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PROGRESS REPORT
TO THE
NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS
COVERING
RESEARCH ON HEAT-RESISTANT ALLOYS

Submitted

by

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INFLUENCE OF MELTING PRACTICE AND OTHER COMPOSITIONAL
VARIATIONS ON THE HIGH-TEMPERATURE PROPERTIES AND
HOT-WORKING CHARACTERISTICS OF A REFRACTORY
Al + Ti HARDENED ALLOY

By Clark L. Corey, Raymond Decker and Karl Kienholz

The research on the physical metallurgy of heat-resistant alloys previously carried out for the NACA had indicated that melting practice probably was a major source of variation in high temperature properties and hot-working characteristics. Accordingly, an investigation was undertaken to study the fundamental factors involved using a refractory Al + Ti hardened alloy.

This report covers progress made on the initial phases of the investigation. Two parallel methods of studying the problem were used:

1. Vary oxygen and nitrogen content of the alloy utilizing vacuum melting and varied techniques of melting
2. Study the basic metallurgy of the alloy so as to be able to establish the mechanism by which the melting conditions operated. In the main, this involved study of solution and precipitation reactions and identification of the phases present.

The alloy selected by the Subcommittee for research is known as Udimet 500. The nominal and aim compositions which were specified were as follows:

	C	Mn	Si	Cr	Co	Mo	Ti	Al	Ni
Nominal	.08/ .15	1.0max	.25max	19/ 21	14.5/ 15.5	3.5/ 4.5	2.75/ 3.25	2.5/ 3.0	bal.
Aim	0.10	0.2	0.2	20	15	3.5	3.0	3.0	bal.

The major characteristic of interest was creep-rupture strength and ductility at 1600°F, together with the hot-working characteristics. Explanations of the results in terms of the metallurgical structural effects were desired in all cases.

The basic test used to evaluate creep-rupture characteristics was a test at 1600°F under 25,000 psi stress. The basic heat treatment was a 4-hour solution treatment at 1975°F followed by air cooling. The standard aging treatment was 24 hours at 1550°F plus 16 hours at 1400°F. The aging treatment was omitted in the majority of tests after it became evident that essentially the same results were obtained for the solution-treated condition.

The initial objective was to melt, roll to bar stock, and heat treat the basic analysis to characteristic creep-rupture properties at 1600°F. With this accomplished, the intent was to systematically vary oxygen and nitrogen, measure the effects on creep-rupture properties and hot-working characteristics, and determine the basic causes of the effects through detailed studies of the alloy structures.

The following interrelated steps were involved:

1. Develop a vacuum melting procedure which would yield satisfactory control of chemical composition in a sound ingot with characteristic properties.
2. Develop the technique of analyzing for oxygen and nitrogen and adjusting the melting procedure to control the amount present. Vacuum fusion analyses were mainly used.
3. Develop procedures for reducing the ingots to sound bar stock with suitable metallurgical structures. The ingots were hot-rolled to bar stock.
4. Concurrently studying the metal structure as influenced by composition and heat treatment on the basis that the compositional effects should be reflected in alterations of the basic metal structure, particularly the aging reactions introduced by the aluminum and titanium. Optical and electron metallographic techniques, X-ray diffraction, resistivity and hardness were utilized.

MELTING AND DEOXIDATION

The melting stock used and analyses reported by the manufacturer are given in the following tabulation:

Electrolytic chromium:-	C - 0.01 Fe - 0.09 Cu - 0.01 S - 0.026 Pb - 0.001 Cr - 99.2 O ₂ - 0.46 N ₂ - 0.026 H ₂ - 0.22 U. M. Vacuum Fusion - 0.48 O ₂ - 0.046 N ₂
Electrolytic nickel:-	Fe - 0.01-0.04 Cu - 0.01-0.03 S-trace Co - 0.10 Ni - 99.95 Si - trace U. M. Vacuum Fusion - 0.0054 O ₂ - 0.00016 N ₂
Electrolytic cobalt:-	C - 0.04 Fe - 0.01 Cu - 0.01 max. S - 0.002 Ni - 0.050 Zn - 0.001 U. M. Vacuum Fusion - 0.0047 O ₂ - 0.0002 N ₂
Aluminum:-	Al - 99.99
Titanium-Ti 55:-	C - 0.002 Fe - 0.20 O ₂ - 0.10 approx. N - 0.012
Molybdenum:-pure Mo:-	C - 0.01
Carbon:-National Carbon, AUC Grade:-	Total ash - 0.10 S - 0.006 P - trace

In most of the heats, the Cr was placed on nickel in the bottom of the crucible together with as much of the Ni and Co as the crucible would hold. When this melted down, the remainder of the Ni and Co were added from the auxiliary charging buckets. When the entire charge was melted, deoxidation was carried out and the Mo, Al and Ti, added in that order.

Two variations from this procedure were also tried:

1. A tube was prepared from Ni sheet and fitted into the crucible to extend above it. The Cr was placed in the bottom of the crucible and the Ni and Co in the tube projecting up from the crucible. As the charge melted, this "stack" dropped down into the crucible. It was found to be difficult to control the bath temperature under this procedure. In addition, difficulty was encountered from violent gas evolution tending to throw part of the melt out of the crucible.

2. Alloys were pre-melted in a vacuum to remove gases. Ni-Cr, Ni-Co, Ni and Co were prepared in this manner. In addition, a high grade 80Ni-20Cr commercial alloy was used as a melting base in one heat. Gas analyses are given at the end of Table I.

If the melting stock contains large quantities of gas, its removal may be the major problem involved in vacuum melting. Gas removal from a molten bath at atmospheric pressure may be attended by a "boil" which, if not properly handled, may become vigorous. When melting in a 1 micron vacuum, gas volumes are expanded by a factor of approximately one million relative to atmospheric pressure. The situation may be even worse since a greater quantity of gas will be removed in the vacuum.

Deoxidation has been carried out using various types of carbon additions, carbon plus hydrogen, solid deoxidizers and pre-vacuumed melted alloys. The removal of the large amounts of oxygen has presented a serious problem due to the difficulty of preventing violent gas evolution. The reaction tended to be so violent that part of the charge would be blown out of the crucible.

When melting at 1 micron, gas volumes are tremendously expanded relative to atmospheric pressure. The Cr contained approximately 0.5% oxygen giving about 0.01 cubic feet of O₂ per pound of charge of the alloy at atmospheric pressure. This becomes 1000 cubic feet at 10 microns and room temperature. Actually, the gas volume in the melt is several times larger due to the temperature expansion from the hot metal.

The several procedures used and the experience obtained were:

1. The melting stock can be treated with hydrogen prior to melting. This was attempted for chromium but was not found convenient and would have required considerably more development to have been successful.

2. Hydrogen can be introduced into the molten charge to remove oxygen. This was used in several heats in combination with insufficient carbon in the charge for deoxidation.

3. Carbon can be added at different points in the melting process to react with oxygen and form CO which can be pumped off. The procedures used were:

- a. Carbon can be added as powdered graphite with the cold charge, effectively removing a good deal of the O₂ before melting is complete. From the standpoint of analysis control, freedom from loss of metal through violent gas evolution throwing the molten metal out of the crucible, control of melt temperature and removal of gases, this was the most successful technique.

b. The addition of carbon to the bath after melt down was tried by two methods. Carbon rods were inserted in the melt so as to reduce the rate of reaction and control rate of gas evolution. This was unsuccessful mainly because it was difficult to tell when the reaction was complete without introducing excessive carbon and splattering of metal. Lumps of graphite were added to the melt to slow down the rate of reaction and thereby obtain controlled gas evolution and carbon content as another method of deoxidizing after melt down with carbon. The rate of gas evolution was very difficult to control.

c. Part of the carbon was added with the charge and part as lump graphite after melt down.

d. Nickel, cobalt, nickel plus cobalt and nickel plus chromium were pre-melted in a vacuum with carbon deoxidation, rolled to bars, cleaned, cut up and used as melting stock in an effort to reduce the amount of gas evolved. A commercial high grade 20Cr-80Ni alloy was also used.

e. Aluminum and other solid deoxidizers were added before adding sufficient carbon for the desired residual carbon.

f. A series of heats were made in which solid deoxidizers were added after carbon deoxidation to determine if they would benefit the properties.

In an effort to vary oxygen and nitrogen, certain special procedures were used:

1. Combined insufficient carbon plus hydrogen deoxidation was used to develop low C, O₂ and N₂.

2. A heat was melted down without deoxidation to develop a high O₂, low N₂ heat.

3. A heat was melted down and deoxidized with carbon plus hydrogen followed by blowing nitrogen over the surface of the melt for 15 minutes to develop low carbon, low oxygen and high nitrogen.

4. Heats were melted in air to develop high O₂ and N₂. Calcium silicide deoxidation was used prior to adding Al and Ti.

In melting the heats, several different groups were prepared as various aspects of their rupture properties became evident:

1. Establishment of carbon control - Heats 1014, 1015 and 1015.

These melted down quite well but various difficulties were encountered mainly in the degree of overheat as various points in the melting schedule and lack of control of pouring temperature.

2. Attempt to reduce gas evolution - Heats 1025, 1031, 1032 and 1033

Carbon rods inserted into the melt were used in the hope of controlling the rate of gas evolution. The main difficulty was that gas continued to evolve at the carbon rods until the result was too high residual carbon contents and gas evolution was still a problem. There was no way to tell when the carbon-oxygen level for 0.10% carbon end point was reached.

3. Heats with variable C, O₂ and N₂.

- Heat 1048 - low carbon by insufficient carbon deoxidation followed by hydrogen deoxidation
- Heat 1049 - low carbon by carbon deoxidation
- Heat 1052 - normal control heat
- Heat 1057 - high O₂ with low C and N by adding no deoxidizer
- Heat 1058 - high N₂ with low C and O₂ by deoxidizing with carbon and hydrogen and then blowing N₂ over the melt for 15 minutes.

The stack melting procedure was used to avoid the bridging problem. Difficulty was encountered in controlling the bath temperature during melt down and gas evolution became violent when the stack slid into the melt.

4. Attempts to control temperatures and gas evolution - Heats 1064, 1066, 1074, 1076, 1079, 1081, 1083, 1085, 1096 and 1097.

Various alterations of charging and melting conditions were tried including extensive use of pre-melted alloys. A high grade commercial 80Ni-20Cr alloy was used as a melting base in one heat. Auxiliary deoxidation with calcium was also tried.

All these heats were poured at 150° to 200°F over the melting point of about 2550°F. However, various complex troubles were encountered from overheating during melt down and excessive rates of gas evolution throwing metal out of the crucible.

5. A series of heats were made with solid deoxidizers after carbon deoxidization - Heats 1109 through 1115.

These heats represent attempts to determine if some additional usual air melting deoxidizers would succeed in improving rupture strength and ductility. The 4 pound heats were used for the generally better control. These heats were poured at 150° to 200°F over the melting point of 2550°F and there was no overheating.

6. Special solid deoxidization before adding carbon-Heats 1021 through 1024.

These heats represent an attempt to see if an oxidizing melt down and deoxidation without fighting gas evolution problem by using solid deoxidation would provide good material. The heats were held 30 minutes before deoxidation to allow pumping off of gases formed in the presence of high oxygen.

7. Varying carbon in the charge - Heats 1117, 1118 and 1119.

These heats were undertaken to see if oxidizing melt down combined with a controlled carbon boil would aid in purifying the heats of other possible deleterious elements and thus improve the rupture strength and ductility. It was thought possible that the carbon in the charge method of melting was avoiding a period of refining while the bath was highly oxidizing. The heats were held for 30 minutes before any deoxidation was attempted. Lumps of graphite were used to add carbon.

The heats were poured considerably hotter than the previous two series on the basis that the low pouring temperature was a contributing factor to low strength.

8. Air melted heats to provide high O₂ and N₂ - Heats UA1, UA2 and UA4.

These heats were made in an open air 12 pound induction furnace using good practice to eliminate inclusions. Deoxidation, in addition to silicon and manganese in the charge, was finished off with calcium silicide prior to adding aluminum and titanium.

9. Effect of varying analysis - Heats 1054, 1092, 1093, 1103, 1094 and 1095.

Heat 1054 was made to check gas contents at time Al + Ti are added. Heat 1092, 1093, 1103, 1094, and 1095 were made for X-ray diffraction microstructural studies.

Metal temperatures and other melt history were not reported in detail due to the complexity of the events which occurred. Many of the heats which were overheated during melting were cooled by additional cold charge or the charge was held and allowed to cool before pouring.

CHEMICAL ANALYSIS

The results of chemical analysis of the heats made are given in Table I.

Carbon, sulfur and phosphorous were obtained by chemical methods. Some checks on Al and Ti were also obtained by chemical methods. Nitrogen was measured mainly by vacuum fusion with a considerable number of checks by the chemical Kjeldahl method from two laboratories. Oxygen was obtained by vacuum fusion. Spectrographic analysis, through the courtesy of the Utica Drop Forge Company, were used for Al, Ti, Cr, Co, Si, and Mn.

The first heat in Table I is for ingots supplied by the Utica Drop Forge Company.

There are a number of features of the results of chemical analysis which raise the usual questions for a complex alloy of this type:

1. Al, Ti and Mo were generally quite close although consistently higher than aim amounts. Because they were added just before pouring to well deoxidized heats in most cases, they should never have fallen below 3.5% Mo, 3.0% Al and 3.0% Ti. One is, therefore, induced to question values below this level.

High values could arise from metal loss before their addition to the heats. In a number of cases splashing of metal due to violent gas evolution caused this to happen. For instance, Heat 1079 shows 4.20Al - 6.0Ti. Since equal amounts were charged, they ought to have been equal - the final weight of the ingot indicated 4.1%.

One laboratory failed to check Al content of U-500 closer than 0.5%. Two separate laboratories gave 0.004 and 0.016% sulfur for this heat.

Low Al and Ti in the air melted heats was probably due to their loss to the slag. This is also probably the case for Heat 1121 when the Ti and Al was added before the carbon.

Kjeldahl nitrogen values commonly varied by a factor of 2 and were sometimes larger than vacuum fusion methods by a factor of 5 to 10. One laboratory gave consistently lower values than another.

Considerable questioning of the vacuum fusion analyses for oxygen and nitrogen have occurred. In general, they are considered to reflect the gas content of the alloys quite well.

Oxygen contents were generally 0.001 percent or less. Nitrogens were usually below 0.003 percent. These both represent rather low values. When gas contents were higher, no difficulty in obtaining checks were encountered. Also Kjeldahl nitrogens checked the vacuum fusion apparatus. Good checks by vacuum fusion were also obtained at low values.

In starting to analyze by vacuum fusion, a few checks were made with satisfactory results as shown by the following tabulation:

Material	Michigan Vacuum Fusion		Source of Check	Check Analyses	
	Oxygen (%)	Nitrogen (%)		Oxygen (%)	Nitrogen (%)
Electrolytic chromium	0.486	0.046	Supplier	0.46	0.026
N-155 alloy	.003	.128	Kjeldahl	--	0.13
18Cr-8Ni	.021 .023	.032 .028	Mass. Inst. Technology	.029	.029
18Cr-8Ni	.025 .025	.022 .021	Mass. Inst. Technology	.004	.023
Carbon steel	.0113	.0035	Kjeldahl	--	.004
Carbon steel	.1270	.0026	National Bureau of Standards	.106	.004
Carbon steel	.0107 .0107	.0170 .0146	National Bureau of Standards	.017	.015

The comparative vacuum fusion and Kjeldahl analyses for N₂ in Table I show values considerably lower for vacuum fusion. However, one laboratory in which more confidence is placed consistently obtained lower values, which in most cases were still higher than vacuum fusion results.

