56.9	1.9	1.6
1147	Zirconia	.01	.01	666.3	17.0	15.5

In figure 4, rupture times are plotted on a rupture band obtained from Utica Drop Forge and Tool Corporation.

Time-elongation curves for heats 1138, 1145, 1146 and 1147 are shown in figure 5. It can be observed that addition of Zr alone resulted in higher minimum creep rate than that of the base alloy, while B or B + Zr additions decreased the minimum creep rate slightly.

Primary creep was relatively independent of B and Zr content.

The main difference in the results seems to be that B and B + Zr additions reduced rate of increase of third stage creep and lengthened its duration.

Both Zr and B markedly increase the stress-rupture ductility. The effect seems to be most pronounced when they are both present in the alloy.

The results of short-time tensile tests on heats 1145 (melted in magnesia with B addition) and 1146 (melted in alumina with no additions) are plotted in figure 6. It is evident that a brittle fracture occurred at very low total elongation

with heat 1146. The sample from heat 1145 exhibited a higher tensile strength and more elongation before fracture.

Observation was made of cracking tendency of the ingots containing B and Zr. Little surface cracking occurred during rolling of the ingots containing either B or Zr alone (see figure 7). However, some centerline voids were found in the finished bar stock, indicating that internal cracking had occurred during ingot breakdown.

Ingots containing both B and Zr cracked severely during ingot breakdown (see figure 8). As-cast microstructures revealed the presence of additional phases in these ingots (see figure 9).

Structural Studies

Preliminary studies have been started to determine the metallurgical mechanism by which B and Zr increase high-temperature strength and ductility.

The plan is to compare the structures of B and Zr bearing heats with the base-alloy heats. It is felt that the trace elements could have their effect through one or both of the following mechanisms:

1. By increasing the effectiveness of general precipitation of $Ni_3(Al\ Ti)$ in the matrix, thereby retarding creep or decreasing the tendency for brittle fracture.

2. By changing localized precipitation at the grain boundaries to retard the onset of brittle fracture.

The first objective is to obtain quantitative comparisons of general precipitate in the heats. Hardness measurements have been taken on aged samples for comparison purposes. These are shown in figures 10 through 12. The following observations can be made from the hardness curves:

1. At 1200° and 1400°F, the precipitation hardening is slower in heat 1145 (with B) than in the base alloy (heat 1146). However, the B alloy hardens to about the same level after 10 hours at 1600°F.

2. In heat 1138 (with Zr), precipitation does not provide as much hardening as in the base alloy. Also, hardness reaches a lower level after long time aging at 1600°F.

3. The aging curve for heat 1144 (high Al alloy melted in magnesia) is relatively flat. Evidently this heat is more resistant to overaging than the other heats.

4. The heat with both B and Zr (heat 1147) hardened in a similar manner to the base-alloy heat(1146).

Light microstructures have not revealed any significant differences in the rolled or heat-treated samples. Electron microstructures of aged and rupture-tested samples will be used to check differences in the general precipitate in the matrix.

After the kinetics of general precipitation are evaluated, it is planned to observe the effect of B and Zr on localized precipitation at the grain boundaries. Figure 13 illustrates that exposure to stressing at 1600°F has a marked effect on a portion of the grain boundaries of the Zr bearing heat (1138). While some of the grain boundaries resemble those obtained with stress-free aging, the remainder are heavily overaged. It is possible that B and Zr additions retard this severe overaging.

Influence of Heat Treatment

Effect of Solution-Treating Temperature

Rupture data included in the progress report of June 30, 1956 indicated that solution treatment at 2150°F resulted in generally higher rupture strengths than the treatment at 1975°F. Further confirming data on heats 1138, 1144,

1145, 1146 and 1147 were obtained and are included in figure 14.

Microstructural examination of many of the heats after rolling and after subsequent heat treatments at 1975°F revealed that, with most of the heats, the duplex grain structure was retained after heat treatment (see figure 15).

In addition, the 1975°F treatment did not completely dissolve the Ni_3 (Al Ti) precipitate (see figure 15). The large particles appearing in the electron micrograph are undissolved Ni_3(Al Ti). It is not clear at this time if the inferior properties result from the incomplete solution effects or from the residual rolling effects.

Effect of Cooling Rate After Solution Treatment

Structural studies of heat 1138 (high Zr) disclosed that cellular precipitation occurred during aging after ice-brine quenching from solution treating (see figure 16). The presence of the precipitate was accompanied by brittle fracture upon loading for rupture testing.

Generally, the high-temperature properties of alloys susceptible to cellular precipitation are improved by furnace cooling after solution treatment. To investigate the effect, samples from heats 1138, 1144, 1145, and 1146 were solution treated 2 hours at 2150°F, furnace cooled and rupture tested at 25,000 psi and 1600°F. The results, listed in Table III and plotted in figure 15, indicate that the expected improvements were not obtained except with heat 1144. The ductility was improved, however.

Structural Studies of Zr Bearing Heat 1138

A structural study of the effect of heat treatment and rupture testing on the precipitates in heat 1138 (with Zr) is nearing completion. The results are now being prepared in the form of a technical report to be published in the near future.

EFFECTS OF OVERHEATING ON CREEP-RUPTURE PROPERTIES OF BLADE ALLOYS

Previous studies of the effects of brief overheats during creep-rupture tests at 1500°F had shown two types of effects, as described in the technical reports submitted. The major effect shown was increasing reduction in life at 1500°F for S-816 and HS-31 alloys from increasing numbers of overheats and increasing temperature of overheating from 1600° to 2000°F. M-252 alloy, on the other hand, underwent pronounced increase in life at 1500°F from overheats to 1900° and 2000°F with some loss for 1600° and 1800°F.

These results were discussed with the Subcommittee at the last meeting. It was pointed out that:

1. The strengthening overheating for M-252 should be checked on another Ti + Al hardened alloy to see if it is characteristic of all such alloys. Inconel 700 alloy was agreed upon for this check.

2. That the effects had been checked for rather limited conditions, the effect on creep-rupture strength at 1500°F from 2-minute overheats on fixed cycles of every 5 or 12 hours. It was felt that erroneous conclusions could be derived from the report unless some checks of the following were made:

(a) Other test temperatures than 1500° - 1600°F and 1350°F should be checked.

(b) Overheat temperatures ought to be checked for temperatures above 2000°F.

(c) The influence of duration of overheats and cycle frequency ought to be checked.

3. Heat-to-heat variations changed the magnitude of the response of the alloy to overheating.

4. The most important additional work should be to establish the metallurgical mechanism for the overheat effects. This is considered most vital because it will place all the results on a general basis.

Test Materials

The M-252 stock used for the tests covered by the technical report submitted was exhausted. Another lot of stock, Heat 837 furnished by The General Electric Company, made from a vacuum-melted heat was available. With the experience gained from the previous work it was felt that this would provide suitable material.

The Wright Aeronautical Division, Curtiss-Wright Corporation supplied bar stock of Inconel 700 Alloy.

The reported chemical analyses of the two heats are given in Table IV.

Results for M-252 Alloy

The first step was to establish a suitable processing procedure and typical properties for the stock from Heat 837. Overheat tests have been started using base-test temperatures of 1500° and 1600°F. The heat of material selected for this work was subjected to several processing conditions in order to select that which would be the most suitable for the purpose at hand. Using the results of the work reported previously, the following program was set up:

1. Determine the effect of rolling the bar stock at 1950° or 2150°F.

2. Determine the effect of including a "mill anneal" of 1 hour at 2150°F after rolling, prior to the standard heat treatment of 4 hours at 1950°F, air cool, followed by 15 hours at 1400°F.

These two variables resulted in four conditions of the material which were subsequently rupture tested at 1500°F and 34,000 psi with the following results:

Rolling Temperature (°F)	Rupture Time (hours)	
	Standard Heat Treatment	Mill Anneal plus Standard Heat Treatment
1950	34.2	66.6
2150	30.3	60.1

These results bore out the fact that the inclusion of a 2150°F mill anneal before the standard heat treatment resulted in a substantial increase in the strength of the material in a normal stress-rupture test. Since the strength of the alloy did not seem to depend appreciably on the rolling temperature, and the previous work had been done on material rolled at 2150°F, it was decided to select this condition of the alloy for the present investigation.

Rupture Test Results

After the material was rolled and heat treated as described above, it was necessary to establish the stress-rupture time curves at 1500° and 1600°F. The results of these tests are included in Table V and are plotted as stress-rupture time curves on figure 17a. When the stress desired for the overheat work was established, duplicate tests were run at this stress to give a check on the variability of the rupture time at a single stress. It was found that duplicate tests gave results which were in very close agreement with each other.

Also included on figure 17a is the average curve at 1500°F for the material used in the previous investigation as well as that considered "typical" of the alloy. It can be noted that the present data fall slightly below that for the former heat and slightly above the normal curve at short times. At longer times the present heat falls below either of the other two curves.

Overheat Test Results

The results for overheating Heat 837 are given in Table VI and shown on figure 18. These data show that for overheating to 2000°F with the temperature between overheats either 1500° or 1600°F, there was an increase in the rupture time as a result of the overheating. When the overheating was carried to 1800°F with a base temperature of 1500°F, there was little effect on the rupture time. The only positive result for the overheats to 1800°F is that there was no damage to this heat. Also included in Table VI is one data point taken from the previous work. This is for the same heat of M-252 rolled and solution treated at 1950°F. This material when overheated to 1900°F in the absence of stress showed about the same degree of improvement in life as did the material overheated to 2000°F after being rolled at 2150°F and given a double solution treatment for the present investigation. All of the overheating for these tests was done at approximately five-hour intervals with two minutes at the overheat temperature for each cycle.

Comparison with the data previously reported which has been replotted on figure 18, indicates that there is a slightly different response to overheating to either 1800° or 2000°F shown by Heat 837 than was given by Heat HT-28 in the previous work. The extent of the improvement in life is not as great for overheating to 2000°F, while overheating to 1800°F apparently had little or no influence on Heat 837 as opposed to the damage to Heat HT-28 which was caused by this temperature. The single test completed to date indicates that the same type of response is obtained when the base-test temperature is 1600°F, as was obtained with 1500°F as a base temperature.

Results for Inconel 700 Alloy

The Inconel 700 stock was heat treated as-received by heating 2 hours at 2160°F, air cooling and aging for 4 hours at 1600°F.

Standard rupture tests were conducted at 1600°F, figure 17b, and are in progress at 1500°F. A stress of 29,000 psi at 1600°F to give a normal rupture time of 90 hours was selected for the first overheat experiments.

Overheat Test Results

Overheat tests were run on samples tested at 1600°F for overheat temperatures of 1800° and 2000°F (Table VI and figure 18). In both cases the rupture time for the overheated samples was less than the normal rupture time of 90 hours at 1600°F. The test overheated to 2000°F had the shorter rupture time of the two samples. It thus appears from these few preliminary tests that Inconel 700 alloy for the overheat conditions employed in these tests does not respond in the same way as M-252 alloy. The pronounced strengthening effect which was noted in the case of the M-252 is lacking for Inconel 700. The behavior exhibited is similar to that which was found for S-816 and HS-31 alloys as reported previously.

Discussion of Results

The differences between the two heats of the M-252 alloy are only qualitative in nature and are probably the result of the difference in response which would normally be expected between two heats of the same material. The only real difference between the present data and that previously obtained is the lack of damage to Heat 837 as a result of overheating to 1800°F.