In running consecutive vacuum fusion analysis on the same heat in the same crucible in the vacuum fusion apparatus, some drift in results was noticed for the standard heats. This was reflected as decreasing N₂ on successive runs as is shown by Figure 3. The oxygens were constant at 0.00108 ± 0.00010 for Heat 1016. Heat 1058 also showed drift in nitrogen. The oxygen values were also less

consistent.

To eliminate the possibility of interference from Al + Ti, Heat 1054 was made up as a standard heat and poured without adding Al and Ti. In this case, the O₂ increased while the N₂ decreased, Figure 4, over successive runs on the same samples from a rolled bar. Similar results were obtained with bath temperatures of 1800° and 1550°C.

All analyses have been obtained by dropping small cubes into a graphite crucible. Standard procedures to avoid surface acclusion of oxygen and nitrogen. Crucibles have been baked out at 2400°C. Blanks between runs have been small and consistent.

Both nickel and tin have been used a small amount to see whether they charge the results and alleviate possible "gettering". The following results were obtained on Heat 1057:

Run	Dilution Agent	O ₂ (%)	N ₂ (%)
1st	None	.0058	.0024
2nd	None	.0020	.0018
3rd	None	.0020	.0010
1st	Nickel	.0011	.0015
1st	2.5 grams tin between each 2.2 gm sample	.0050	.0015
2nd	2.5 grams tin between each 2.2 gm sample	.0025	.0003
3rd	2.5 grams tin between each 2.2 gm sample	.0048	.0002

It is possible that further work with dilution agents would solve the problem.

The major difficulty seems to occur principally in the range of 0.001 to 0.005 percent of oxygen or nitrogen. Below this range the reproducibility seems to be better, as shown by the following tabulation:

Heat	Run	O ₂ (%)	N ₂ (%)
1099	1st	0.00072	0.00013
	2nd	.00065	.00008
1096	1st	0.0019	0.00045
	2nd	.0015	.00023

In reporting results in Table I, the number of the samples in a series of successive determinations in the same crucible has been reported. Where more than one was available the first run was reported since this gave the highest oxygen.

HOT-WORKING CHARACTERISTICS

After considerable experimentation with the ingots supplied by Utica Drop Forge Company, it was determined that the hot-working temperature should be 2150°F. The rolling mill at the University gave better results than the small low capacity hammer. Only one pass could be used between reheats and it was necessary to use at least 10 minutes for reheats. The reduction per pass was limited to about 10% reduction of area. Larger reductions, lower or higher temperatures of heating, shorter reheat times and more passes per heat all led to increased cracking during rolling.

As far as could be determined, the alloy work hardened rapidly during working. The reheats were necessary to either relieve the cold work or soften the material. There may be a possible relation to precipitation hardening involved also. The rather high hardness of the alloy, even when ice-brine quenched from the mill, are shown by Figure 5. This also shows the rapid increase in hardness when working temperatures are lowered below 2150°F. It also shows the rate of softening on reheating to 2150°F. Time periods as long as 6 minutes at 2150°F are required to obtain minimum hardness.

The ingots were normally rolled to 3/4-inch squares. In some cases, they were carried down to 1/2-inch squares.

Two main types of difficulty in rolling were encountered:

1. Corner cracking:- These ranged from immediate break-up of the ingots through all degrees to complete freedom from cracks. If these occurred, they would continue to grow during succeeding passes through the mill unless ground out. This was the usual type of difficulty in working the standard analysis.

2. Some heats developed surface cracks of the type shown for Heat 1095 in Figure 6. These appear very damaging but are very shallow and do not grow with continued reduction. Furthermore, if they are ground out, they immediately return on the next pass. When this type of cracking was encountered, no attempt to remove them was made. This type of cracking was most prevalent in the high Al and Ti heats (Heat 1079-4Al+4Ti and Heat 1095-6Al+6Ti).

Hot-workability appeared to be a complex interrelationship of a number of factors. The problem of evaluating this characteristic was further complicated by the alloy being difficult to work at best. Careful attention to details of the procedure were necessary to produce crack-free bars. Within this limitation, certain general trends developed:

1. Workability definitely increased with carbon content up to the upper range studied, 0.20 to 0.30%. When the carbon content approached 0.10% the workability became reasonably good in most cases. Heat 1015 with 0.13% carbon had about as good working characteristics as any heat. Other heats with similar carbon contents were, however, difficult to work down. Those heats with higher carbon content were, however, all relatively easy to work.

Some heats with very low carbon, such as, 1064 and 1074, were, however, worked down to a crack free condition by use of considerable care and grinding away cracks as they formed.

The 0.10% carbon level appears to be close to the amount where insoluble carbides appear in the structure. One factor involved appears to be the grain growth inhibiting effect of these phases because all the high carbon heats which rolled well remained much finer grained than the lower carbon heats. The possibility also remains of there being an effect of carbon on the oxygen level which is not apparent in the analyses.

2. Aluminum and titanium content also influenced workability. Alloys with 1.25%Al and 2.25%Ti have been given reductions of 75% from 2150°F without reheating. The 3Al-3Ti heats were far more difficult to work. This is shown by Figure 7. However, Heat 1079 with 4.1% of Al and Ti was successfully worked down although it was difficult. Heat 1095 with 6%Al and 6%Ti was rolled down to a 5/8-inch square when it split longitudinally. This heat probably had 0.25% carbon. Two other heats with about 0.08% carbon broke up on the first pass, again showing the beneficial effect of carbon. The longitudinal split suggested excessive cold work from the lack of relief of cold work in the 10 minute reheats.

3. Oxygen and nitrogen content appeared to influence workability even though it was not reflected in the gas analyses. Heat 1058 which was made by blowing nitrogen on the surface worked differently than any other heat. It required far more power than any other heat. It would nearly stop the mill, whereas, all other heats rolled without slowing the mill. It was also susceptible to corner cracking; but by frequent regrinding, it was possible to roll it down to barstock.

Heat 1057, which was made without deoxidation, also rolled with difficulty although it was not as stiff as Heat 1058.

The gas contents of Heats 1057 and 1058 together with those for the air melted heats were:

Heat	O ₂ (%)	N ₂ (%)	C (%)	Kjeldahl N ₂ (%)	Comments
1057	0.0058	0.0024	0.03	0.003	No deoxidation
1058	.00004	.0022	.03	.006	N ₂ blown on melt surface
UA4	.0011	.0066	.13	.0084	Air melted
1014	.0002	.0012	.06	--	Vacuum melted-poor workability
1015	.00008	.0030	.13	--	Vacuum melted-good workability

The poor workability of 1057 and 1058 seems related to the melting variables even though their carbon content was low. Heat 1057 was definitely worse than other heats which had equally low carbon but had been deoxidized.

4. The air melted heats all rolled relatively easily. Figure 7 shows that air melted Heat UA4 rolled better than vacuum melted Heat 1014 with less carbon. Their nitrogen contents were all relatively high. Oxygen, however, was not particularly high. These heats were exceptionally free from visible inclusions in the microstructure. Aluminum and titanium, however, were somewhat low, presumably due to losses to the slag. Their relatively good workability seems related to the carbon content and possibly inclusions restricting grain growth and to the lower Al and Ti.

The relation of the analyzed O_2 and N_2 to the hot-workability serves to indicate, however, the difficulty of relating these factors to workability. There were numerous cases where the workability was worse at equivalent or lower analyzed gas contents for a given carbon level.

4. No particular benefit to workability was noted for the use of solid deoxidizers or from the use of pre-vacuum melted alloys. There seemed to be some improvement when the heats were melted down without carbon or only part of the carbon in the charge so that there was a carbon boil.

5. Cobalt appears to be very beneficial to rolling characteristics as is shown by the three heats of Figure 8. The heat without Al and Ti and the heat with cobalt as well as Al and Ti rolled very well. The heat with Al and Ti but no cobalt cracked badly in rolling in spite of a high carbon content.

The reasons for the variation in working characteristics have not been well established. Corner cracking is reduced by lowered Al and Ti, increased carbon and possibly grain growth restriction by certain size of oxides and nitrides. It is promoted by O_2 and N_2 , or at least, certain melting practices which should give high O_2 and N_2 . This suggests that these elements in solution or as oxides and nitrides below a certain size or in a particular distribution is the cause. It is uncertain why the heat made by blowing nitrogen over the melt was so stiff. All that analyses showed was very low O_2 with N_2 only slightly on the high side.

The surface cracking increased with Al and Ti past 3% of each. The compound Ni(Al, Ti) has been definitely identified in the 6Al + 6Ti Heat 1095, particularly near the as-cast surface. The NiAl phase diagram shows that with more than 12%Al, NiAl is the first phase to form. It is presumed that the diagram for the complex alloy causes it to form with 6Al + 6Ti. The similarity of surface cracking in the 4Al-4Ti Heat 1079 suggests its presence in this alloy. Some 3Al-3Ti heats also cracked in the same way suggesting the possibility that some segregation causes it to form in these alloys under certain conditions.

HEAT TREATMENT

The heat treatment established for the alloy was:

Heat 4 hours at 1975°F, air cool
Heat 24 hours at 1550°F, air cool
Heat 16 hours at 1400°F, air cool

Experience demonstrated that the treatments at 1550° and 1400°F did little to change results in the rupture tests and the aging was omitted in most of the rupture tests.

A few tests have been made with solution temperatures of 1800° and 2150°F.

Quench cracking was encountered from water quenching some heats. High gas content and low carbon increased the susceptibility to cracking.

The high hardness of the alloy after solution treatment is shown by Figure 9 and the change in hardness with solution temperature. The increase in hardness with decrease in cooling rate from solution treatment is shown by Figure 10. Aging characteristics, as measured by hardness, are shown in Figure 11 for material solution treated at 1800°, 1975° and 2150°F.

RUPTURE-TEST CHARACTERISTICS

All of the rupture-test data obtained in rupture tests are given in Table II. These data are extracted and discussed in the following sections in accordance with the groupings of heats made with certain objectives in mind.

All data in these groups are for tests at 1600°F under a stress of 25,000 psi for material solution treated at 1975°F and tested without aging unless otherwise specified.

Ingots Furnished by Utica Drop Forge Company

Heat	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
U500	313	14	.08	.00026	.0026
	258	18.1			
(Aged)	329	15.1			

At the time this material was furnished, it was indicated that a rupture time of about 110 hours was typical. A very recent typical band for rupture strengths for two heats, Figure 12, was furnished by Utica. It will be noted that the above data fall in this band.

At the time the tests given above were obtained, they were higher than usual. It was thought that they might reflect the complicated thermal and mechanical history involved in learning to hot-work the alloy. The ingot was heated at several temperatures as high as 2300°F, forged, rolled, ground and otherwise manipulated until only a small piece was finally produced as rolled bar.

It should be noted that the stock used for rupture strength band of Figure 12 included a "mill anneal" at 2150°F prior to the standard 1975°F solution treatment and double age.

The microstructure of the U-500 material is shown in Figure 13.

Variable Carbon Content

Three heats were made early in the program to establish carbon control and effect of carbon on properties:

Heat	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1014	33	6.5	.06	.0002	.0012
(Aged)	32	3.0			
1015	160	4.0	.13	.00008	.003
(Aged)	60	6.9			
1016	104	4.9	.22	.0007	.0011
(Aged)	81	6.0			

These results show probable maximum strength at about the 0.08 to 0.15% carbon content. The test on Heat 1015 at 160 hours is at the bottom of the range of Figure 12. However, it was not given the 2150°F mill anneal.

The as-rolled microstructure of Heat 1014 is shown by Figure 14 and shows the absence of excess carbides and the partial recrystallization.

These heats were made by the carbon in the charge technique. Considerable overheating occurred during melting and were poured hot. Heat 1015 rolled better than any other heat within the preferred carbon range.

Carbon Rod Deoxidation

These heats were deoxidized after melt down by inserting carbon rods until gas evolution stopped. This led to high carbon since gas was evolved until the carbon content built up to 0.23 to 0.28%.

Heat	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1025	62	9	0.25	.0006	.0010
1031 (Aged)	112	18	.27	.0014	.0005
1032	90	11	.23	.0007	.0002
(Aged)	97	13			
1033	69	14	.28	.00016	.00015

The strengths were medium and the ductilities good. There did not seem to be a direct relation to gas content. A typical microstructure is shown by Figure 15.

Overheating occurred during melt down and the heats were held in the crucible for a considerable time while the deoxidation was carried out. They were also poured hot.

Variable Carbon, Oxygen and Nitrogen - Stack Melted

These heats were made to have a series in which it was intended that C, O₂ and N₂ would all be low followed by each interstitial element on the high side. Stack melting was used. Hydrogen deoxidation was used in some cases.

Heat	Deoxidation	Aim	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1048	C + H ₂	Low C, O ₂ +N ₂ (Aged)	44 33	3 5	.02	.0013	.0013
1049	C	Low C, O ₂ +N ₂ (Aged)	53 50	3 4	.04	.0007	.0006
1052	Carbon	Control normal C (Aged)	65 56	8 9	.09	--	--
1057	None	High O ₂ (Aged)	18 14	4 4	.03	.0058	.0024
1058	Carbon	High N ₂ by blowing N ₂ on melt (Aged) (S. T. 2150°F)(broke on loading)	33 19	3 5	.03	.00004	.0022

All of the heats were difficult to roll, presumably due to low carbon although Heat 1052 had reasonably high carbon.

All of the strengths were low and, with the exception of Heat 1052, the ductilities were also low.

The poorest heats were those in which attempts were made to make oxygen high (Heat 1057) and nitrogen high (Heat 1058).

The microstructures of Figure 16 show the absence of carbides and the relatively coarse grain size.

Table II includes data for bars rolled to smaller sizes which apparently cracked when water quenched from the mill. The 2150°F solution treat resulted in a very brittle material for the low carbon-high N₂ Heat 1058.

These data illustrate difficulties of obtaining more than one oxygen for a given carbon content. The nitrogen treatment appeared to have purged oxygen.

Temperatures during melting and pouring were quite variable and were high at one time or another.

Development of Procedures for Melting with Minimum of Overheat and Low Pouring Temperatures - Use of Pre-Vacuum Melted Alloy

Various technique changes were tried to prevent overheating and pouring temperatures were kept low in this series of heats. All heats were deoxidized with carbon in the charge and calcium, when used, was added before the Al and Ti.

Heat	Variable	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1064		65.8	4	.02	.0004	.00027
1066		18.9	3	.01	--	--
1074	Vac. melted Ni	17.9	4	.01	--	--
1076	Vac. melted Ni+Co	22.2	4	.01	--	--
1079	Vac. melted Ni+Co+Ca deoxid. 4.1Al-4.1Ti (2150°F S. T.)	54 204	3 5	(.08)	.0016	.00015
1081	Vac. melted Co+Ca deoxid. (1800°F S. T.) (2150°F S. T.) (2 hr 2150°F+1 hr 2150°F)	52 51.3 122.6 126.4	8 15 5 4	- - - -	-- -- -- --	-- -- -- --
1083	Vac. melted Cr-Ni+Co (Aged)	18	19.5	.10	--	--
1085	80Ni-20Cr Comm. Alloy (Aged)	25.8 14.3	13 32	.09	--	--
1096	High carbon	10.4	34	.36	.0019	.00045
1097	Calcium rod final deoxidation	13.2 26.2	15 11	.07	--	--

Heat 1079 gave the highest strength in the investigation when solution treated at 2150°F. It, however, was high in Al and Ti due to metal loss during deoxidation.

Heat 1081, with normal Al and Ti, came up when solution treated at 2150°F.

Considerable difficulty was encountered in the early heats due to overheating during melt down. It is thought that this combined with the low carbon was responsible for the varied and generally low rupture strengths. Heat 1064, however, had high strength for its carbon level.

In the latter heats the maintenance of low temperatures during melting plus low pouring temperatures seemed responsible for low strength. This probably accounts for the suggested tendency to improve in strength when treated at 2150°F. This is supported by the large elongations which decrease with the 2150°F treatment.

The microstructure of the 4Al-4Ti Heat 1079, Figure 17, shows a normal structure for carbon on the low side.

So far as could be determined the use of pre-vacuum melted alloys or the commercial 80Ni-20Cr alloy did not influence results.

Some of the heats showed severe banding, Figure 18. This has only been found in heats with less than 0.04% carbon. It is presumed that the banding is due to segregation related to low melting and pouring temperatures. Figure 18 shows the effect of a 0.5 hour soak at 2300°F prior to the last 40% reduction at 2150°F in reducing the banding. Hardness tests showed the bands to be 40VBH numbers harder than the clear areas.

It seems possible that segregation, whether or not it is severe enough to produce banding, may be involved in low strengths.

The low pouring temperatures were generally associated with difficult rolling even when carbon contents were normal.

Use of Solid Deoxidizers after Carbon Addition

Concern over low strengths, particularly for approximately 0.10 carbon heats, led to a series of experiments involving additions of solid deoxidizers after carbon in the charge deoxidation to see if strength and working characteristics would be improved. Low melting and pouring temperatures were maintained:

Heat	Variable	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1109	Control heat	86	8	.16	.0017	.00017
1110	CaSi prob. blown out (2150°F S. T.)	125 111	11 9	.18	.0005	.0003
1111	Si added-8.5 grams	84	5	.14	.0009	.0004
1112	Mn added-10 grams	111	15	.08	.0001	.0005
1113	2 gms. calcium rod	49	9	.03	--	--
1114	7 gms. calcium rod	55	8	.02	.0024	.0004
1115	Only calcium deoxidation	40	6	.03		

These values generally ran higher than had previously been obtained when the carbon was above 0.08%. Presumably, the two heats treated with calcium were low in strength due to low carbon. There is a suggestion that low gas content gave better strength. A 2150°F treatment did not bring up the strength of Heat 1110.

Special Deoxidation Before Adding Carbon

It was thought possible that low strengths might be due to the carbon-in-the-charge deoxidation suppressing an oxidizing period. Accordingly, the following heats were made by adding solid deoxidizers before adding carbon, avoiding the gas evolution problems of a carbon boil, and still melting with an oxidizing bath. High pouring temperatures were used:

Heat	Order of Additions	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1121	Al deoxid. -Al-Ti-C+Ca rod	27 25	19 28	.11	.0005	.0004
1122	Si-carbon-Al-Ti	42	44	.13	.0005	.0004
1123	Si-Al-carbon-Ti (2150°F S. T.) (Heated at 2300°F during rolling)	31 88 96	19 10 14	.14	.0007	.0004
1124	Al-C-Si deoxid. followed by Al-C-Ti additions	67 (2150°F S. T.) 55	8 17	.06	--	--

The ductilities were increased by this procedure but the strengths were low except when Heat 1123 was heated to 2300°F for 0.5 hours during rolling before the last 40% reduction or when the solution treating temperature was raised to 2150°F.

Varying Carbon in the Charge

These heats represent an attempt to melt down with high oxygen followed by the most vigorous carbon boil possible by adding lump carbon. The heats were held 30 minutes under vacuum before adding carbon.

Heat	Variable	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
1117	40% C in charge	51	10	.04	.0010	.0002
1118	20% C in charge	65	15	.05	.0002	.0002
1119	No carbon in charge	73	18	.05	.0017	.0002

There may have been some improvement in strength and ductility as a result of carrying out this procedure in view of the low carbons. Analyses for S and P did not show a reduction as a result of the 30 minute holding period or carbon boil.

Air Melted Heats

Heats were made early in the program by standard laboratory procedures for air melting. These were intended to be a base line to compare with the vacuum melted heats. The following results were obtained.

Heat	Rupture Time (hours)	Elongation (%)	C (%)	O ₂ (%)	N ₂ (%)
UA1	47	9	-	.0003	.0070
(Aged)	20	8			
UA2	38	1	-	.0009	.0019
UA4	13	5	.13	.0011	.0066
(Aged)	33	4			

These strengths are generally low although no lower than many of the many vacuum melted heats. They are generally similar to those heats deoxidized in the vacuum with solid deoxidizers before adding carbon, except that their ductility was low.

Gas analyses did not show particularly high values except possibly for nitrogen. This is presumably due to good separation of oxides to the slag. There were few inclusions as is shown by Figure 19.

As discussed previously, these heats rolled quite well.

6%Al + 6%Ti Heat

Heat 1095 was melted to obtain additional data regarding the influence of composition on basic structure and behavior of the alloy. A rupture test gave the following low strength and very high ductility:

2 hours for rupture with 48% elongation

The microstructure, Figure 20, shows the second phase which formed in the structure and which was not evident in other alloys. The second phase may have increased with solution temperature. It has been identified as probably Ni(Al, Ti).

STRUCTURAL ANALYSIS OF THE ALLOY

Work has been done using x-rays, the electron microscope, and electrical resistivity, along with hardnesses and optical microscopy. The purpose of this work was to provide a basis for suggestions as to how interstitial atoms, and other chemical variations, could affect strength in general and the microstructures in particular.

(1) Electron Microscopy

Figure 2 shows the change in structure of nickel-base alloys as a function of Ti-Al content. Inconel-X, Waspalloy, and the 3 Al-3 Ti, 4.1 Al-4.1 Ti, and 6 Al-6 Ti alloys have been used to show this progression. The small cubes in Inconel-X are assumed to be a Ni_3Al type structure. The increase in amount of Ni_3Al type structure in Waspalloy leads to many globular particles of indefinite shape. As the amount of Ni_3Al type structure $[\text{Ni}_3(\text{Al}, \text{Ti}, \text{Cr})]$ increases, the globular particles become more closely spaced until they tend to coalesce into comparatively large cubes. A very few of these large cubes were found in one 3 Al - 3 Ti alloy (Heat, 1015). Many were found in the 4 Al-4 Ti alloy. Furthermore, the 6 Al-6 Ti alloy shows another phase under the optical microscope (Figure 20) which is believed to be the intermetallic NiAl type compound.

Electron microscope work has shown that quench rates obtainable with ice-brine are required to prevent the formation of the above particles when quenching the 3 Al-3 Ti alloy from 1975°F. Even ice-brine quenching, of small specimens, was not rapid enough to prevent the formation of particles when quenching alloys higher in titanium and aluminum. Perhaps the large cubes, which sometimes appear as more irregular particles, form and are in equilibrium at the solution temperature of 1975°F. and the volume between them fills in with the "pig-skin" pattern of Ni_3Al on cooling (See Figure 21).

Electron microscopy has also indicated that precipitation must be nearly complete after a very short aging period. It appears that, at 1400°F, the amount of precipitate may increase some on aging, but generally the main alteration is merely the gradual coalescence of existing particles. However Figure 11 does show a hardness increase with aging time at 1400°F.

(2) X-Ray Data

The x-ray data are of two types: (1) Debye lines for phases other than the matrix, and (2) determination of lattice parameters.

Debye pictures have indicated that there are diffraction lines from the alloy other than those from the face-centered cubic matrix. It is assumed in view of the electron micrographs that these added lines arise from Ni_3Al type of compounds. It is recognized, however, that lattice spacings for Ni_3Al and for a possible order reaction in the matrix are so similar that definite proof of whether one or the other or both exists cannot be obtained from the x-ray diffraction patterns directly.

The lines obtained from 1095 are shown in Figure 22. This is the 6 Al-6 Ti alloy. The lines found in addition to the matrix were from:

1. Ni_3Al type of structure and/or ordered matrix
2. NiAl type of structure
3. Ni_3Ti type of structure.

The interpretation of these data is still incomplete. However, the new phase appearing in the optical micrographs of Figure 20 probably is NiAl type of structure.

One important characteristic that the x-ray data have indicated is that this ordering of the matrix or precipitation of Ni_3Al , takes place very rapidly, thus confirming similar electron microscope data. An ice-brine-quench at 1975°F is just rapid enough to eliminate these lines. A water quench or air cool will result in the reappearance of the lines. Ice-brine quenching, from 1975°F, of Waspalloy or a 3.0 - 3.0 Al-Ti alloy will result in an electron micrograph completely free from the precipitate.

It has been considered that the drastic quench might have induced sufficient stresses into the sample so as to smear out the lines beyond recognition. Large stresses are known to occur in the alloy during a rapid quench; many of the alloys with low ductility, usually with a high gas content, crack during even a water quench. But the above mentioned fact that the electron micrographs are clear after an ice-brine quench seems to eliminate

the stress interference theory; it also definitely indicates that the ordering or Ni_3Al forms at some temperature below 1975°F , at the 3-3 level of titanium and aluminum. Presumably, as the temperature of formation is reduced, the particle size is smaller for a given aging time and the hardness will increase for longer aging periods.

Lattice parameter data are not included because it is as yet incomplete.

(3) Electrical Resistivity

Electrical resistivity measurements have been made on some of the heats studied. Electrical resistivity changes normally parallel hardness changes in the sense that lattice or structural alterations which will cause hardness changes will also change the electrical resistivity. Hence, electrical resistivity was used to follow structural alterations at temperature.

Figure 25 shows the changes in electrical resistivity with temperature for three heats 1015, 1057 and U-500. It was hoped that electrical resistivity might be correlated with strength, or that it might be used to indicate an optimum heat treatment. No such relationship has been found.

Figure 24 shows a plot of resistivity vs. temperature for some compositions simpler than the 3Al - 3Ti standard alloy. This work was done for the purpose of obtaining a better understanding of the standard alloy.

The main factor observed from the resistivity data is that either disordering or resolution of Ni_3Al continues up to the solution treating temperature or about 2000°F . This is assumed from the reduction in resistivity from about 1600°F to 2000°F , see Figure 23.

Figure 23 shows resistivity as a function of aging time at 1400°F . The resistivity increased slightly when measured at the aging temperature, but when the sample was cooled by water quenching to room temperature and then measured, the resistivity appeared to decrease with aging. A similar type of behavior has been reported for other ordered matrix systems, and was assumed to be due to a change in the temperature coefficient of resistivity. These data further confirm the very rapid rate of reactions in this alloy; essentially very

little happens after one minute at 1400°F other than coalescence of existing particles. This latter statement is supported by the electron microscope studies.

(4) Status of Application of Structural Studies

As yet it has not been possible to relate this type of data to melting practice and gas content. Work in progress to interrelate melting variables to hardness data of the type in Figures 9, 10 and 11, resistivity measurements, electron micrographs, and x-ray diffraction data, including the lattice parameter results not included in this report. It is believed that the structural work is very near to the point where the basic metallurgy involved in the research can be clarified.

SUMMARY AND CONCLUSIONS

1. The rupture strengths of the best standard analysis heats is on the low side of the most recent strengths reported by the Utica Drop Forge Company.

It is possible that the mill anneal of two hours at 2150°F used by Utica contributed substantially to the strength and that some of the heats used in this investigation would have come up with similar treatment prior to the standard heat treatment.

2. Strength and ductilities at 1600°F are apparently affected by a complex inter-relationship of a number of factors:

(a) Strength and ductility increase with carbon content up to about 0.10%. Larger amounts apparently do not increase strength and probably reduce it by removing Ti as an inactive carbide.

(b) Conditions which indicate high O₂ and N₂ contents in the melt at the time Al and Ti are added generally are associated with low strength and ductility. This included air melting and melting down in a vacuum without deoxidation. The use of aluminum and other solid deoxidizers prior to the addition of carbon gave low strength but high ductility.

(c) High melt temperatures and high pouring temperatures were generally beneficial to strength. This combined with some improvement, particularly in ductility, from use of holding time and carbon boil suggests that refining through oxidation and cleansing involved in high temperatures and high vacuum with agitation is involved.

(d) There is some evidence that keeping pouring temperatures low results in a condition where either the use of higher temperatures heat treatments than 2150°F during working or higher solution treatments than 1975°F brings the strength up.

(e) Evidence of segregation in low strength heats together with the beneficial effects of high temperature treatments indicates that segregation is a very important factor in strength and ductility. This is supported by the finding of massive Ni₃Al particles in some 3 Al- 3 Ti heats. Probably the bands in low carbon-high gas heats is related to such phenomena.

3. There is considerable evidence that hot-working conditions are important to strength and while it cannot be supported by data the suggestion is that it probably is the single most important variable. One of the reasons for suspecting this was the high strength obtained on the ingots furnished by Utica where there was ample opportunity for homogenization and other variations and the one or two cases where high temperature treatments during rolling improved strength. There is also indications that working effects increase with decreasing carbon.

4. Difficulties were encountered in obtaining high gas contents in the heats. One factor is the carbon-oxygen equilibrium and probably an interrelation with nitrogen. Thus, the necessity of having about 0.10% carbon in the alloy restricts the gas content. If carbon deoxidation is omitted, the solid deoxidation products from the Al + Ti in the alloy apparently flux out so that it is impossible to build up the gas content at low carbon levels.

5. Hot workability increased with carbon content up to 0.30%. It was decreased by high gas contents in the vacuum melted heats. Treatments giving high gas content at the time Al and Ti are added to the heat, reduced workability. Low pouring temperatures also reduced workability. The air melted heats worked quite well.

The suggestion is that oxides and/or nitrides are detrimental to workability if below a certain size or are in solution. If the treatments produce larger grain refining inclusions and good separation to a slag are obtained, they are not as detrimental. Carbon content probably operates through the grain refining characteristics of excess carbides although the carbon-oxygen equilibrium might be involved as well. The reduction of the effective Ti content through carbide formation may also contribute to workability in high carbon heats. The evidence for improved workability through "refining" techniques was not conclusive.

It was shown that general surface cracking during working was associated with NiAl type of structure in high Al + Ti alloys suggesting that in some cases segregation causes the same thing to occur in the 3 Al - 3 Ti alloy.

6. The alloy exhibits complex structural reactions. Sufficient work has been done so that the fundamental structural factors are nearly clarified. It is expected that the influence of compositional, melting, hot-working, and heat treatment variables can be made general through application of the principles being developed. It is expected that definite structural differences connected with each variable can be developed through study of the microstructure by electron microscope techniques and identification of the phases present.

EFFECT OF REPEATED OVERHEATING ON THE RUPTURE PROPERTIES
AT 1500°F OF S816, HS31 AND M252 ALLOYS

by

John P. Rowe

Summary of Results to Date

Overheats in Absence of Stress

The results of the tests on samples in which the stress of rupture tests in progress at 1500°F was removed, the overheat applied, and the stress re-applied as soon as the sample cooled back to 1500°F are presented in Figures 26, 27 and 28 as percentage of normal rupture time. Temperatures of overheating were 1650°, 1800°, 1900° and 2000°F. These results are summarized as follows:

S816 Alloy:

Tests were run under two stresses: 16200 psi normally causing rupture in 1210 hours and 22000 psi causing rupture in 94 hours. Overheats were for two minutes every 12 hours under the lower stress and every five hours under the higher stress. Some tests used only a limited number of overheats while many were continued to fracture.

(a) Nearly all conditions gave lower rupture time with the damage increasing with temperature.

(b) The damage was greater for a stress of 16200 than 22000 psi.

(c) Relatively few overheats may have increased rupture strength under 16200 psi.

(d) As the overheat temperature was increased the degree of damage by a few overheats increased markedly. One 2-minute cycle to 2000°F reduced rupture life 63%.

(e) A saturation point beyond which no further damage occurred was reached in most cases.

HS31 Alloy:

These tests were similar to those for S816 except that the stresses were 23000 and 27,500 psi normally giving rupture in 670 and 90 hours.

(a) Overheating generally reduced rupture time similar to S816, as shown by Figure 27.

(b) It is uncertain if there was a difference at the two stresses due to inherent scatter in rupture times of the cast specimens.

(c) A saturation effect for no further damage was not found.