The results on Inconel 700 alloy, on the other hand, indicate a major difference in the response of this alloy to overheating in the temperature range considered. All conditions investigated resulted in a decrease in the rupture life. In this case, the seeming anomaly may be the result of the testing procedures which were used. The overheats were all carried out using the procedures previously set up for use in conjunction with the 100-hour rupture stress. This

involved one overheat of two minutes duration approximately every five hours. It is possible that in the case of the Inconel 700 alloy, that this cycle is such that the response to the overheating is different than would be obtained with some other cycle. For example, if the effect of the overheating is to cause resolution of the precipitates which form during testing at 1500°F, the efficiency of the overheat is intimately connected with the kinetics of the precipitation reaction. Thus, the two minutes of overheating every five hours may not be sufficient to redissolve all of the precipitates which can form in five hours at 1600°F. In order to check on this possibility, it will be necessary to determine the effect of cycle frequency and duration on the response of both alloys to the overheating conditions being studied.

It appears now that it is more important to establish the mechanisms of the effects than it seemed before Inconel 700 alloy tests were started.

TABLE I

MELTING PRACTICE FOR EXPERIMENTAL HEATS

Heat	Crucible	Refining Time (minutes)	Refining Temperature (°F)	Deoxidant	Superheat Temperature (°F)	Pouring Temperature (°F)
1129	Zirconia	0	----	C in charge	3050	3000
1130	Zirconia	0	----	C in charge	3050	3000
1133	Zirconia	20	2700	C in charge + chunk C	3000	3000
1136	Zirconia	20	2700	Chunk C	2900	2900
1137*	Zirconia	20	2700	Chunk C	2950	2950
1138	Zirconia	20	2700	Al	3100	3000
1139	Zirconia	20	2 700	Si	2975	2975
1141	Zirconia	20	2700	Al	2975	2750
1142	Zirconia	20	2700	Al	2750	2750
1143	Zirconia	20	2900	Al	3100	3000
1144	Magnesia	0	----	C in charge	3000	3000
1145	Magnesia	0	----	C in charge	3000	3000
1146	Alumina	0	----	C in charge	3000	3000
1147	Zirconia	0	----	C in charge	3000	3000
UA3**	Zirconia	0	----	C in charge	2980	2980

* tapered ingot mold (other heats poured in straight ingot mold).

** air heat (other heats were vacuum heats).

TABLE II

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	Mg	S	P	Zr	B
1129	.05	.0006	.0003	.013	2.82	3.30	4.00	21.2	14.9	.12	.13	<.01	.021	.003	.09	none added
1130	.04	.0007	.0004	.010	3.17	3.45	4.00	21.7	14.6	.14	<.10	<.01	.025	.004	.08	none added
1133	.06	.0004	.0008	.005	3.22	3.35	4.00	20.7	14.8	.11	.16	<.01	.025	.005	.04	none added
1136	.05	.0003	.0005	.007	2.98	3.10	4.10	18.1	15.2	.10	.10	<.01	.021	.006	.06	none added
1137	.09	.0003	.0006	.005	3.18	3.15	4.20	18.2	15.1	.10	.10	<.01	.025	.008	.09	none added
1138	.08	.0012	.0004	.008	3.14	3.14	4.15	18.8	15.1	.10	<.10	----	.026 .008 (1)	.008	.19	none added
1139	.08	-----	-----	.006	3.30	3.35	4.10	19.4	15.0	.14	.15	----	.026	.006	<.03	none added
1141	.20	-----	-----	.006	2.98	3.00	4.20	19.2	14.5	.22	.15	<.01	.020	.007	<.03	none added
1142	.19	-----	-----	.006	2.93	2.85	4.10	19.8	14.5	.23	.13	<.01	.018	.004	<.03	none added
1143	.13	-----	-----	.005	3.05	3.15	4.10	19.8	15.2	.12	.10	<.01	.008	.005	.06	none added
1144	.05	-----	-----	.005	3.25	3.58	4.20	20.0	16.2	.18	.10	<.01	.018	.004	<.01	none added
1145	.10	-----	-----	.007	3.15	3.25	4.20	20.9	14.8	.20	<.10	<.01	.018	.004	<.01	.01 added
1146	.05	-----	-----	.007	3.25	3.37	4.20	20.4	14.8	.25	.12	<.01	.015	.007	<.01	none added
1147	.09	-----	-----	.009	3.20	3.30	4.20	20.8	14.8	.19	.11	<.01	.020	.007	.01	.01 added
UA-3	.13	-----	-----	----	3.15	3.36	3.90	20.0	16.7	.10	<.10	<.01	.018	----	<.01	none added

(1) check analysis at second laboratory.

TABLE III

STRESS-RUPTURE DATA AT 25,000 PSI AND 1600°F

Heat	Solution Treating			Aged	Rupture Time	Elongation	Reduction of Area
	Time (hr)	Temperature (°F)	Cooling Medium	24 hrs at 1550°F 16 hrs at 1400°F	(hours)	(percent)	(percent)
1129	4	1975	Air Cooled	none	63.9	5.0	8.0
	1	2150	Air Cooled	none	82.3	---	4.7
1130	4	1975	Air Cooled	none	102.2	2.0	5.6
	2	2150	Air Cooled	none	117.7	8.0	3.2
1133	4	1975	Air Cooled	none	62.1	6.0	7.9
	2	2150	Air Cooled	none	90.7	4.0	4.0
1136	4	1975	Air Cooled	none	79.0	12.0	16.7
	1	2150	Air Cooled	none	99.5	6.0	4.0
1137	4	1975	Air Cooled	none	29.3	13.0	18.4
	1	2150	Air Cooled	none	87.1	6.0	6.4
1138	4	1975	Air Cooled	none	71.7	7.0	7.9
	2	2150	Air Cooled	none	147.4 133.7	5.0 6.1	4.6 7.9
	2 4	2150+ 1975	Ice-brine Quenched	aged	Broke in loading	0	0

TABLE III (continued)

Heat	Solution Treating: Time (hr)	Solution Treating: Temperature (°F)	Solution Treating: Cooling Medium	Aged: 24 hrs at 1550°F 16 hrs at 1400°F	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1138	2	2150	Furnace Cooled	none	77.3	14.0	7.5
	2 4	2150+ 1975	Air Cooled	aged	152.9	3.0	5.6
	2	2150	Air Cooled	aged	127.6	5.0	3.2
1139	4	1975	Air Cooled	none	126.9	5.3	4.7
	2	2150	Air Cooled	none	75.5	4.0	3.1
	2 4	2150+ 1975	Air Cooled	aged	91.9	3.0	4.0
	2 4	2150+ 1975	Ice-brine Quenched	aged	Broke during machining	0	0
1141	4	1975	Air Cooled	none	28.2	7.0	9.0
	2	2150	Air Cooled	none	81.2	8.0	11.5
	2 4	2150+ 1975	Air Cooled	aged	42.1	9.7	11.7
	2	2150	Air Cooled	aged	66.9	8.5	10.0
1142	4	1975	Air Cooled	none	37.7	9.0	10.1
	2	2150	Air Cooled	none	50.8	8.0	12.3
	2 4	2150+ 1975	Air Cooled	aged	52.5	10.0	10.1
	2	2150	Air Cooled	aged	85.4	8.0	8.8

TABLE III (continued)

Heat	Solution Treating Time (hr)	Solution Treating Temperature (°F)	Solution Treating Cooling Medium	Aged 24 hrs at 1550°F 16 hrs at 1400°F	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
1143	4	1975	Air Cooled	none	82.5	5.7	11.5
	2	2150	Air Cooled	none	85.5	15.0	9.3
					86.5	8.5	10.0
	2	2150+	Air Cooled				
	4	1975		aged	80.6	11.5	13.0
1144	4	1975	Air Cooled	none	78.7	3.9	7.8
	2	2150	Air Cooled	none	185.2	3.0	2.0
	2	2150	Furnace Cooled	none	212.6	5.9	7.1
1145	4	1975	Air Cooled	none	249.7	17.1	24.6
	2	2150	Air Cooled	none	428.8	10.0	11.3
	2	2150	Furnace Cooled	none	317.8	11.8	14.5
1146	4	1975	Air Cooled	none	26.2	5.1	3.2
	2	2150	Air Cooled	none	56.9	1.9	1.6
	2	2150	Furnace Cooled	none	48.6	3.0	1.0
1147	4	1975	Air Cooled	none	325.5	23.3	25.6
	2	2150	Air Cooled	none	666.3	17.0	15.5
UA3	2	2150	Air Cooled	none	21.9	1.0	1.1
					24.0	4.9	<1.0

TABLE IV

REPORTED CHEMICAL ANALYSES OF OVERHEAT ALLOYS

Alloy and Heat Number	Composition (percent)											
	C	Mn	Si	S	Cr	Ni	Co	Mo	Ti	Al	Fe	Cu
M-252 Heat 837	0.16	0.82	0.60	-----	18.70	54.15	9.70	10.00	2.71	0.96	2.20	----
Inconel 700 Heat Y7952	0.12	0.07	0.24	0.007	15.70	46.25	28.69	3.08	2.02	3.13	0.65	0.02

TABLE V

STRESS-RUPTURE TIME DATA

Material	Temperature (°F)	Stress (psi)	Rupture Time (hours)	Elongation (percent)	Reduction of Area (percent)
M-252	1500	22,000	427	34	46
		32,000	94	37	42
		33,000	82	38	45
			76	38	39
	1600	18,000	92	46	57
		27,000	11	38	45
Inconel 700	1600	20,000	633	18	21
		28,000	104	16	22
		29,000	90	18	20
			89	17	22

TABLE VI

OVERHEATS IN THE ABSENCE OF STRESS

ALL OVERHEATS OF TWO MINUTES DURATION APPLIED EVERY FIVE HOURS

Material	Nominal Test Conditions			Overheat Conditions		Rupture Time		Elongation (percent)	Reduction of Area (percent)
	Temp (°F)	Stress (psi)	Rupture (hours)	Temp (°F)	No. of Cycles	hours	percent of nominal		
M-252(1)	1500	24,000	115	1900	36	182	158	32	38
M-252(2)	1500	33,000	79	1800	17	87	110	19	45
					10	77	98		
				2000	24	114	145	12	16
					10	99	125	36	42
	1600	18,000	92	2000	27	138	150	40	33
Inconel 700	1600	29,000	90	1800	14	70	78	10	15
				2000	12	56	62	4	8

(1) Heat 837 rolled at 1950°F, ST 1950°F, age 1400°F.
(2) Heat 837 rolled at 2150°F, ST 2150° + 1950°F, age 1400°F.

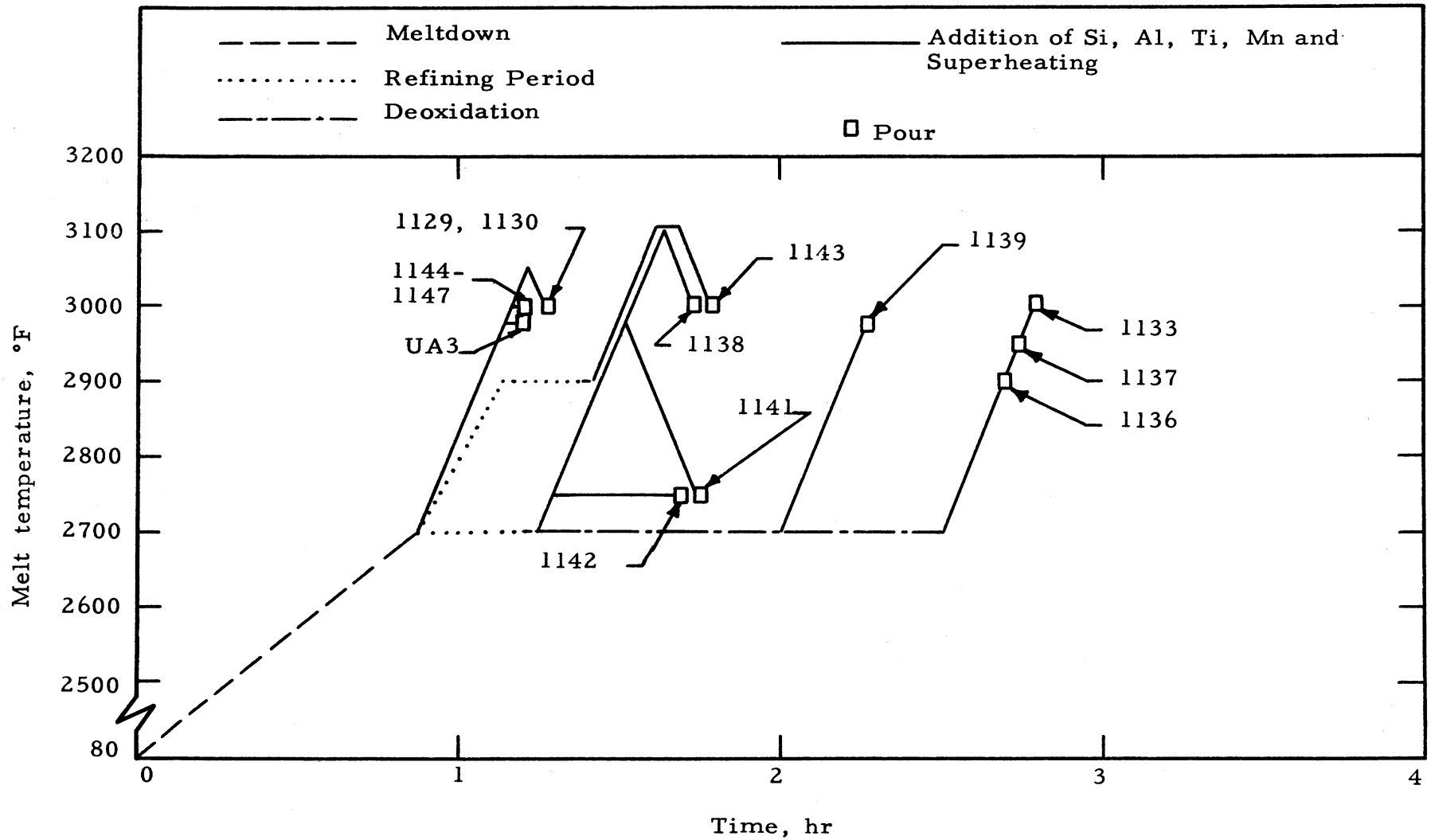


Figure 1. - Melting cycles used in study of effect of melting variables on high temperature properties of Udimet 500.

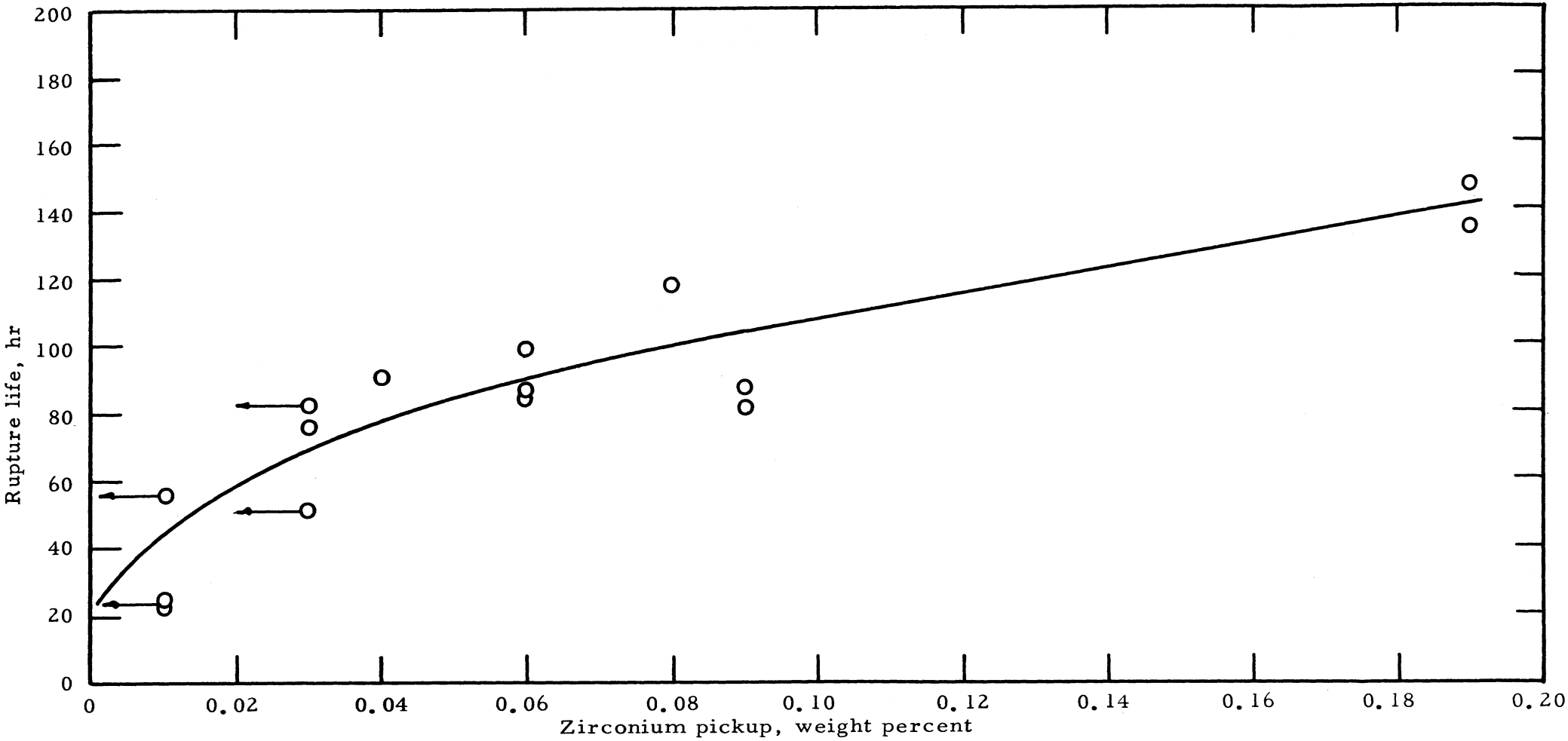


Figure 2. - Effect of Zr pickup from crucible on rupture life of Udimet 500 at 25,000 psi and 1600°F. Prior treatment was 2 hours at 2150°F, air cooled.

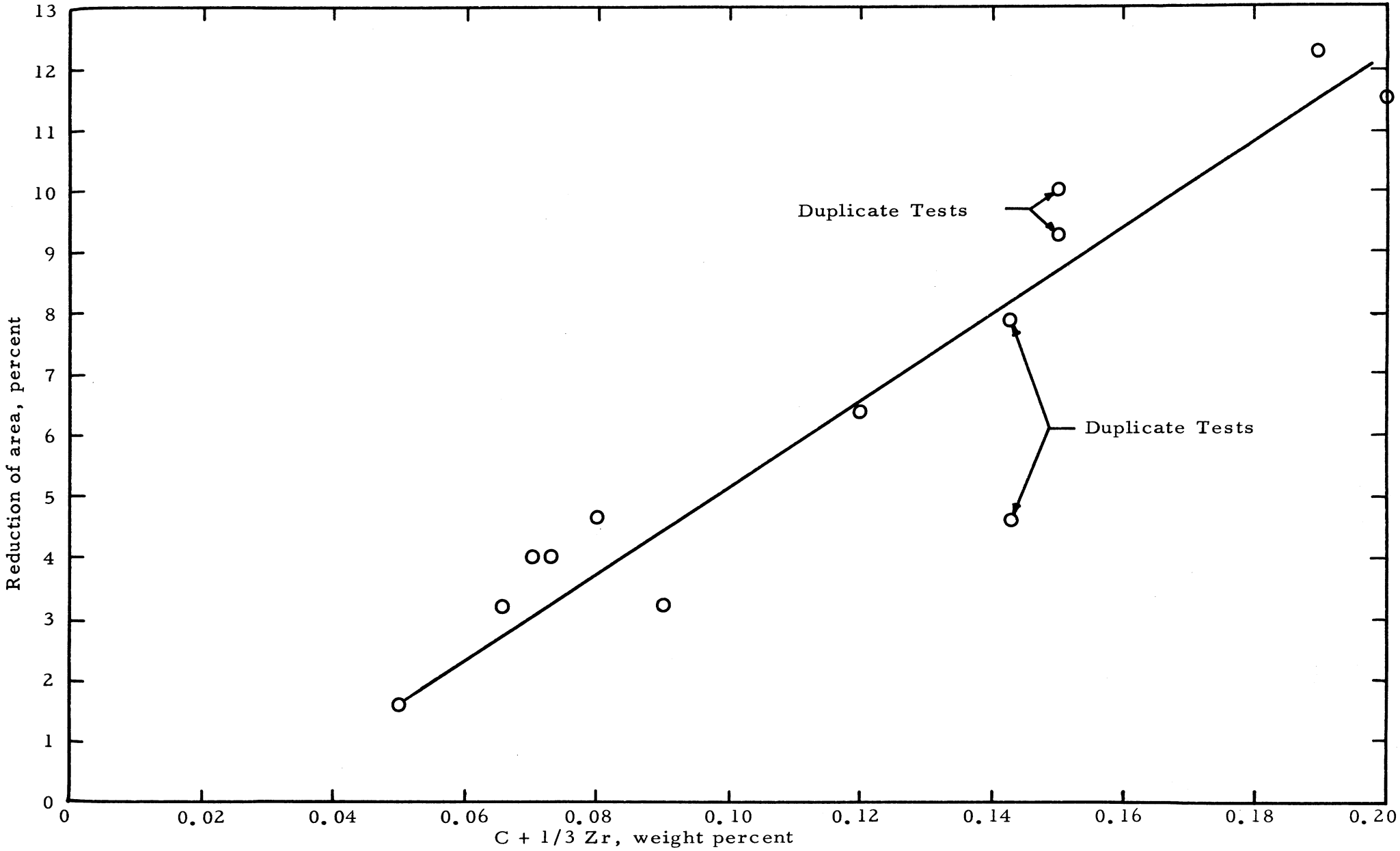


Figure 3. - Influence of C and Zr on reduction of area of vacuum-melted Udimet 500 during rupture testing at 25,000 psi and 1600°F. No Boron added. Treatment prior to testing was 2 hours at 2150°F, air cooled.