M252 Alloy:

The tests on M252 used higher stresses (shorter rupture times) in anticipation of increased life from overheating without stress. The 34000 psi stress caused rupture in 80 hours and 24000 psi in 490 hours in uninterrupted tests. The effects of the overheats were:

(a) Overheating to 1900° and 2000°F improved life in every case.

(b) The extent of improvement on a percentage basis was greater in the shorter tests.

(c) Overheating to 1650° or 1800°F resulted generally in a decrease in strength.

Overheating Under Stress

The tests were selected to check the addibility of rupture life under stress at the two temperatures existing during the test. The stress level at 1500°F was selected intermediate to those used in the absence of stress so as to have a sufficiently long rupture time to give significant results. These stresses and the uninterrupted rupture time at 1500°F were:

S816: 18000 psi - 520 hours for rupture

HS31: 24000 psi - 770 hours for rupture

The overheat conditions were selected to use approximately 30% use of rupture life at the high temperatures. To do this, the stress was either left constant or reduced to keep within the 30% limit. These stresses and the rupture times in uninterrupted rupture tests at the overheat temperatures were:

Material	Temperature (°F)	Stress (psi)	Average Rupture Time
S816	1650	18,000	14.2 hours
	1800	12,500	3.1 hours
		18,000	18 minutes
HS31	1650	24,000	5.9 hours
	1800	15,500	6.0 hours
		24,000	13 minutes

All tests were overheated twice a day until they failed. After a few tests it became evident that addibility of rupture life failed to account for all the damage. (See Table III). The following method seems to account for most of the damage and life can be calculated by adding the following fractions:

(1) Damage due to temperature cycling alone as calculated from Figures 26 and 27; and

(2) Damage from stress at overheat temperature by adding the total time at the high temperature and calculating the percentage of total interrupted rupture time.

The damage due to stress during heating and cooling seems negligible. The calculated rupture life by steps (1) and (2) is shown in the last column of Table III.

In all cases except HS31 overheated to 1650°F, the calculated time for rupture is still longer than the experimental value. Some tests have been made to suggest more scatter in data when heating is carried out by the resistance heating equipment. Also, there may be a stress effect such as was found in the tests overheated without stress.

Discussion of Results

The cause of damage from temperature alone in S816 and HS31 alloys is being studied. Several factors could be involved, the most important of which probably is agglomeration of precipitates at the high temperature probably causing more precipitation at 1500°F. It also seems probable that there is recovery from strain hardening.

In the case of M252 alloy it appears most probable that resolution of precipitates and thereby prevention of overaging is involved. The solution temperature is in excess of 1700°F so that heating to 1650° and for short times at 1800°F does not help. There is evidence as indicated in the work on 3 Al-3 Ti alloy previously cited that such reactions occur very rapidly in alloy hardened with Al + Ti. Consequently, even the brief times at 1900° and 2000°F could account for the strengthening.

The planned tests on S816 and HS31 are complete and the data is being prepared in report form. The tests on M252 overheated in the absence of stress are nearly complete and the tests under stress are in progress.

OTHER INVESTIGATIONS

The work on grain growth in blade alloys is being written as a report. Certain features of the grain growth work are apparently pertinent to work carried on to relate hot-working conditions in Waspaloy to strength at high temperatures. These results of the research on hot-working are being analyzed in preparation for a report with at most only a few additional check experiments needed.

Apparently large grains alone are not necessarily weak and brittle. Tests for the Materials Laboratory, WADC, on coarse grained samples show good strength in both smooth and notched rupture-test specimens. It appears, however, that coarse grains are often associated with other structural alterations which cause weakness and brittleness. It is hoped that the two programs will yield a better answer than as has been obtained to date for this problem.

TABLE I

Results of Chemical Analyses of Heats

Heat	Variable	C (%)	Vacuum Fusion		Kjeldahl		Ti (%)	Al (%)	Mo (%)	Cr (%)	Co (%)	Si (%)	Mn (%)	S (%)	P (%)
			Run	O (%)	N (%)	N ₂ (%)									
U-500	Made by Utica	.08	1	.00026	.0026	--	2.79 2.90	2.94 3.45	3.7 3.9	19.5 20.3	14.8 14.8	.50 .41	.17 .15	.004 .016	-- --
1014	Carbon in charge aimed for variable carbon content	.06	6	.0002	.0012	--	3.07 3.19*	3.29 3.22*	3.3	19.0	14.4	.21	.15	--	--
1015		.13	3	.00008	.003	.01	2.80 3.13*	3.17 3.16*	3.6	19.4	16.0	<.20	<.10	--	--
1016		.22	1	.0007	.0011	.01 .0056 .0054	2.68 2.96*	3.00 2.98*	3.5	19.4	15.3	<.20	<.10	--	--
1025	Carbon rod deoxidation	.25	3	.0006	.0010	<.005	3.23	3.50	3.7	20.9	16.1	.18	<.10	--	--
1031		.27	4	.0014	.0005	<.005	3.13	3.25	3.7	18.0	17.5	.20	<.10	--	--
1032		.23	5	.0007	.0002	<.005	3.27	3.20	3.4	18.0	15.2	.19	<.10	--	--
1033		.28	1	.00016	.00015	<.005	2.85 2.95*	3.23 2.85*	3.6	18.4	16.8	.18	<.10	--	--
Stack Melted - Variable Carbon, Oxygen and Nitrogen															
1048	Low C-C+H deoxidation	.02	1	.0013	.0013	.003	3.23	3.80	3.6	19.8	14.9	.30	<.10	--	--
1049	Low C-C deoxidation	.04	6	.0007	.0006	.0024 .003	3.05	3.55	3.5	19.2	14.9	.28	<.10	--	--
1052	Control heat	.09	--	--	--	--	3.08	3.50	3.5	19.0	14.2	.27	<.10	--	--
1057	High O ₂ -Low C+N No deoxidation	.03	1	.0058	.0024	.003 .003 .010									
1058	High N-C+H ₂ deoxidation+N ₂ blown on bath	.03	1	.00004	.0022	.007 .0056 .015									

* Wet chemical analysis

TABLE I (continued)

Heat	Variable	C (%)	Vacuum Fusion		Kjeldahl		Ti (%)	Al (%)	Mo (%)	Cr (%)	Co (%)	Si (%)	Mn (%)	S (%)	P (%)
			Run	O ₂ (%)	N ₂ (%)	N ₂ (%)									
Attempts to Melt Without Overheating with Control of Gas Evolution															
Use of Alloys Premelted in Vacuum															
1064	Carbon in charge	.02	1	.0004	.00027	.005	3.32	3.40	3.8	21.3	15.8	.26	.01	--	--
1066	Carbon in charge	.01	-	--	--	.006	3.00	3.20	3.85	19.5	15.2	.25	<.10	--	--
1074	Vacuum melted Ni Carbon in charge	.01	-	--	--	.004	3.07	3.28	3.8	20.6	15.2	.25	.10	--	--
1076*	Vacuum melted Ni +Co Carbon in charge	.01	-	--	--	.005	3.50	3.60	3.9	20.6	16.6	.26	.10	--	--
1079*	Vacuum melted Ni +Co Carbon in charge + Ca	--	-	.0016	.00015	--	6.0**	4.20	3.7	19.0	16.8	.25	<.10	--	--
1081	Vacuum melted Co Carbon in charge +Ca	--	-	--	--	--	3.25	3.40	4.10	20.7	15.8	.27	.11	--	--
1083	Cr-Ni alloy + vacuum melted cobalt-carbon in charge.	.10	-	--	--	.008	--	--	--	--	--	--	--	.019	--
1085	80Ni-20Cr alloy + Cr- Ni + Co vacuum melt- ed-carbon in charge	.09	-	--	--	--	--	--	--	--	--	--	--	.023	--
1096	Carbon in charge	.36	1	.0019	.00045	--	--	--	--	--	--	--	--	--	--
1097	Carbon in charge Calcium rod	.07	-	--	--	--	--	--	--	--	--	--	--	--	--

* Metal loss due to excessive rate of gas evolution

**4.1 Ti 4.1Al based on additions to melt.

TABLE I (continued)

Heat	Variable	C (%)	Vacuum Fusion			Kjeldahl		Ti (%)	Al (%)	Mo (%)	Cr (%)	Co (%)	Si (%)	Mn (%)	S (%)	P (%)
			Run	O ₂ (%)	N ₂ (%)	N ₂ (%)										
Melted with Solid Deoxidizers after Carbon Deoxidation																
1109	Control heat; carbon in charge	.16	3	.0017	.00017	--	--	--	--	--	--	--	--	--	.015	--
1110	Carbon in charge CaSi(prob. lost)	.18	1	.0005	.00030	--	--	--	--	--	--	--	--	--	.014	--
1111	Carbon in charge Silicon added	.14	2	.0009	.00037	--	--	--	--	--	--	--	--	--	.020	.007
1112	Carbon in charge Mn added	.08	1	.0001	.00053	--	3.25	3.35	4.1	20.0	14.1	<.10	.46	.020	--	--
1113	Carbon in charge 2gms calcium rod	.03	-	--	--	--	3.22	3.38	4.1	20.0	14.2	<.10	<.10	.020	--	--
1114	Carbon in charge 7gms calcium rod	.02	2	.0024	.00043	--	3.02	3.07	3.9	20.0	15.0	.21	<.10	.018	.006	--
1115*	Calcium only	.03	-	--	--	--	4.30	4.15	4.5	20.9	15.0	.23	<.10	--	--	--
Special Deoxidation before Carbon Addition																
1121	Al deoxidation + Al + Ti + Carbon + calcium rod	.11	3	.0005	.00038	--	2.74	2.80	3.8	19.8	15.5	.21	<.10	.015	.006	--
1122	Silicon + carbon before Al + Ti	.13	2	.0005	.00035	--	3.50	3.40	4.1	19.8	14.1	.11	<.10	.014	--	--
1123	Silicon + Al + carbon + Ti	.14	1	.0007	.00043	--	3.33	3.28	4.0	19.8	15.0	.14	<.10	.016	--	--
1124	Al + carbon + silicon + Al + C + Ti	.04	-	--	--	--	--	--	--	--	--	--	--	.021	.004	--
* Part of heat lost during gas evolution																

* Part of heat lost during gas evolution

TABLE I (continued)

TABLE II

Stress-Rupture Data

Heat	Size Ingot (lbs)	Solution Treatment		Aged 24 hrs. at 1550°F 16 hrs. at 1400°F	Vickers Hardness	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in)	Red. of Area(%)	C (%)	Comments
		hrs.	(°F)								
Alloy Made by Utica Drop Forge											
Carbon in Charge - Aimed for Variable Carbon											
U500	12	4	1975	None		25,000	312.8	13.6	17.5	.08	
	12	4	1975	None		25,000	257.8	18.3	23.2		
	12	4	1975	Aged		25,000	329.0	15.0	18.1		
1014	10	4	1975	None	374	25,000	32.7	6.5	1.6	.06	
	10	4	1975	Aged	402	25,000	32.2	3.0	1.6		
	1015	10	4	1975	None	358	25,000	160.4	4.0	4.0	.13
10		4	1975	None	-	32,500	19.5	5.7	4.0		
10		4	1975	Aged	389	25,000	60.4	6.9	6.4		
1016	10	4	1975	None	348	25,000	103.6	4.9	7.1	.22	Rolled to 1/2'sq.
	10	4	1975	None	-	25,000	66.4	5.0	5.6		
	10	4	1975	Aged	371	25,000	81.2	6.0	5.6		
Carbon Rod Deoxidation (0.23/0.28%C)											
1025	10	4	1975	None	398	25,000	62.1	9.0	7.9	.25	1/2"sq.
1031	10	4	1975	Aged	380	25,000	112.7	18.0	11.0	.27	
1032	10	4	1975	None	351	25,000	89.6	11.0	13.8	.23	
	10	4	1975	Aged	402	25,000	97.4	13.0	14.4		
1033	10	4	1975	None	362	25,000	19.9	17.0	29.5	.28	1/2"sq. 1/2"sq.
	10	4	1975	None	-	25,000	69.0	15.0	16.0		
	10	4	1975	None	-	25,000	69.1	14.0	16.2		

TABLE II (continued)

Heat	Size Ingot (lbs)	Solution Treatment hrs.	Aged 24 hrs. at 1550°F 16 hrs. at 1400°F	Vickers Hardness	Stress (psi)	Rupture Time (hours)	Elongation Red. of (% in 1 in) Area(%)	C (%)	Comments
Stack Melted - Variable C, O ₂ and N ₂									
1048	10	4	1975	348	25,000	44.4	3.2	.02	C+H ₂ deoxid.
	10	4	1975	359	25,000	33.4	0		
1049	10	4	1975	333	25,000	52.7	1.6	.04	C deoxid.
	10	4	1975	408	25,000	49.6	5.6		
1052	10	4	1975	345	25,000	65.5	8.6	.09	Control heat
	10	4	1975	392	25,000	56.0	8.6		
1057	10	4	1975	359	25,000	18.3	2.4	.03	No deoxid.
	10	4	1975	-	25,000	Broke on loading	4.0		(1/2'sq. W.Q.
	10	4	1975	-	25,000	14.3	1.0		from S. T.)
1058	10	4	1975	362	25,000	32.9	1.6	.03	N ₂ blown on
	10	4	1975	-	25,000	4.2	0.8		melt for 15 min.
	10	1	2150	-	25,000	Broke on loading	4.0		(1/2" sq.)
	10	4	1975	-	25,000	19.5	1.0		
Attempts to Melt Without Overheating and Controlled Gas Evolution									
Use of Pre-Vacuum Melted Alloys									
1064	10	4	1975	-	25,000	65.8	4.8	.02	C in charge
1066	10	4	1975	-	25,000	18.9	0	.01	C in charge
1074	10	4	1975	-	25,000	17.9	0	.01	Vac. melted Ni
1076	10	4	1975	-	25,000	22.2	4.0	.01	Vac. melted Ni+G
1079	10	4	1975	-	25,000	54.3	6.3	-	Vac. melted Ni+
	1	1	2150	-	25,000	204.0	5.0	-	Cot+Ca deoxidized

TABLE II (continued)

Heat	Size Ingot (lbs)	Solution Treatment hrs.	Aged 24 hrs. at 1550°F 16 hrs. at 1400°F	Stress (psi)	Rupture Time (hours)	Elongation Reduction of		C (%)	Comments
						(% in l in)	Area (%)		
1081	10	4	1975	25,000	52.1	8.0	13.5	-	Vac.melted Co+Ca
		4	1800	25,000	51.3	15.0	14.7		deoxidized
		1	2150	25,000	122.6	5.0	4.8		
		2	2150						
		plus							
		1	2150	25,000	126.4	4.0	3.2		
1083	10	4	1975	25,000	18.1	19.5	23.4	.10	Vac.melted Cr-Ni+ Co
1085	10	4	1975	25,000	25.8	13.4	19.2	.09	80Ni-20Cr alloy + Vac.melted 50Cr- 50Ni + Co
		4	1975	25,000	14.3	22.8	31.8		
1096	4	4	1975	25,000	10.4	34.1	37.9	.36	C-carbon in charge
1097	4	4	1975	25,000 ⁽¹⁾	13.2	14.9	12.4	.07	Calcium rod
		4	1975	25,000 ⁽²⁾	26.2	10.9	14.4		(1)bottom of ingot (2)top of ingot
Melted with Solid Deoxidizers after Carbon Deoxidation									
1109	4	4	1975	25,000	86.0	6.3	7.9	.16	Control heat
1110	4	4	1975	25,000	125.0	11.0	9.8	.18	CaSi (prob. lost)
1111	4	1	2150	25,000	111	9.0	7.9		
		4	1975	25,000	83.7	4.7	8.6	.14	Silicon added
1112	4	4	1975	25,000	111.4	15.6	8.0	.08	Mn added
1113	4	4	1975	25,000	49.2	9.3	5.6	.03	2gm. Ca rod
1114	4	4	1975	25,000	55.3	8.6	4.8	.02	7gm. Ca rod
1115	4	4	1975	25,000	40.5	6.0	4.8	.03	Ca deoxid. only