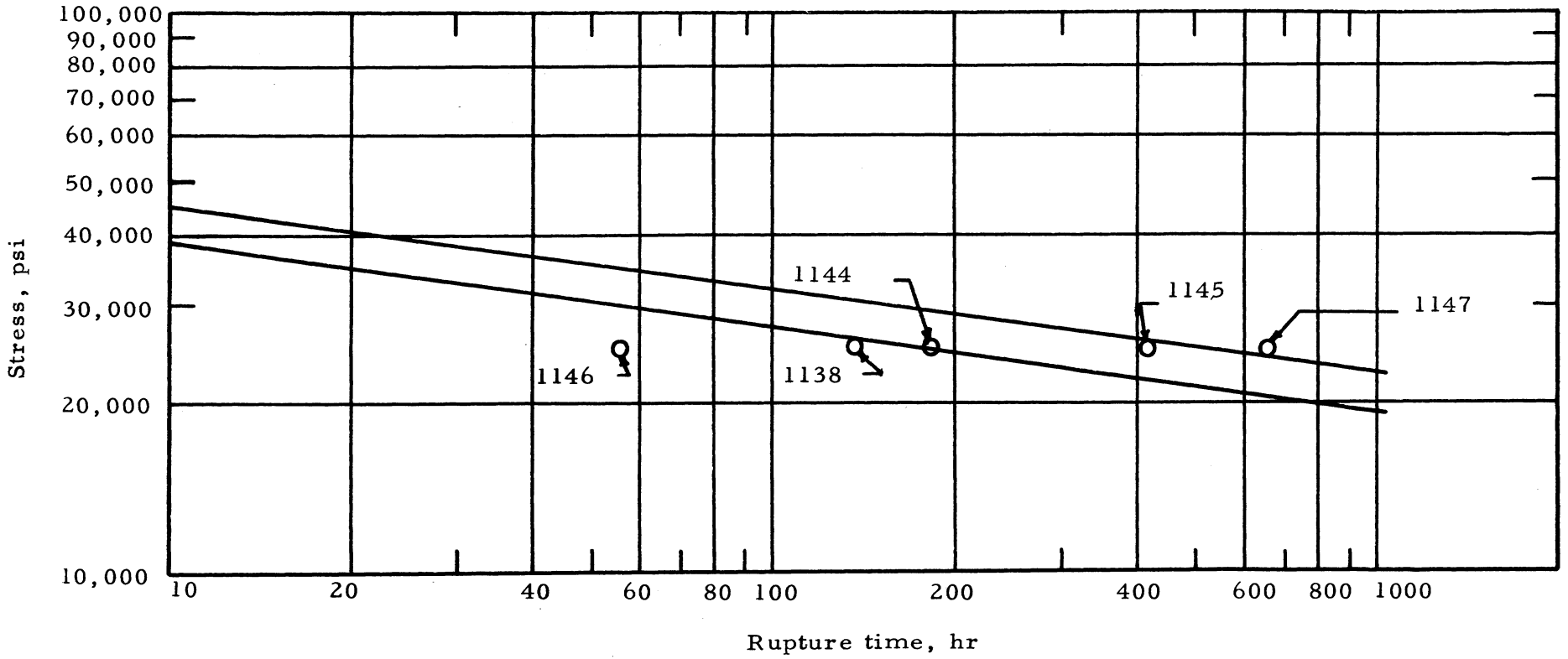


Figure 4. - Rupture life of experimental heats plotted on rupture strength band at 1600°F for two heats of Udimet 500 as reported by Utica Drop Forge and Tool Corporation. Treatment of experimental heats was 2 hours at 2150°F, air cooled. Treatment of Utica heats was 2 hours at 2150°F, air cooled plus 4 hours at 1975°F, air cooled plus 24 hours at 1550°F plus 16 hours at 1400°F. Heat 1146 - base alloy; heat 1138 - with Zr; heat 1144 - magnesia crucible; heat 1145 - with B; heat 1147 - with B and Zr.

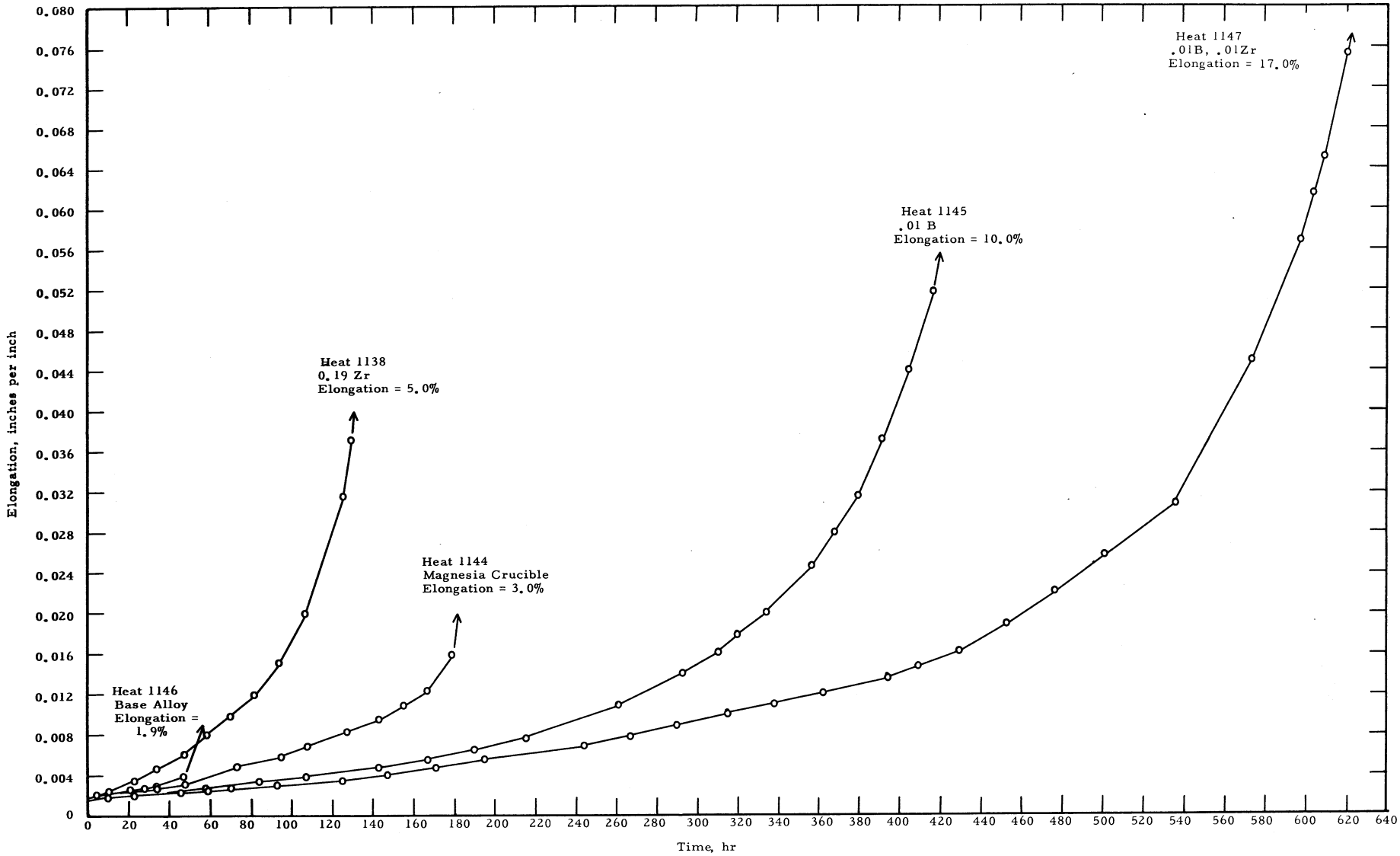


Figure 5. - Comparative creep curves at 25,000 psi and 1600°F for experimental heats of Udimet 500.

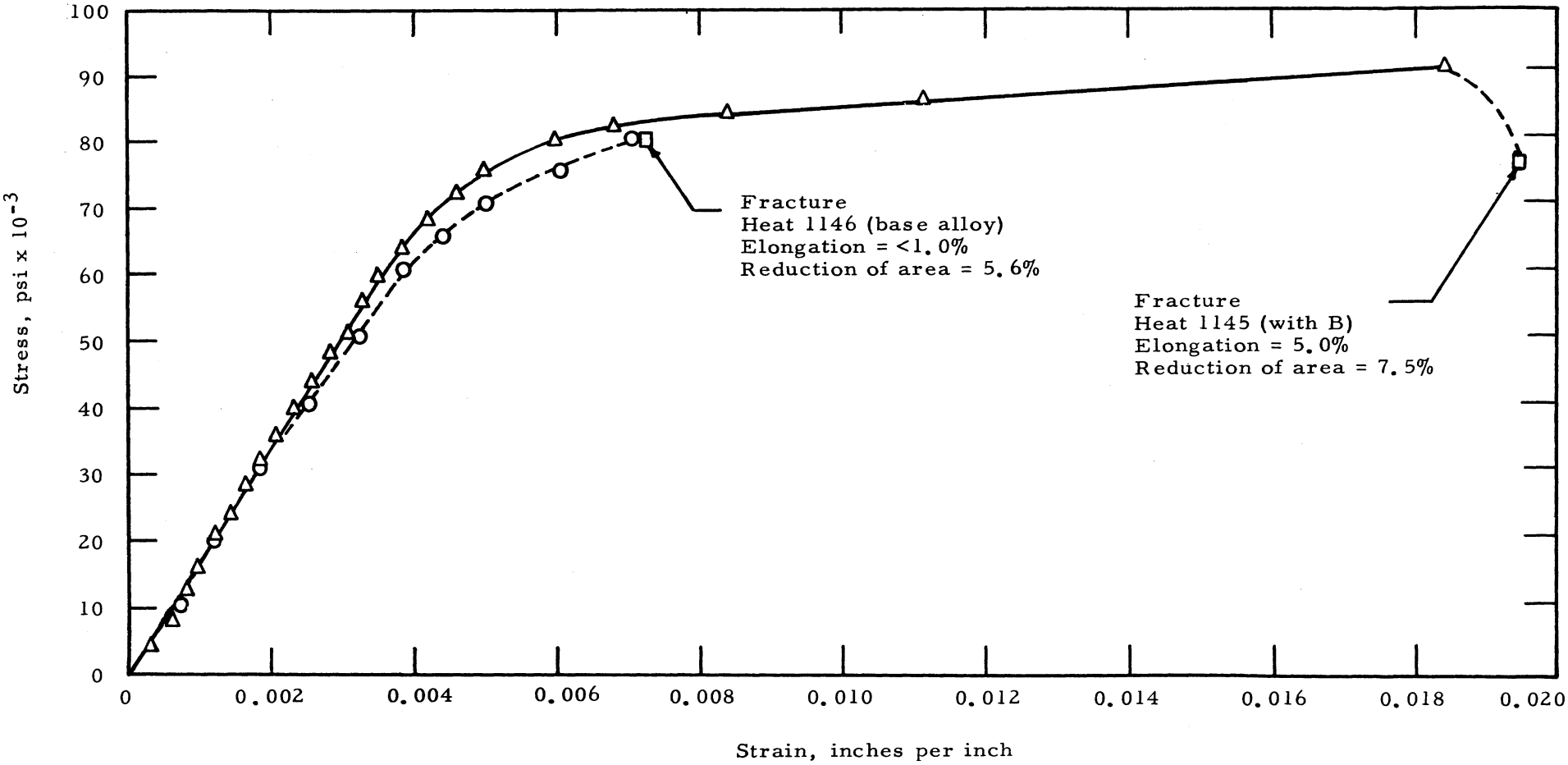
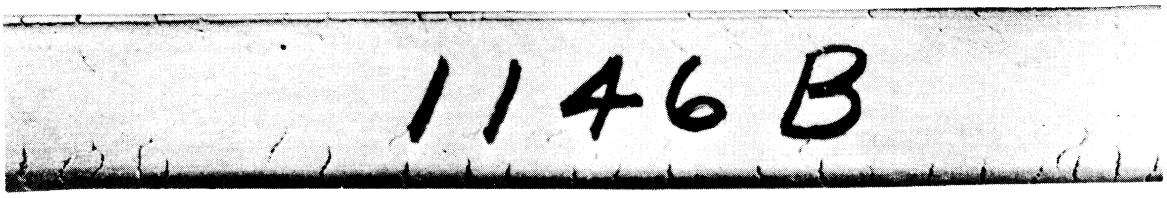
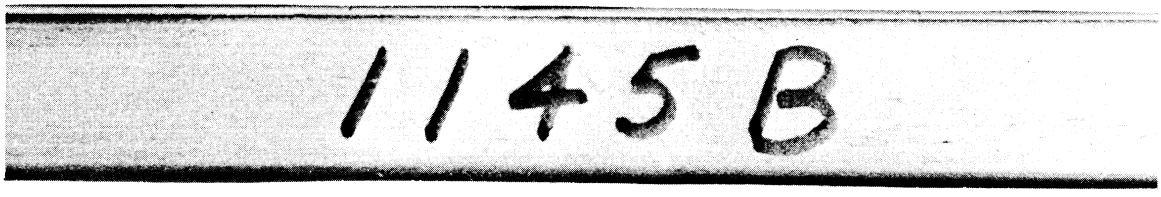


Figure 6. - Stress vs. strain during tensile test at 1600°F for base Udimet 500 (heat 1146) and Udimet 500 plus B (heat 1145).



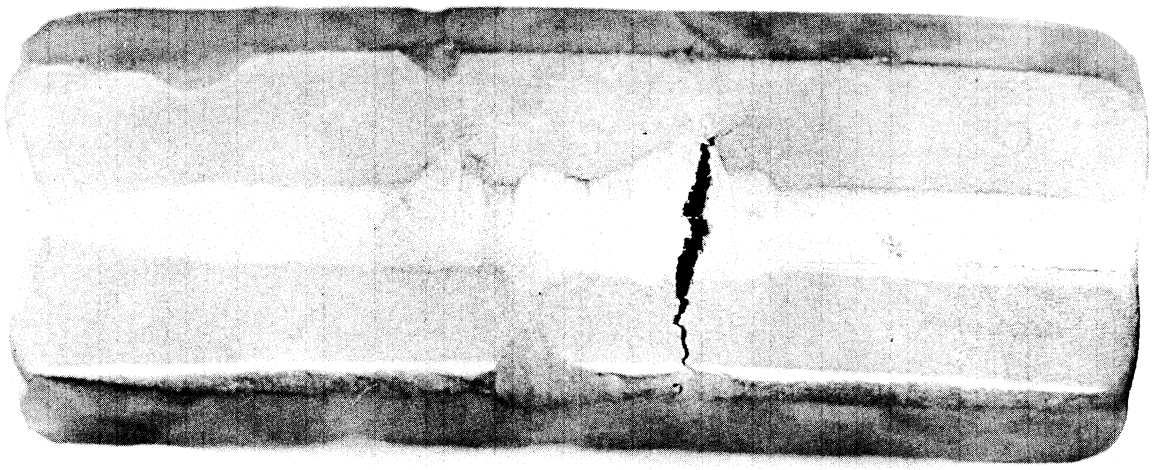
a. Heat 1146, no B addition, <.01 Zr pickup.

b. Heat 1138, no B addition, .19 Zr pickup.

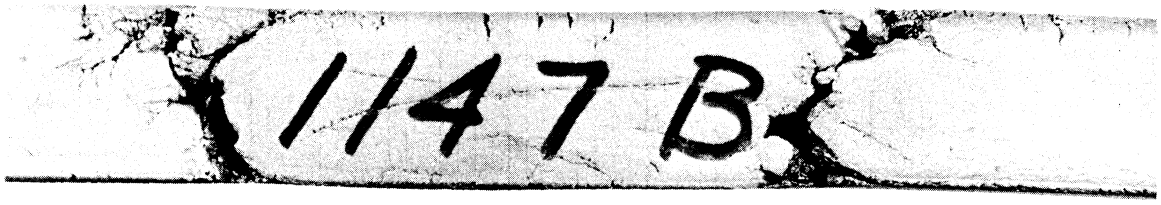


c. Heat 1145, .01 B addition, <.01 Zr pickup.

Figure 7. - Effect of B additions or Zr pickup on surface cracking of Udimet 500.

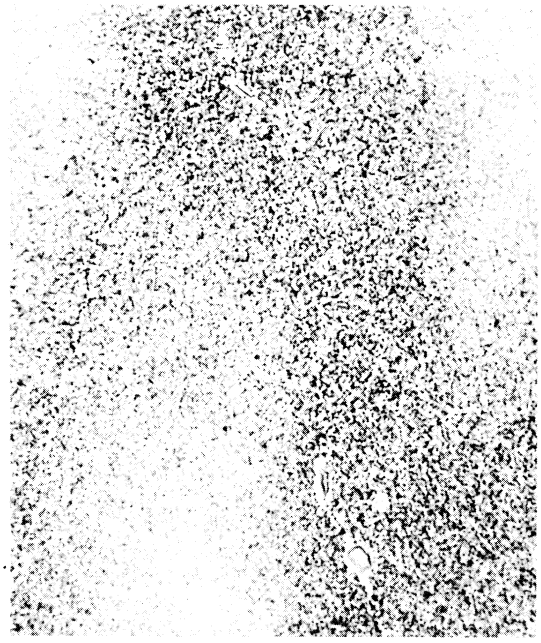


a. Ingot after three passes at 2150°F.



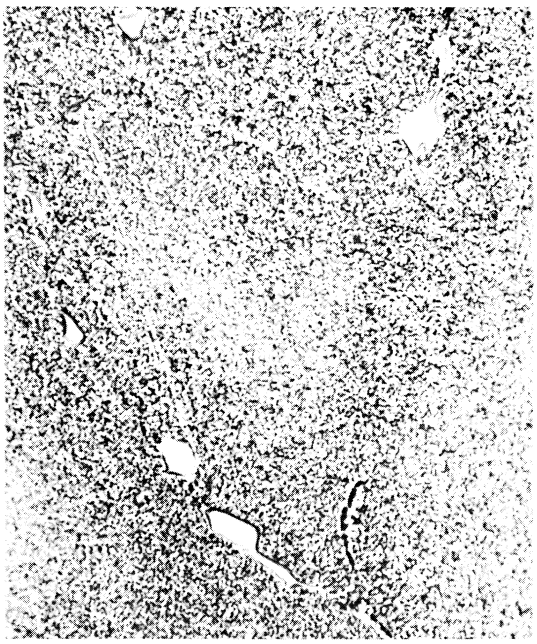
b. Bar stock after reduction to 7/8-inch bar stock. Large cracks occurred during ingot breakdown and could not be ground out.

Figure 8. - Effect of combined addition of .01 B and .01 Zr pickup on cracking of heat 1147 during ingot breakdown.



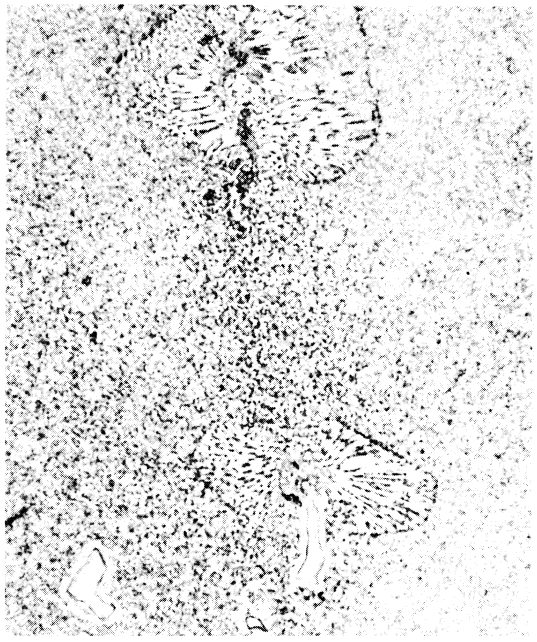
X1000D

a. Heat 1146, no B addition, <.01 Zr pickup, as-cast surface.



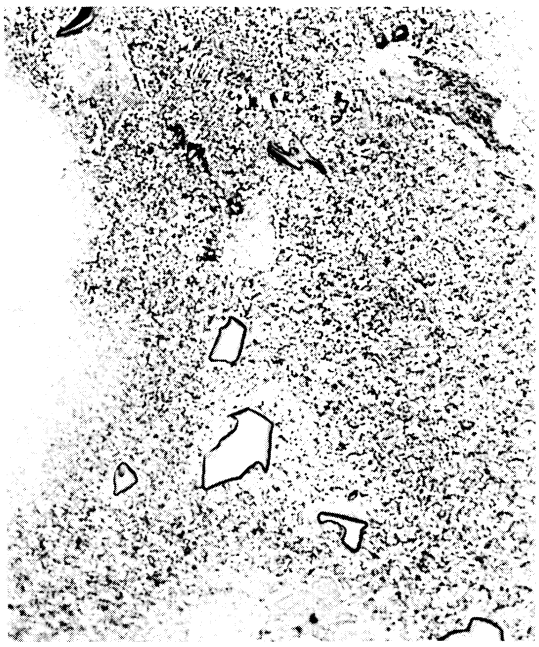
X1000D

b. Heat 1146, no B addition, <.01 Zr pickup, as-cast center.



X1000D

c. Heat 1147, .01 B addition, .01 Zr pickup, as-cast surface.



X1000D

d. Heat 1147, .01 B addition, .01 Zr pickup, as-cast center.

Figure 9. - Effect of combined addition of .01 B and .01 Zr pickup on as-cast microstructures.