TABLE II (continued)

Heat	Size Ingot (lbs)	Solution Treatment hrs.	Aged 24 hrs. at 1550°F 16 hrs. at 1400°F	Stress (psi)	Rupture Time (hours)	Elongation Reduction of (% in 1 in)	C (%)	Comments
Special Deoxidation Before Carbon Addition								
1121	4	4	1975	25,000	24.6	28.0	16.0	.11 Al deoxid. before Al+Ti+Ca rod
			1975	25,000	27.0	19.0	16.0	
1122	4	4	1975	25,000	15.2	41.8	44.4	.13 Silicon+Carbon before Ti + Al
1123	10	4	1975	25,000	30.8	18.8	15.2	.14 Si+Al before Carbo and Ti+Al
		1	2150	25,000	88.0	10.0	8.7	
		4	1975	25,000	96.0	14.0	8.6	
			(2300°F during rolling)					
1124	10	4	1975	25,000	55.1	17.0	13.0	.06 Al followed by C+ Si+Al before adding final C+Ti+Al
		1	2150	25,000	66.6	8.0	7.1	
Varying Carbon in Charge								
1117	4	4	1975	25,000	54.1	7.8	7.1	.04 40%carbon in charge
1118	4	4	1975	25,000	65.4	15.0	6.4	.05 20%carbon in charge
1119	4	4	1975	25,000	73.3	18.0	17.6	.05 No carbon in charge
Air Melted Heats								
UA1	12	4	1975	25,000	46.8	8.8	3.1	-
		4	1975	25,000	19.5	7.7	2.4	-
UA2	12	4	1975	25,000	37.9	0.9	0.8	-
		4	1975	25,000	26.2	2.9	4.0	-
		4	1975	25,000	16.6	2.8	3.2	-
UA4	12	4	1975	25,000	12.7	5.0	2.4	.13
		4	1975	25,000	32.7	3.8	0.0	-
6%Al + 6% Ti								
1095	4	4	1975	25,000	1.9	48.0	73.0	.25

TABLE III

Overheats under Load

Material	Normal Stress (psi)	Overheat		No. of Cycles	Rupture Time (hours)	Elongation (%)	Calculated Time (hours)
		Stress (psi)	Temp (°F)				
S816	18,000	12,500	1800	14	186.7	38	235
	18,000	12,500	1800	13	156.1	37	255
	18,000	18,000	1800	1	346.8	44	385
	18,000	18,000	1650	27	347.6	39	405
HS31	24,000	15,500	1800	13	161.0	19	260
	24,000	15,500	1800	15	179.0	14	208
	24,000	15,500	1800	15	175.6	19	208
	24,000	15,500	1800	15	187.8	17	208
	24,000	24,000	1800	1	237.0	17	615
	24,000	24,000	1800	1	167.3	21	615
	24,000	24,000	1650	29	375.6	8	270
	24,000	24,000	1650	33	410.6	14	310

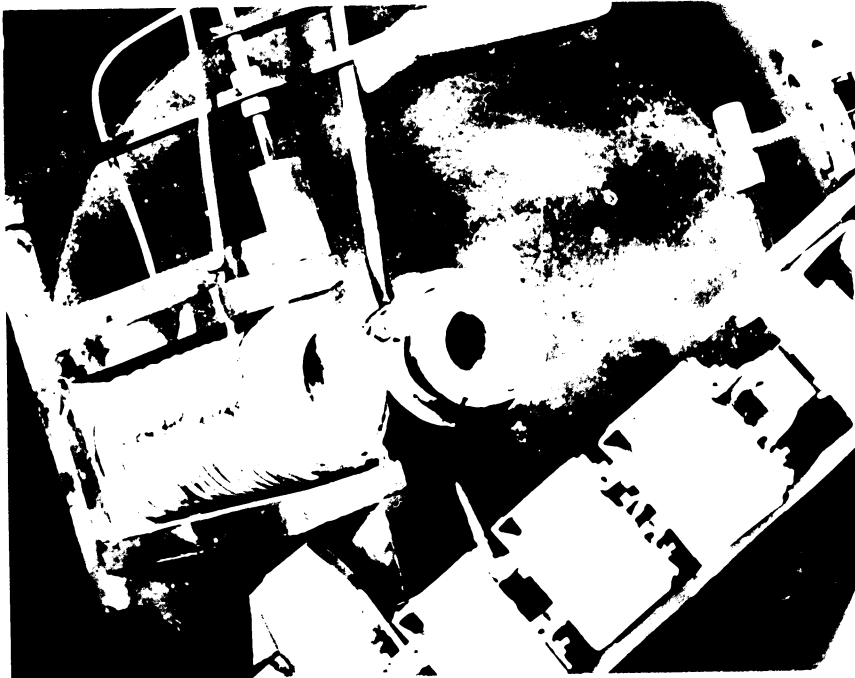
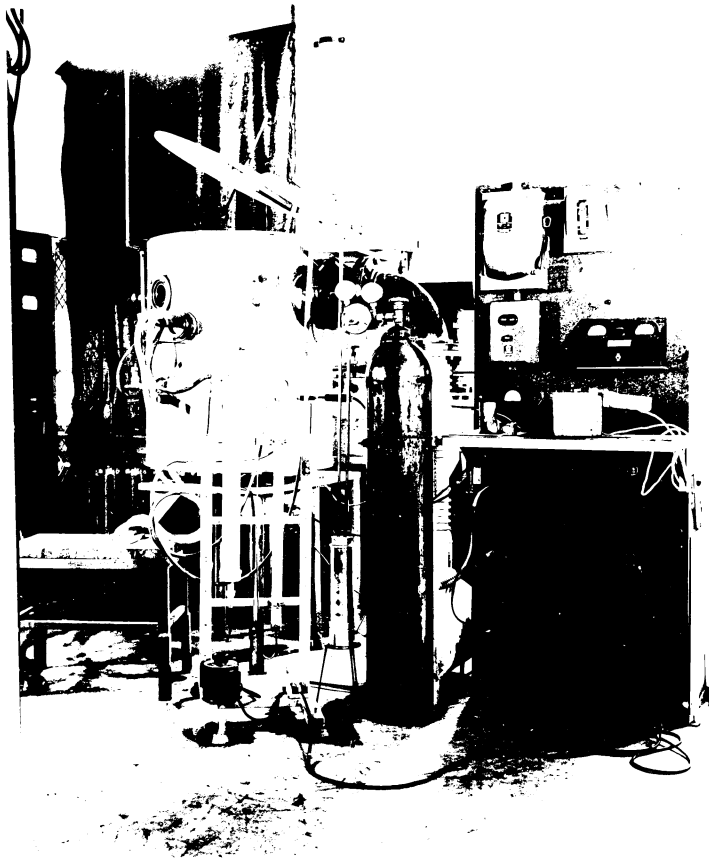


Figure 1. - External and internal views of vacuum melting unit.

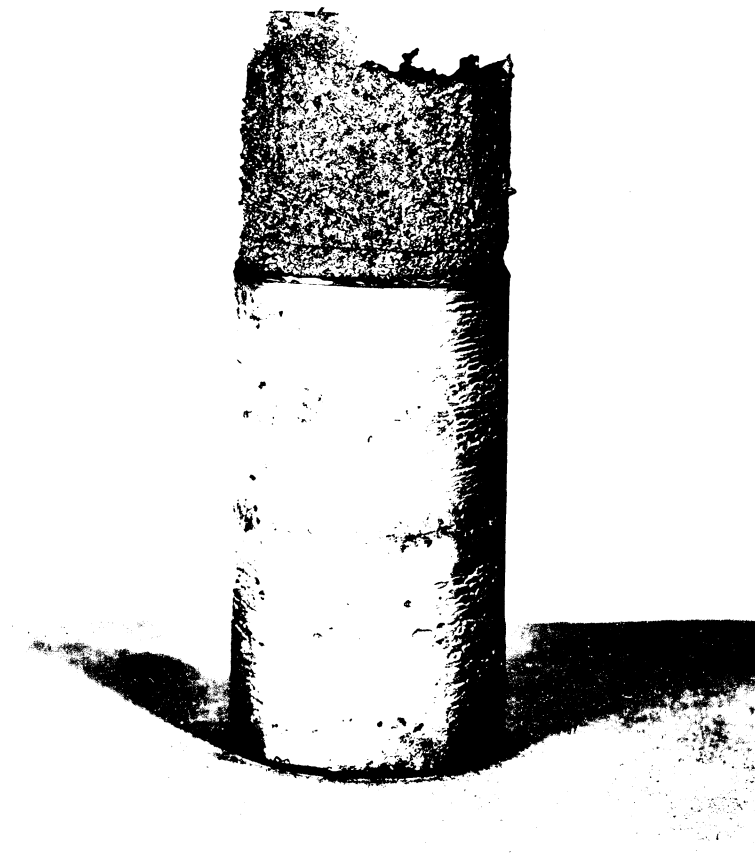


Figure 2. - Typical vacuum cast ingot; 9 inches overall length by 2-5/8 inches diameter, weight 10 pounds.

Gas Content (weight percent)

0.0024
0.0020
0.0016
0.0014
0.0012
0.0010
0.0008
0.0006
0.0004
0.0002

1

2

3

4

5

6

Sample Number

Figure 3. - Vacuum fusion analyses of identical samples from bar stock. Oxygen and nitrogen contents versus sample number of a series for Heats 1016 and 1058. Degassing temperature 1850°C.

O₂ - 1016

N₂ - 1058

O₂ - 1058

N₂ - 1016

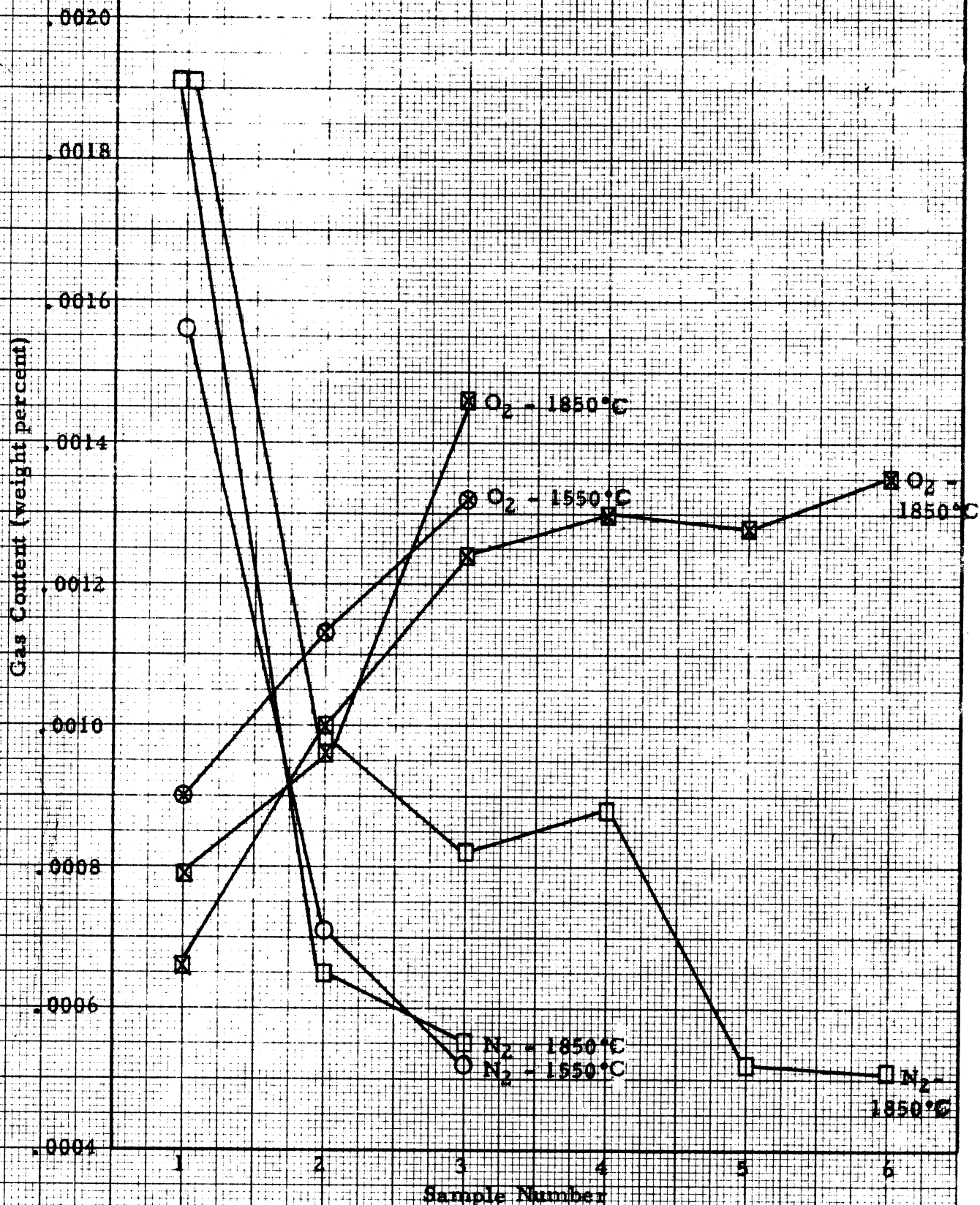


Figure 4. = Vacuum fusion analyses of identical samples from bar stock of Heat 1054 which contained no Ti or Al. De-gassing temperatures of 1550°C and 1850°C.

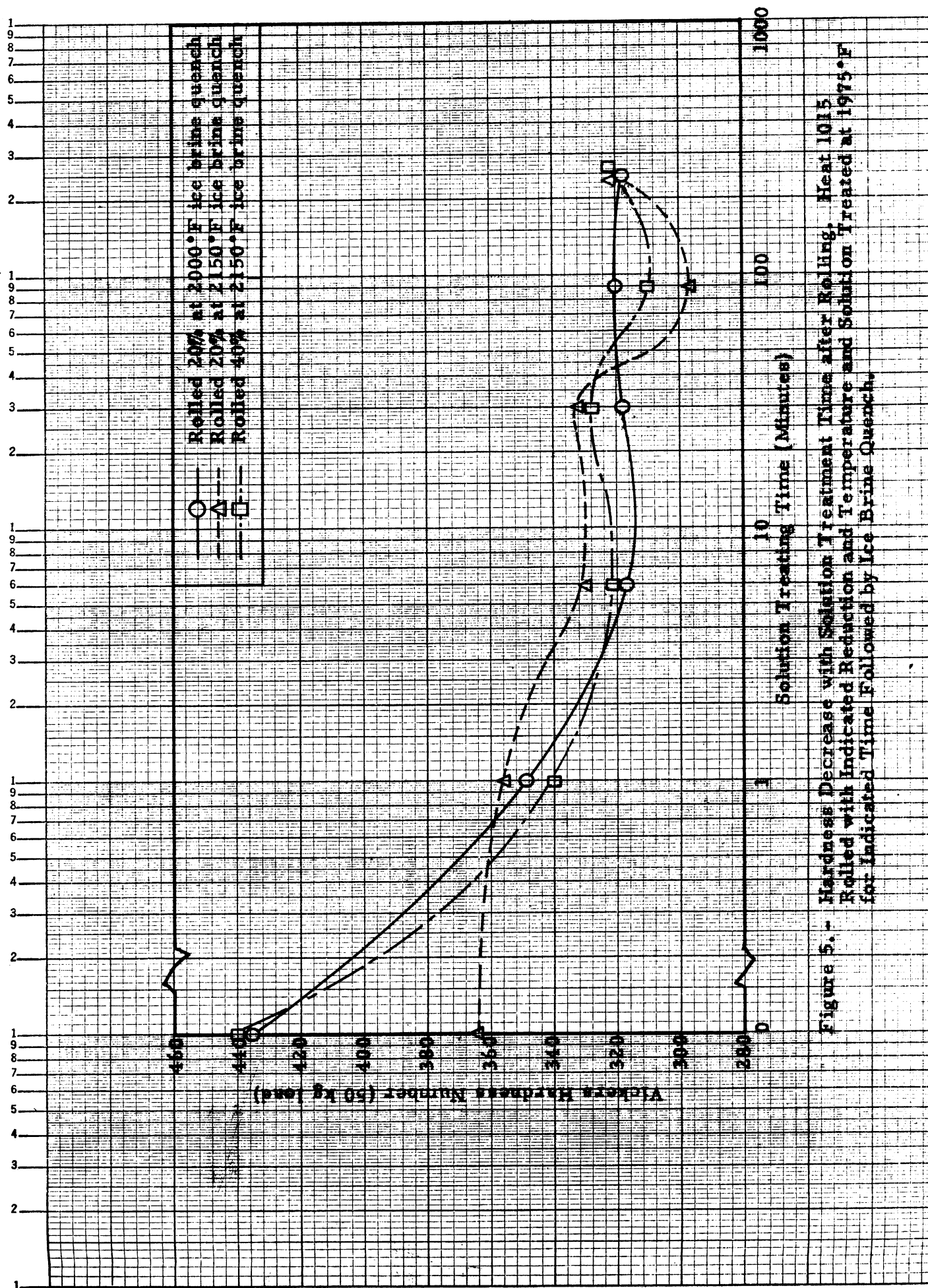
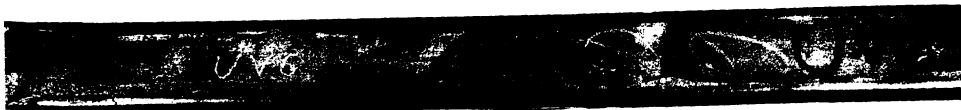


Figure 5. - Hardness Decrease with Solution Treatment Time after Rolling, Heat 1015
 Rolled with Indicated Reduction and Temperature and Solution Treated at 1975°F
 for Indicated Time Followed by Ice Brine Quench.



Heat 1079: 4.1% Al +
4.1% Ti



Heat 1032: 0.23% C



Heat UA4: 0.13% C
(Air Melted)



Heat 1123: 0.15% C
(Al deoxidized)

Figure 6. - Appearance of Rolled Bars of 4.1% Al + 4.1% Ti and Standard Composition Melted in Air and in Vacuum.



Heat 1079: 4.1% Al +
4.1% Ti



Heat 1032: 0.23% C



Heat UA4: 0.13% C
(Air Melted)



Heat 1123: 0.15% C
(Al deoxidized)

Figure 6. - Appearance of Rolled Bars of 4.1% Al + 4.1% Ti and Standard Composition Melted in Air and in Vacuum.


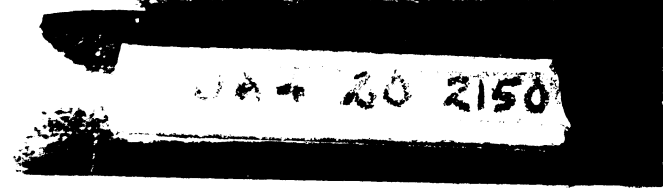

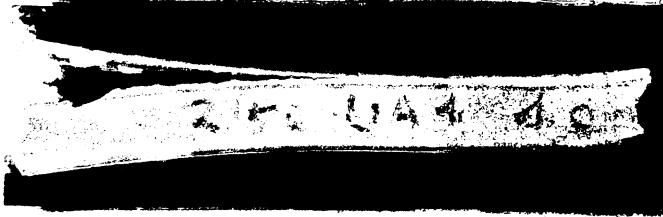

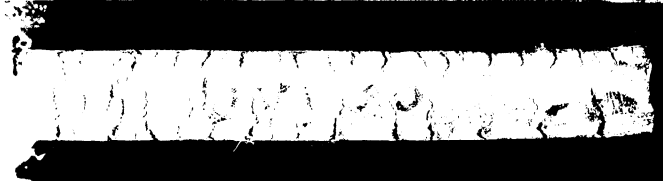

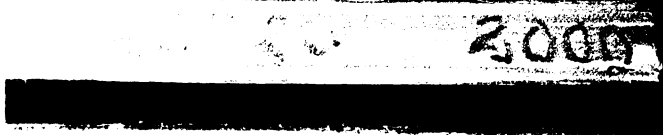

Heat 1014-.06%C		Vacuum melted 20%, Reduction from 2150°F
Heat UA4-0.13%C		Air melted 20%, Reduction from 2150°F
		Waspaloy 20%, Reduction from 2150°F
Heat UA4		Air melted 40% Reduction from 2150°F
		Waspaloy 40%, Reduction from 2150°F
Heat 1014		Vacuum melted 20%, Reduction from 2000°F
Heat UA4		Air melted 20% Reduction from 2000°F
		Waspaloy 20%, Reduction from 2000°F
		Waspaloy 40%, Reduction from 2000°F

Figure 7. - Comparison of Workability at 2000°F and 2150°F of Vacuum Melted and Air Melted 3Al - 3Ti alloy and Waspaloy Bar Stock. Reduced with No Reheats.

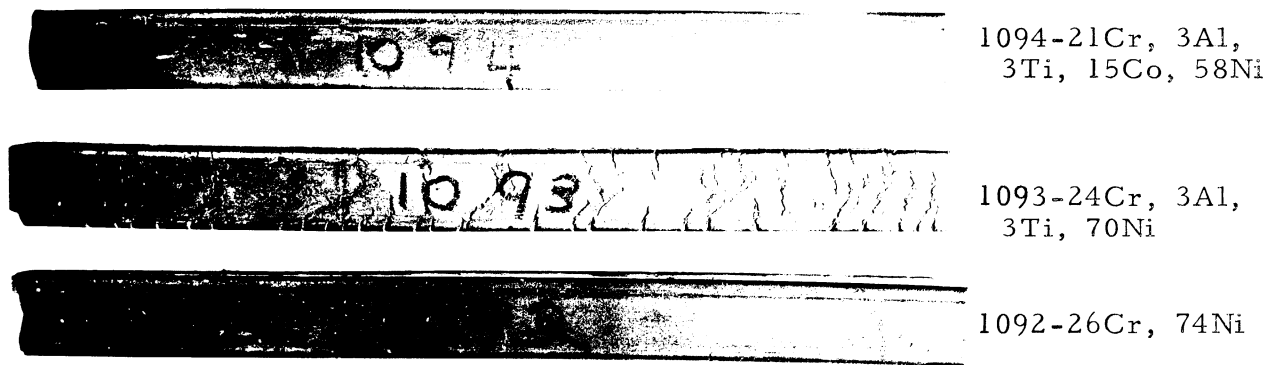


Figure 8. - Influence of Aluminum plus Titanium and Cobalt on Hot Rolling Characteristics of Nickel Base Alloys.

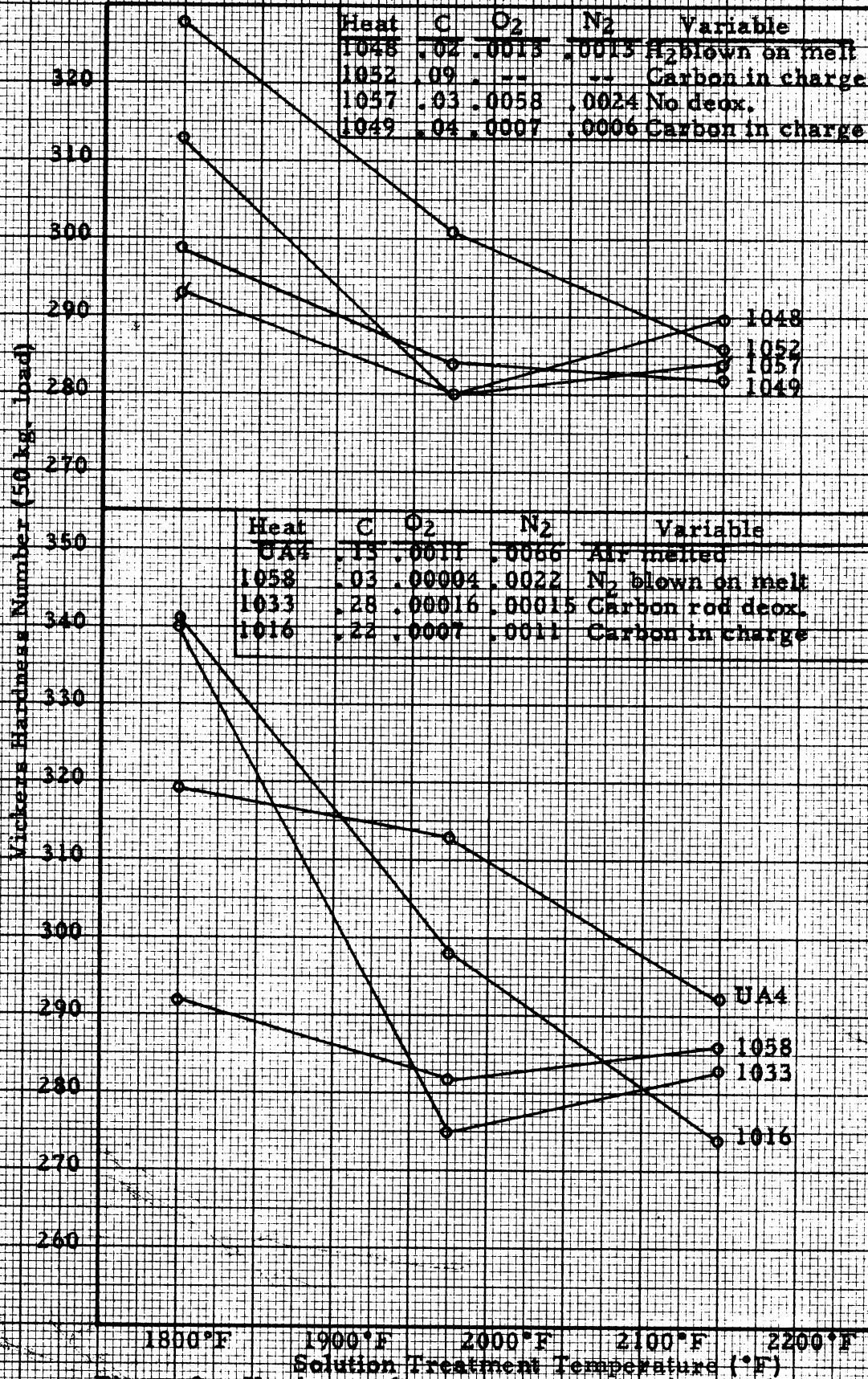
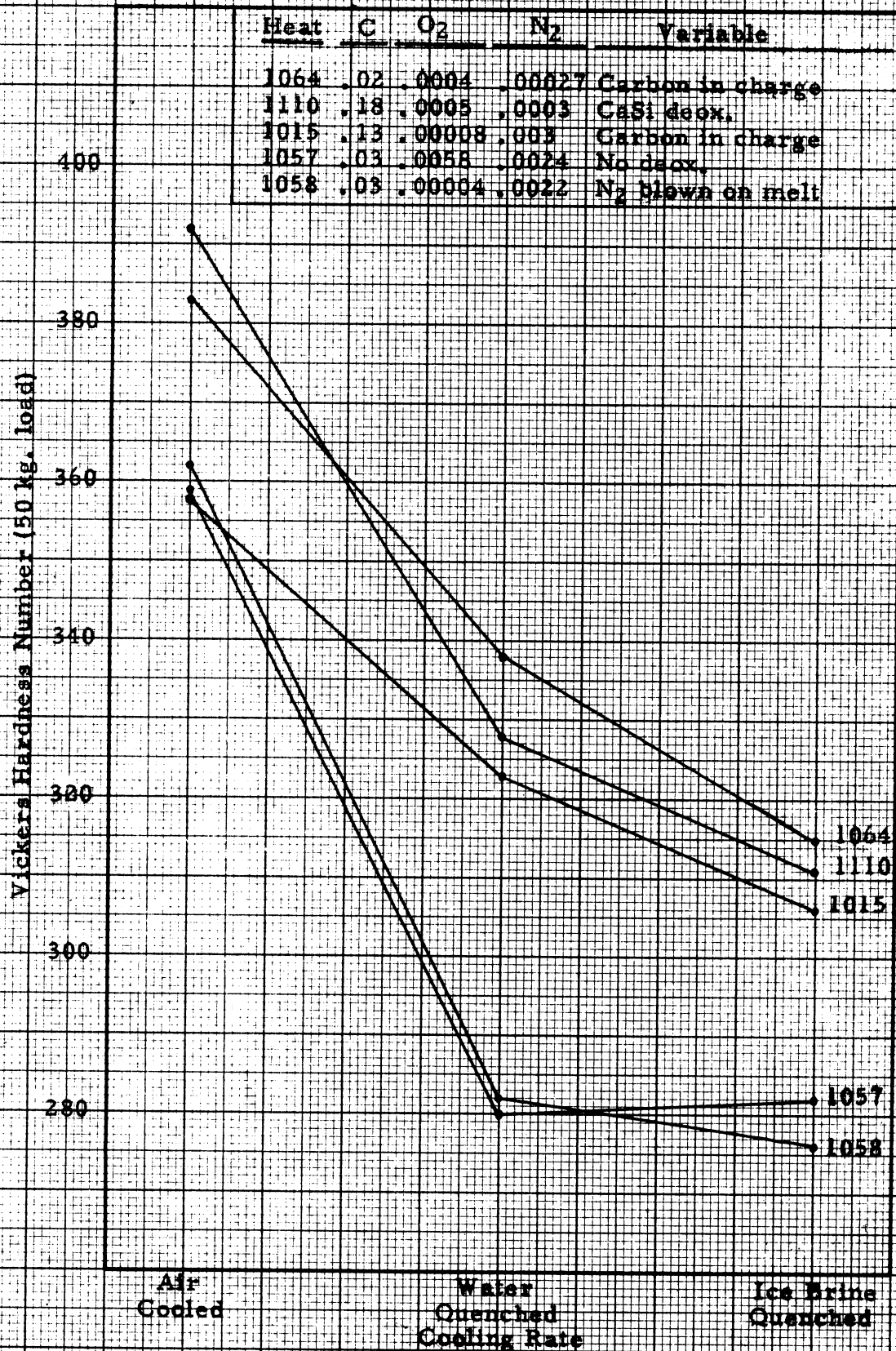


Figure 9. - Hardness after water quenching from solution treatment as affected by solution temperature for several heats.