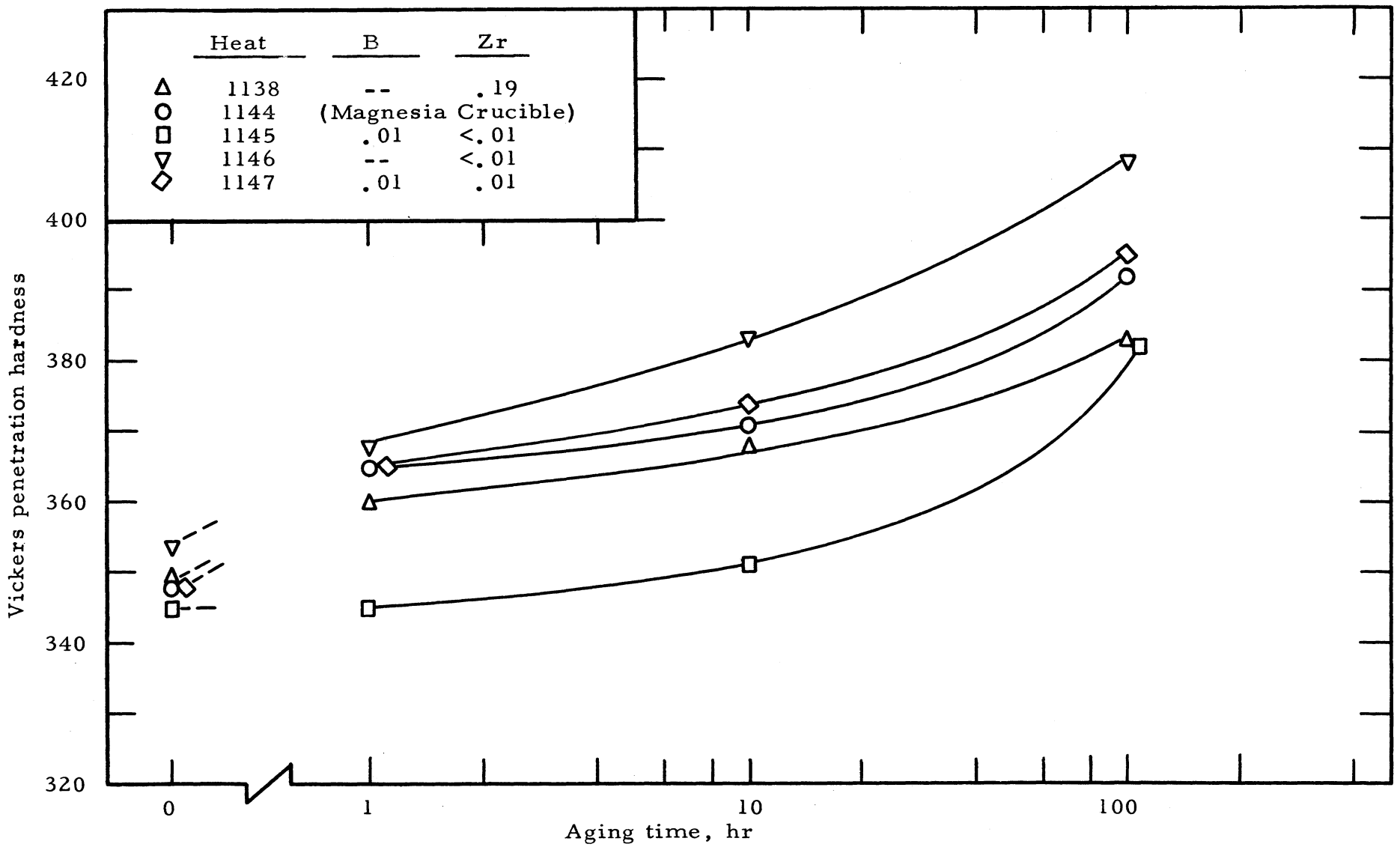


Figure 10. - Effect of aging time at 1200°F on hardness of Udimet 500. Treatment prior to aging was 2 hours at 2150°F, air cooled.

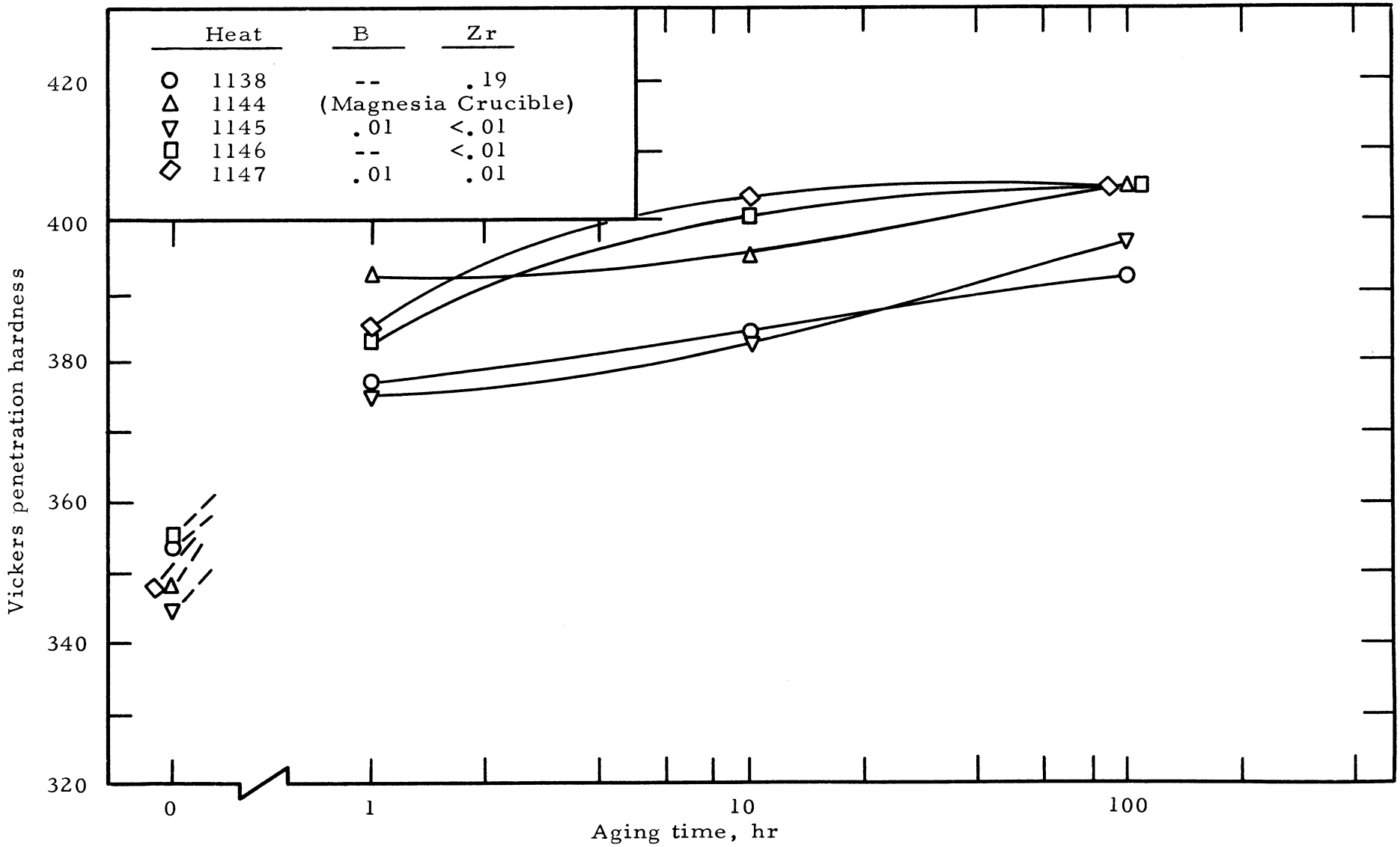


Figure 11.- Effect of aging time at 1400°F on hardness of Udimet 500. Treatment prior to aging was 2 hours at 2150°F, air cooled.

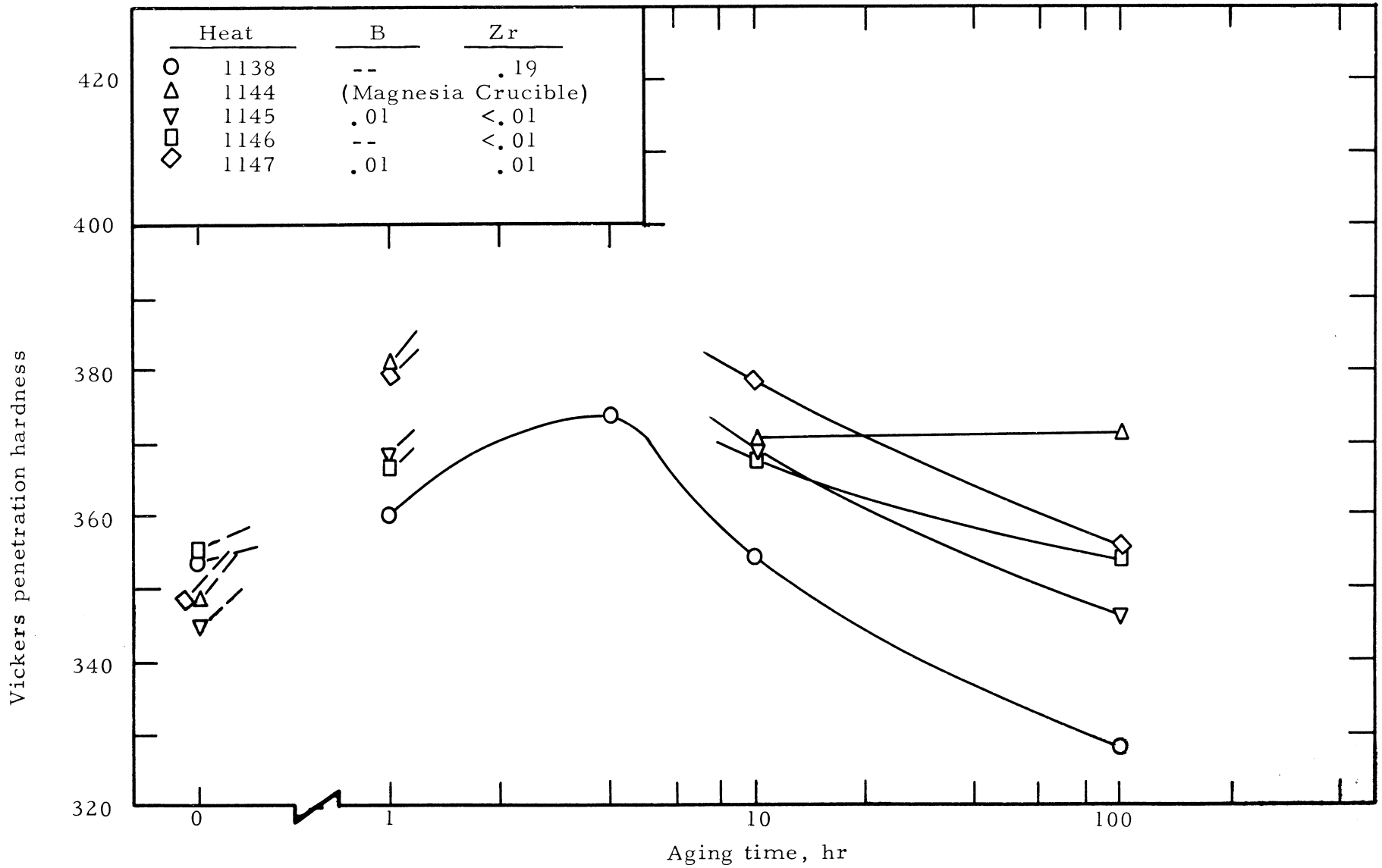
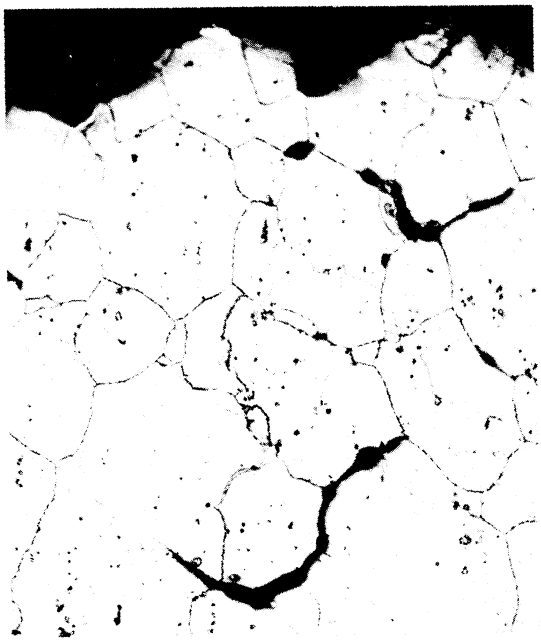


Figure 12. - Effect of aging time at 1600°F on hardness of Udimet 500. Treatment prior to aging was 2 hours at 2150°F, air cooled.