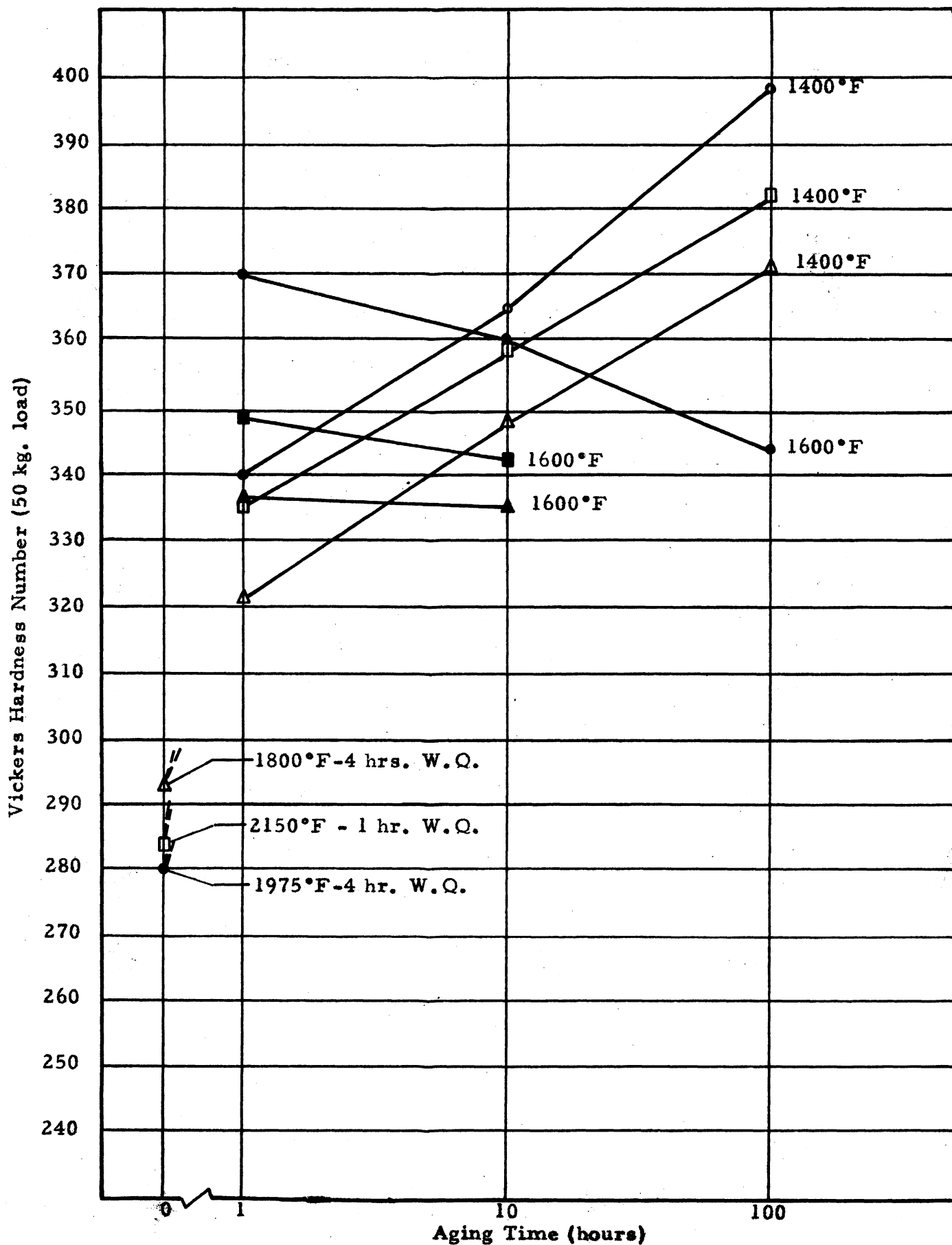


Figure 11a. - Influence of solution treating temperature and aging temperature and time on hardness. Heat 1057, C - .03, O₂ - .0058, N₂ - .0024. No deoxidation.

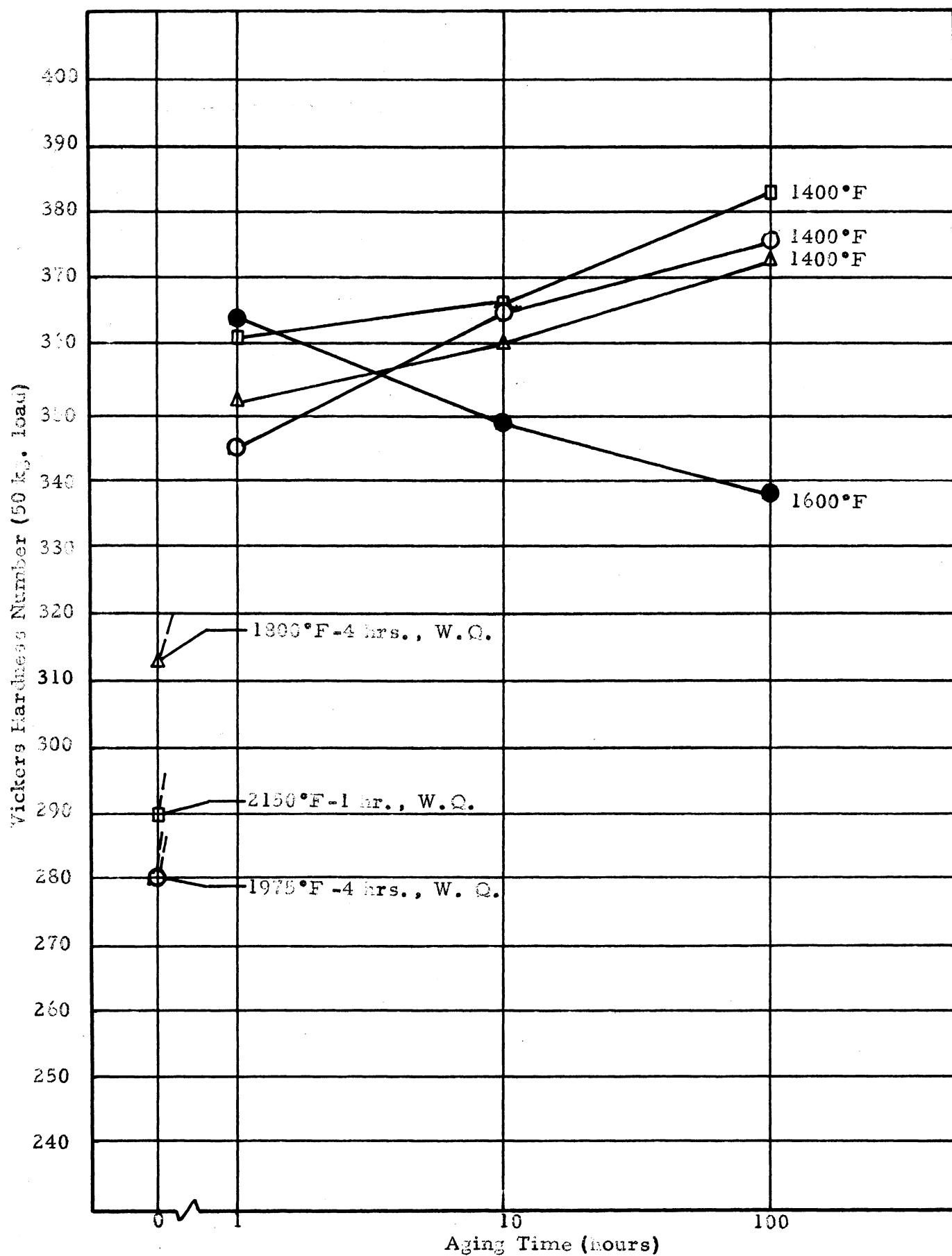


Figure 11b.- Influence of solution treating temperature and aging temperature and time on hardness. Heat 1048, C - .02, O₂ - .0013, N₂ - .0013.

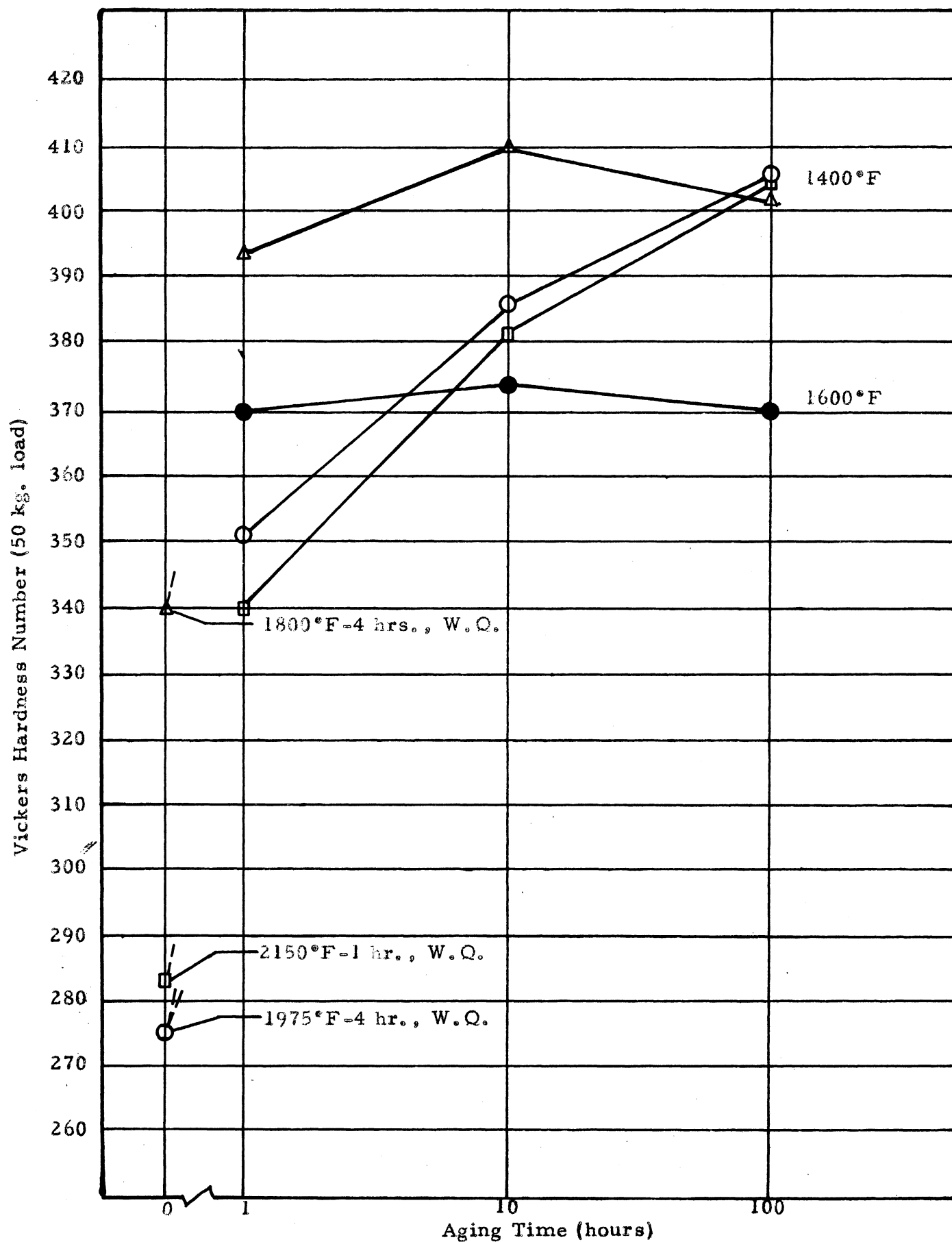


Figure 11c. - Influence of solution treating temperature and aging temperature and time on hardness. Heat 1033, C-.28, O₂-.00016, N₂ -.00015. Carbon rod deoxidized.

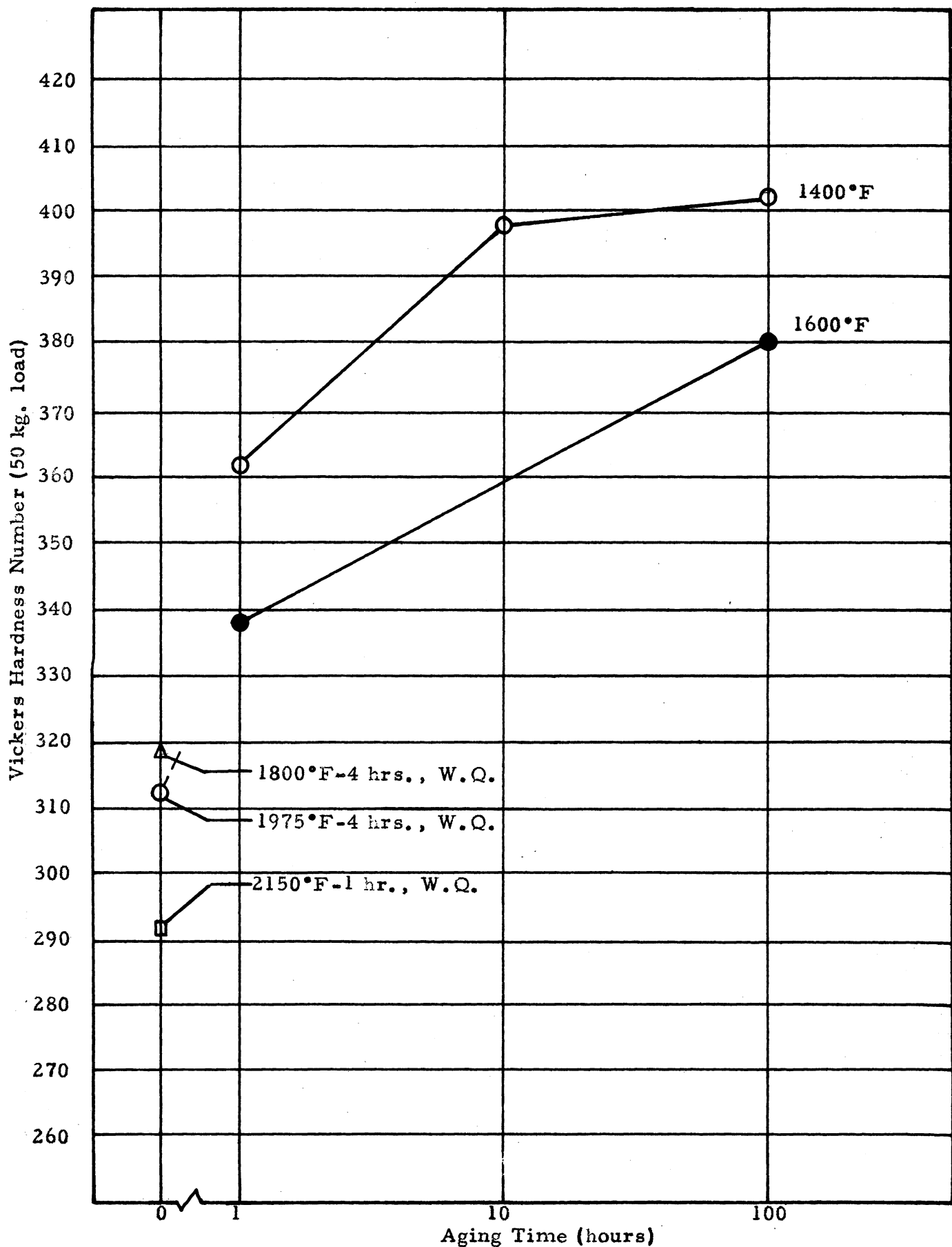


Figure 1ld. - Influence of solution treating temperature and aging temperature and time on hardness. Heat UA4, C - .13, O₂ - .0011, N₂ - .0066. Air melted.