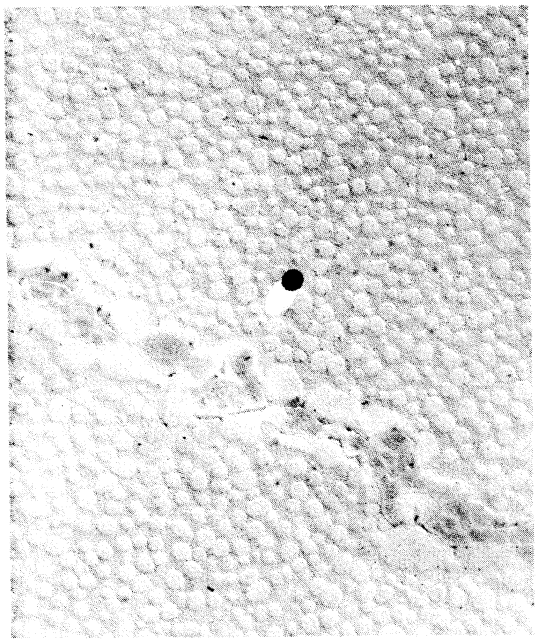


X100D

a. Light micrograph near fracture.

X1000D

b. Light micrograph near fracture.



X13,000D

c. Electron micrograph of lightly agglomerated grain boundary.

X13,000D

d. Electron micrograph of heavily agglomerated grain boundary.

Figure 13. - Microstructure of heat 1138 (high Zr) after rupture testing. Treatment prior to testing was 2 hours at 2150°F, air cooled.

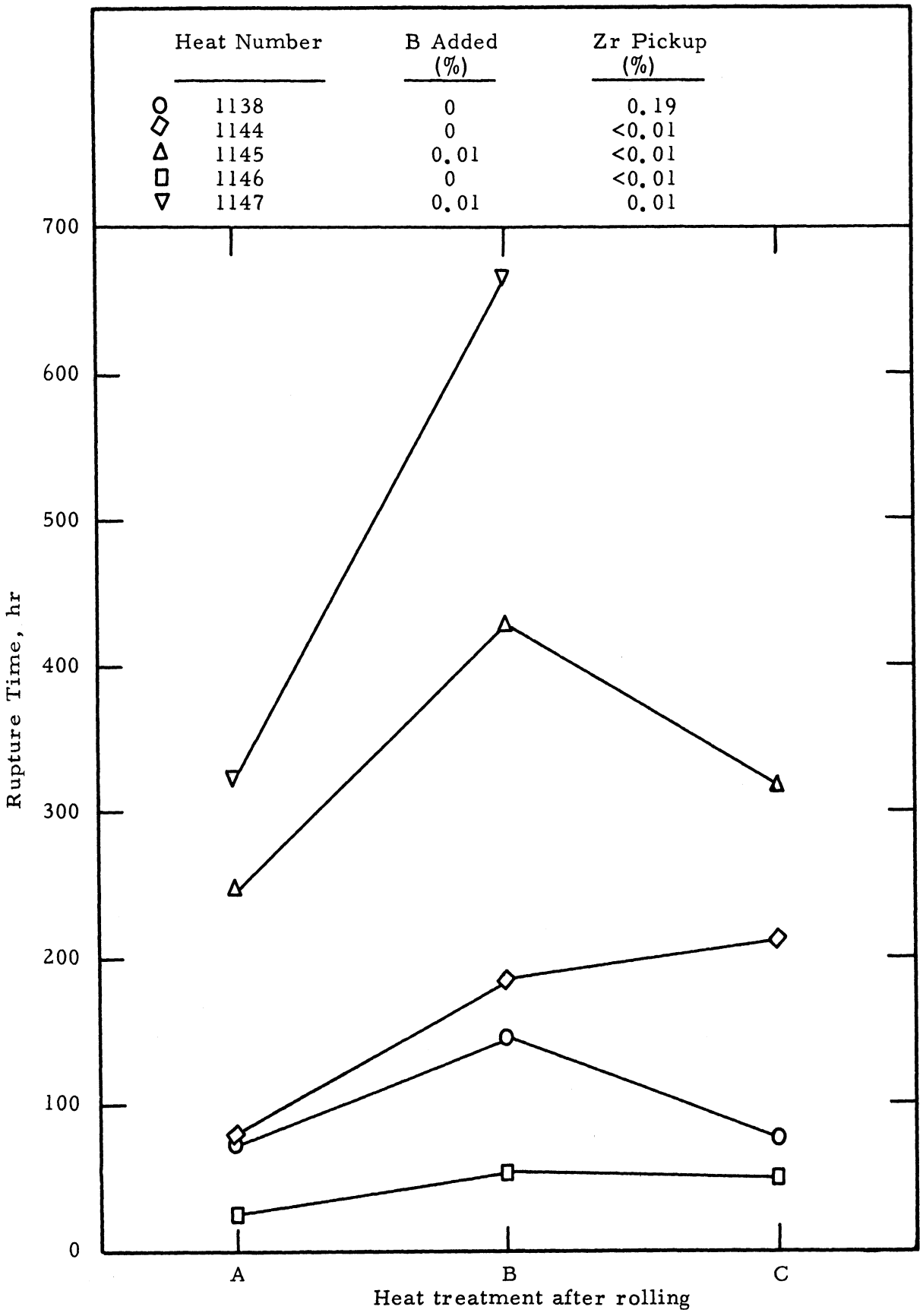
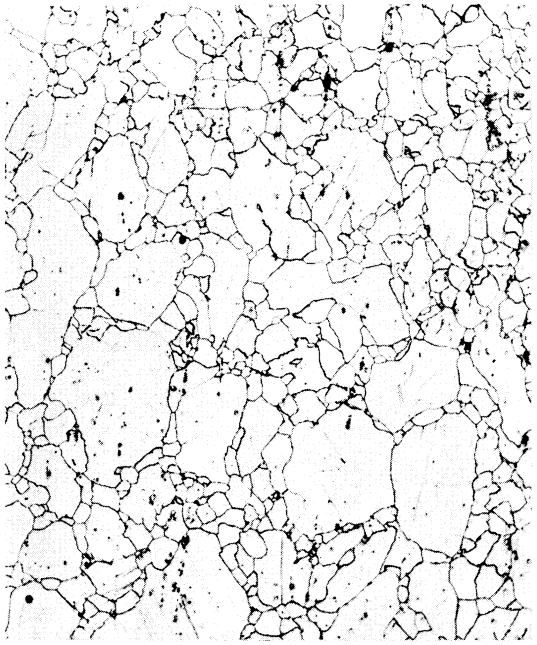


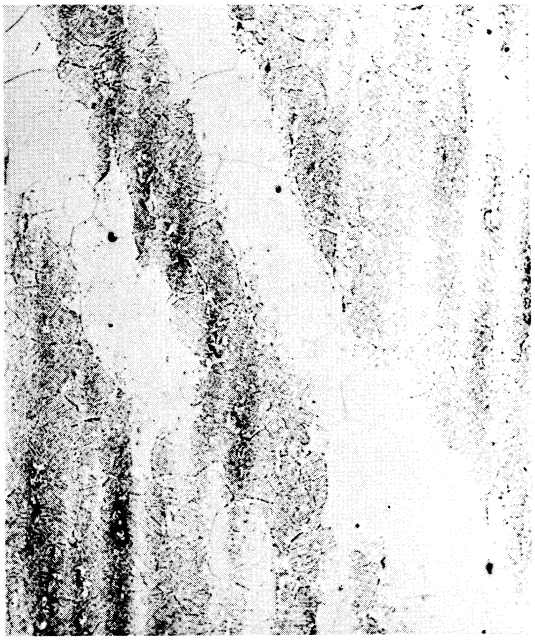
Figure 14.- Effect of heat treatment after rolling on the rupture time at 1600°F and 25,000 psi for several heats. Heat treatments as follows:

A- 4 hours at 1975°F, air cooled
B- 2 hours at 2150°F, air cooled
C- 2 hours at 2150°F, furnace cooled.



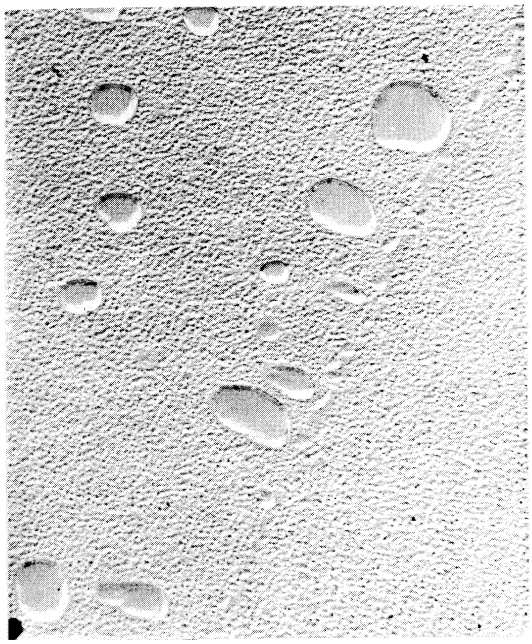
X100D

a. As rolled; light micrograph.



X100D

b. As rolled + 4 hours at 1975°F, air cooled; light micrograph.



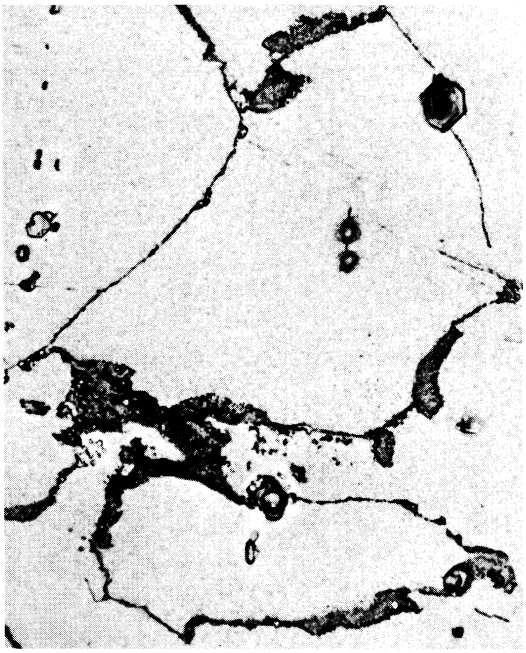
X13,000D

c. As rolled + 4 hours at 1975°F, air cooled; electron micrograph.

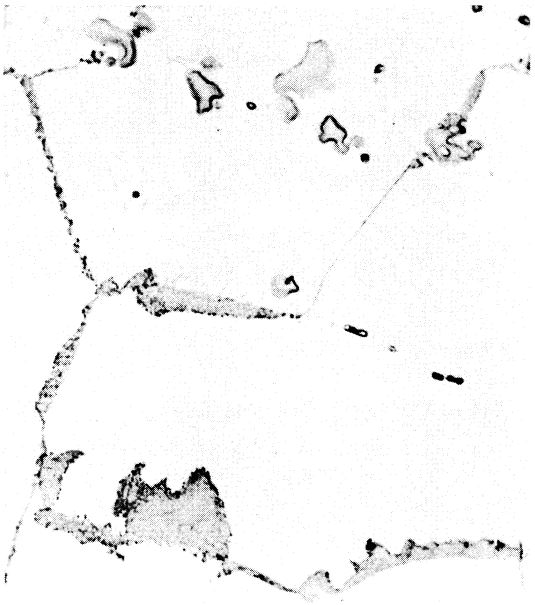
Figure 15. - As-rolled and heat-treated microstructures of heat 1138 (high Zr).

a. Light micrograph of sample which ruptured on loading at 25,000 psi and 1600°F.

Prior treatment was 2 hours at 2150°F, ice-brine quenched + 4 hours at 1975°F, ice-brine quenched + 24 hours at 1550°F, air cooled + 15 hours at 1400°F, air cooled.

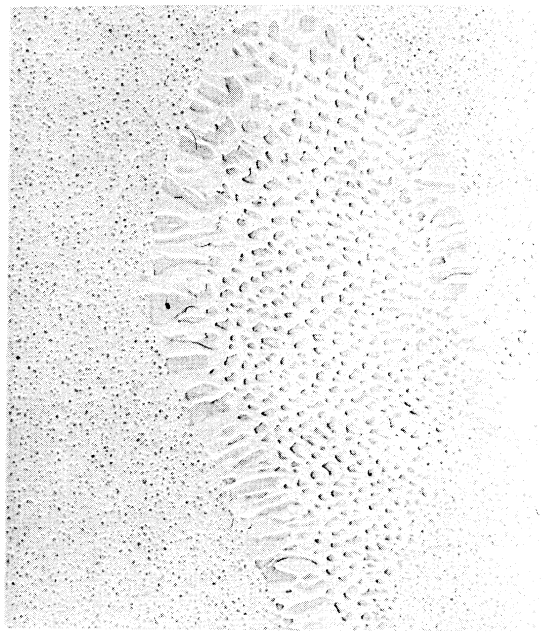


X1000D



X1000D

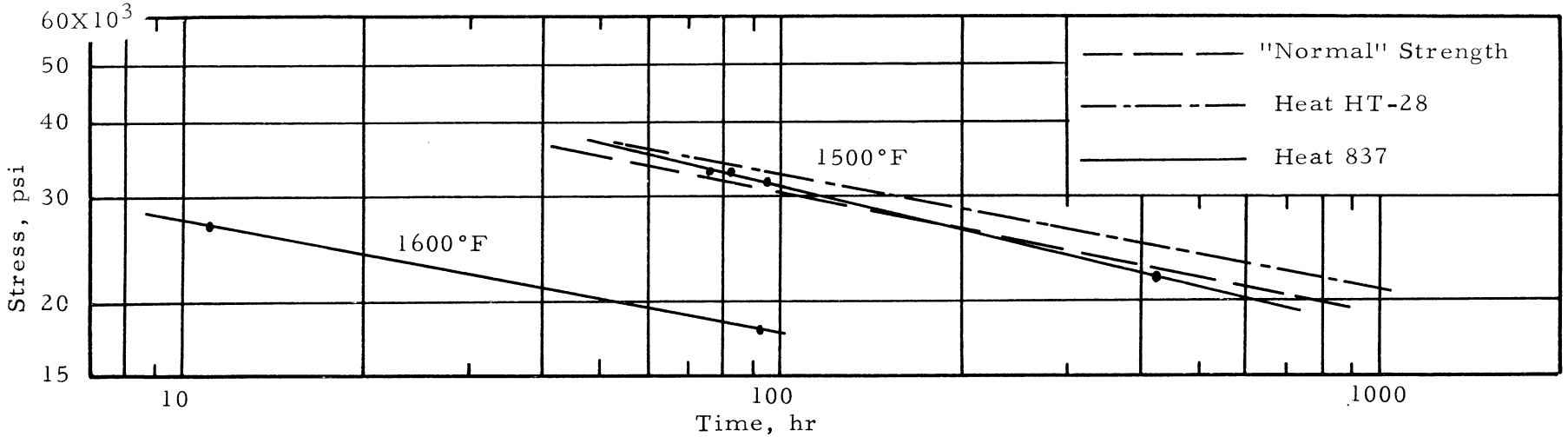
b. Light micrograph of stock after 2 hours at 2150°F, ice-brine quenched + 100 hours at 1400°F.



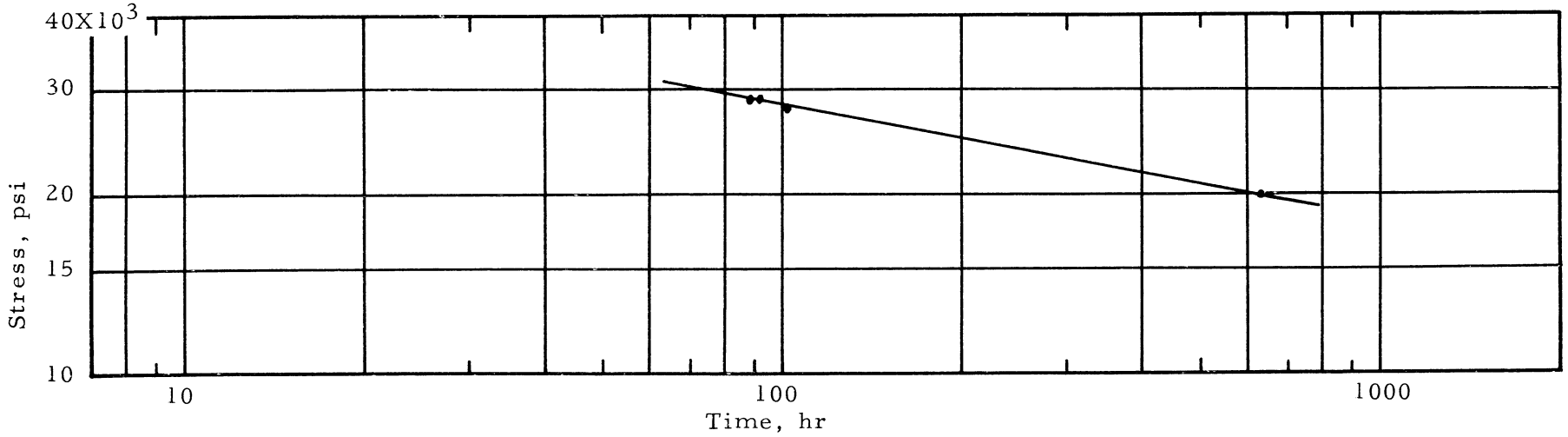
X13,000D

c. Electron micrograph of stock listed in b.

Figure 16. - Cellular precipitate induced by ice-brine quenching of heat 1138 (high Zr).



(a) M-252 alloy at 1500° and 1600°F.



(b) Inconel 700 alloy at 1600°F.

Figure 17. - Stress-rupture time data for M-252 and Inconel 700 alloys.

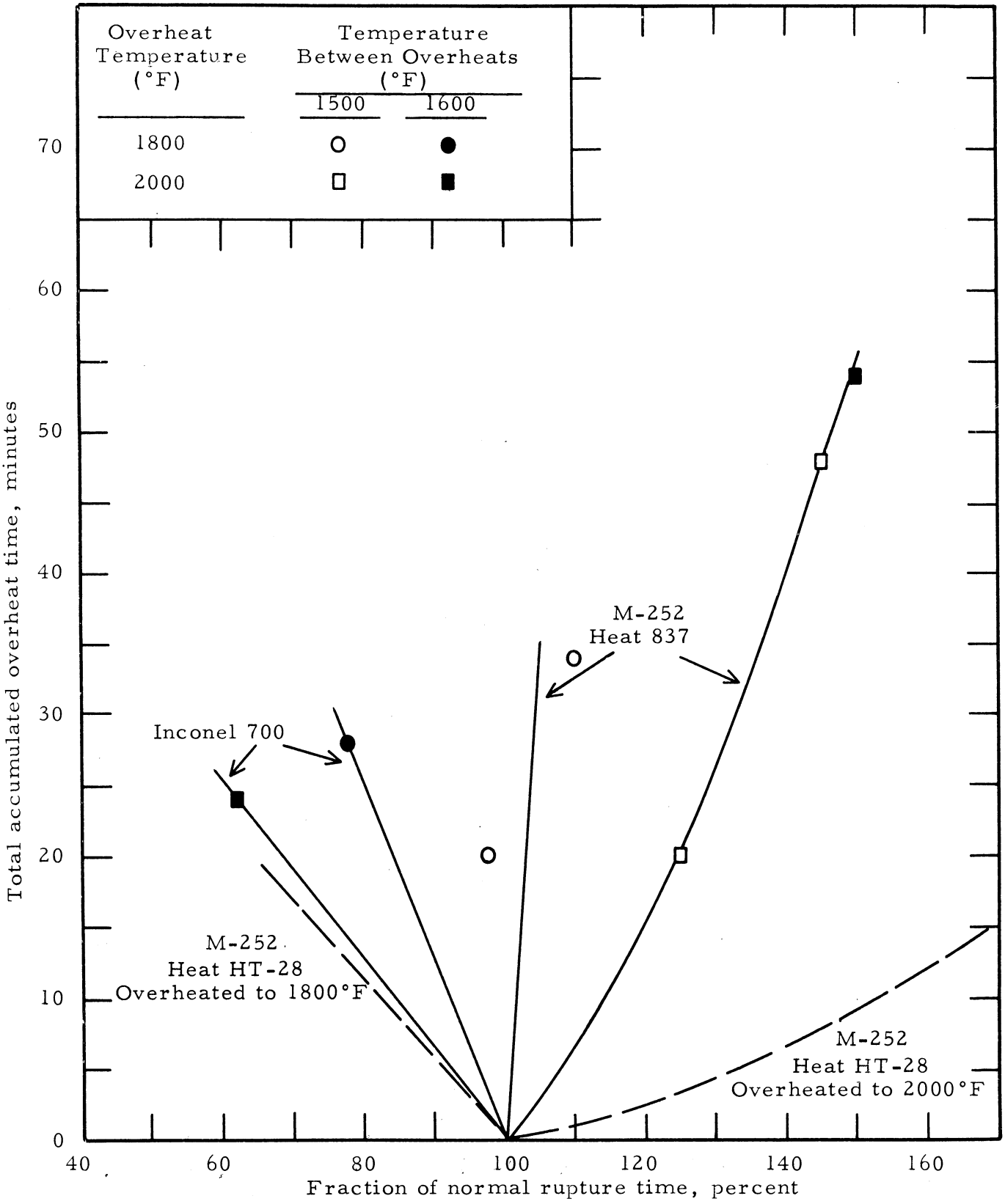


Figure 18. - Effect on M-252 and Inconel 700 alloys of overheating in the absence of stress for two minutes every five hours to 1800° and 2000°F on the rupture life at the indicated temperatures.