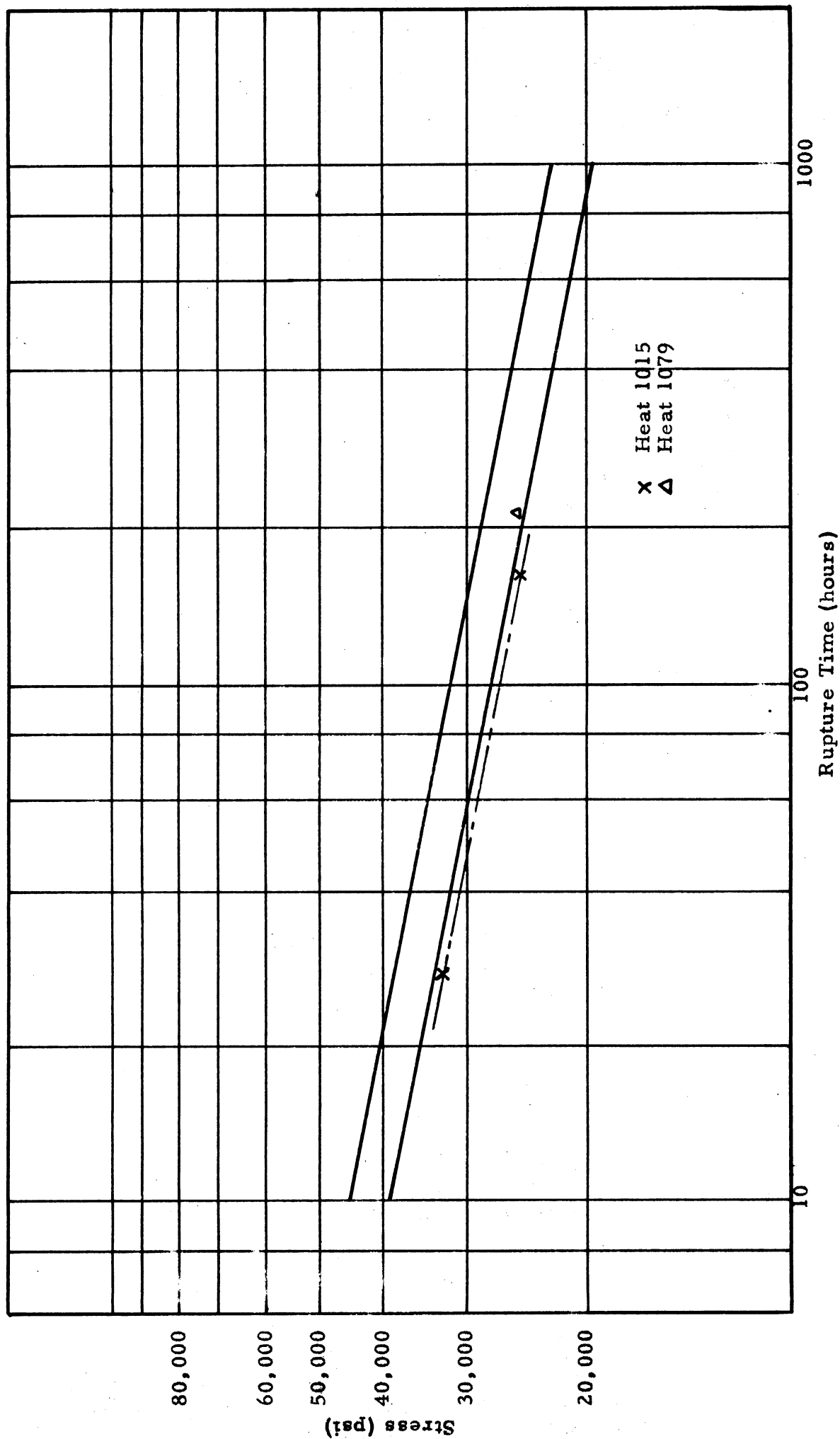
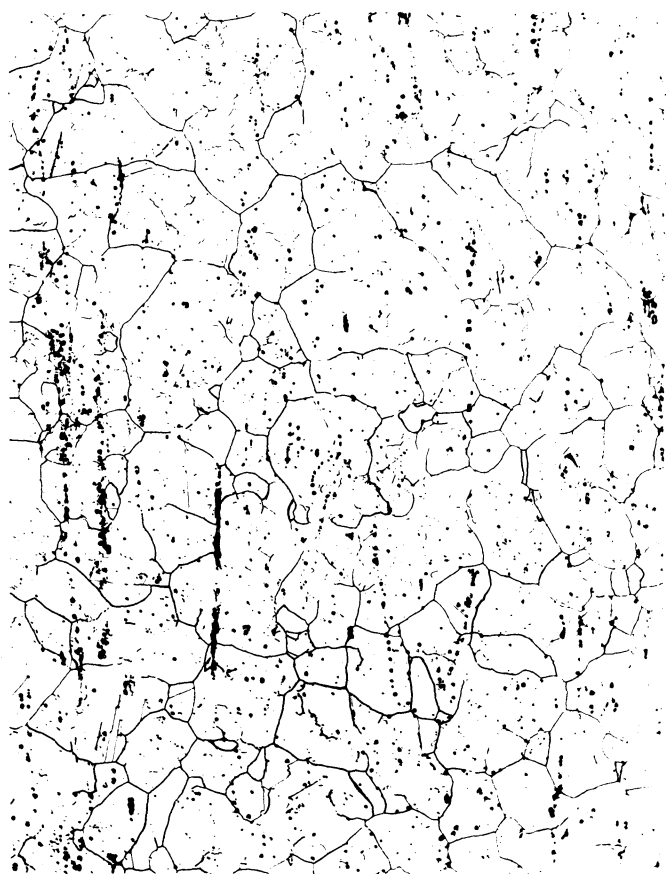
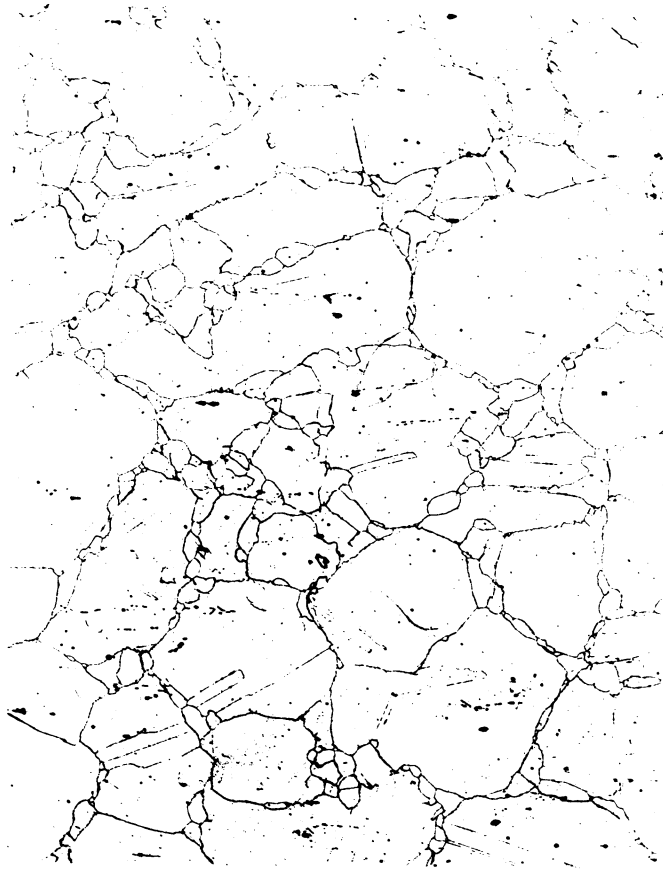


Figure 12.- Rupture strength band at 1600°F for two heats of UDIMET 500 as reported by Utica Drop Forge Company. Heat treatment: mill anneal 2 hours at 2150°F plus 4 hours at 1975°F, air cool; 24 hours at 1550°F, 16 hours at 1400°F. Also two values are shown for Heat 1015(4 hrs. at 1975°F, air cool) and one value for Heat 1079 (1 hr. at 2150°F), air cool.



X100

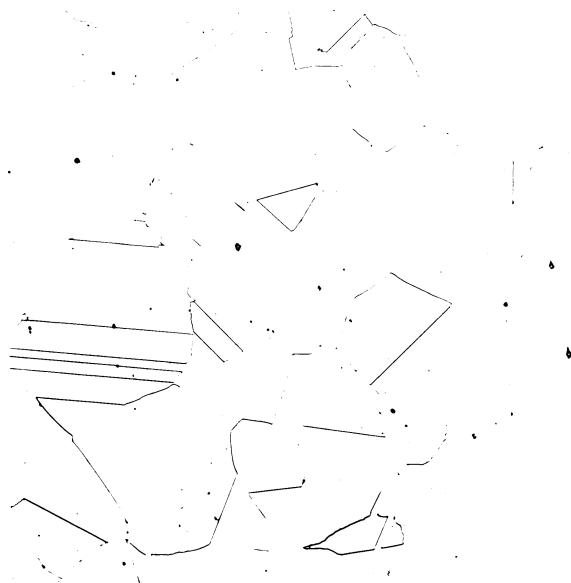
Figure 13. - Microstructure of Heat U500 made by Utica Drop Forge Company. Specimen was solution treated 4 hours at 1975°F, air cooled and aged 10 hours at 1400°F. Rolling direction is vertical.



X100

Figure 14.- Microstructure of Heat 1014 (.06%C) in as rolled condition.

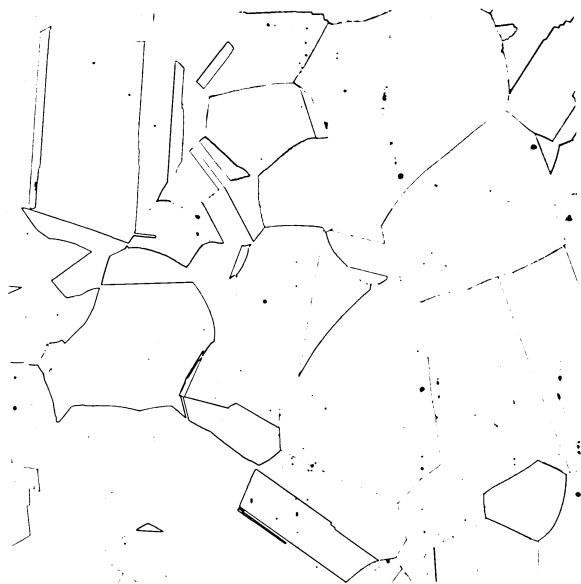
See file copy
for fig. 15-



X100
1057-4 hours at 1975°F, water quench.



X100
1057-4 hours at 1975°F, water quench
plus 10 hours at 1400°F.

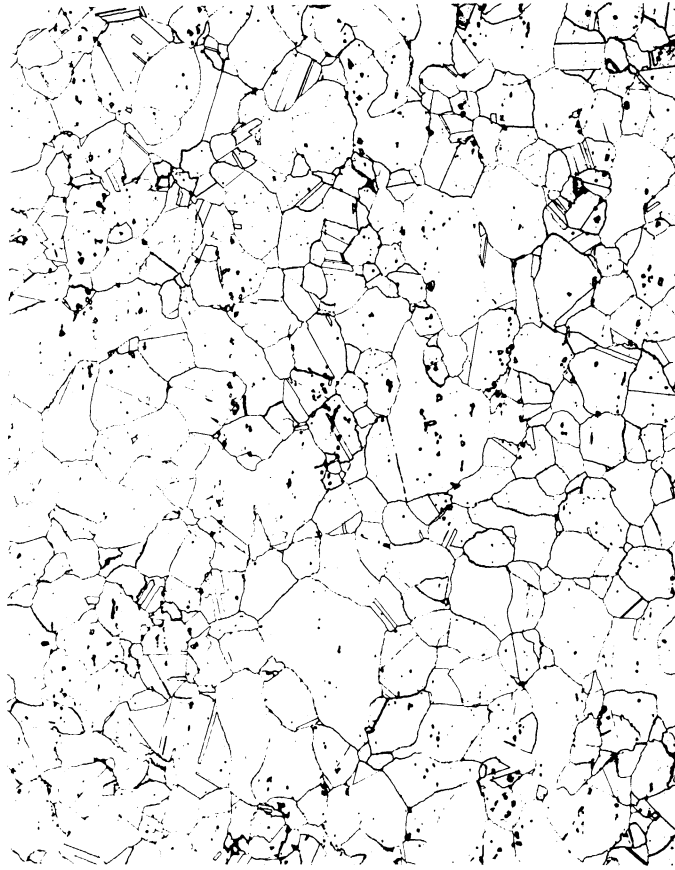


X100
1058-4 hours at 1975°F, water quench
plus 10 hours at 1400°F.



X100
1058-4 hours at 1975°F, water quench
plus 100 hours at 1400°F.

Figure 16. - Microstructures of Heat 1057 (.03%C, no deoxidation) and Heat 1058 (.03%C, N₂ blown on surface) after indicated solution treatment and aging. Rolling direction is vertical.



X100

Figure 17. - Microstructure of Heat 1079 (4.1%Al, 4.1%Ti) after solution treatment of 4 hours at 1975°F, ice brine quench plus aging of 100 hours at 1400°F. Rolling direction is vertical.



X100

Rolled at 2150°F



X100

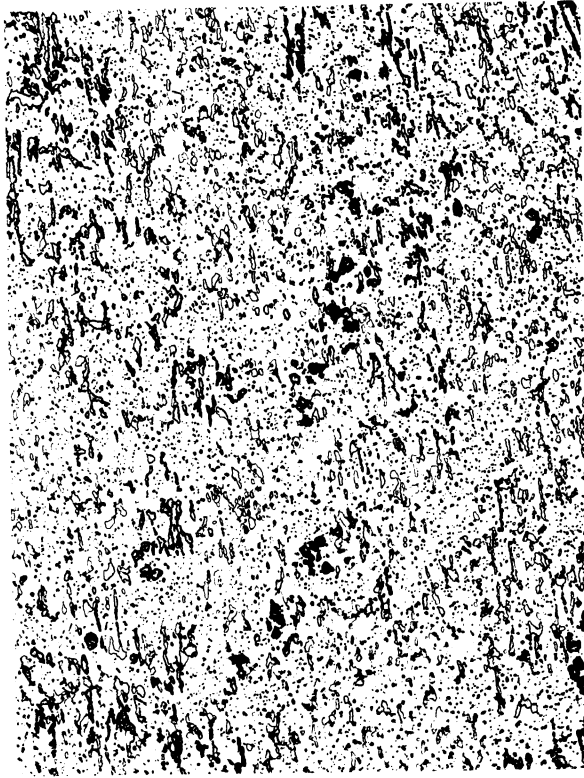
Partially rolled at 2150°F,
heated to 2300°F, finish, rolled
at 2150°F

Figure 18.- Microstructures of Heat 1074 air cooled after the indicated rolling treatment. Rolling direction is vertical.



X100

Figure 19.- Microstructure of Heat UA4 (air melted) in as-rolled condition.
Rolling direction is vertical.



X100

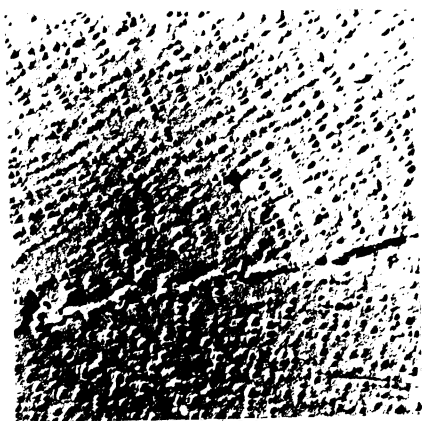
Solution treated 4 hours at
1975°F



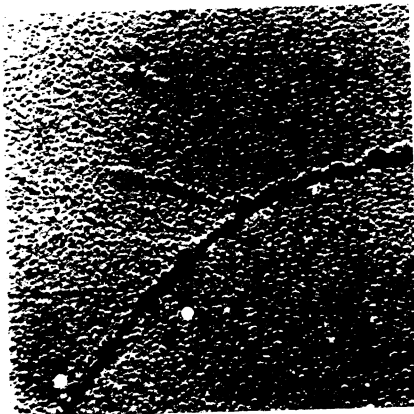
X100

Solution treated 1 hour at
2150°F

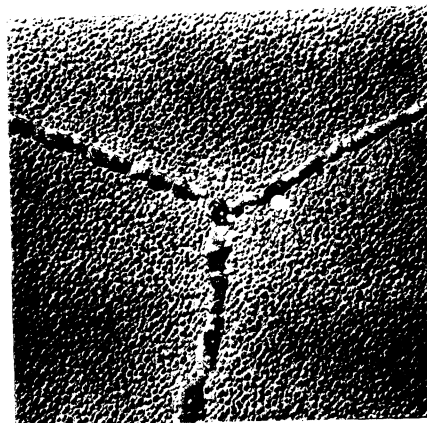
Figure 20.- Microstructure of Heat 1095 (6%Ti-6%Al) after indicated solution treatment and air cool. Rolling direction is vertical.



X9000
Inconel-X

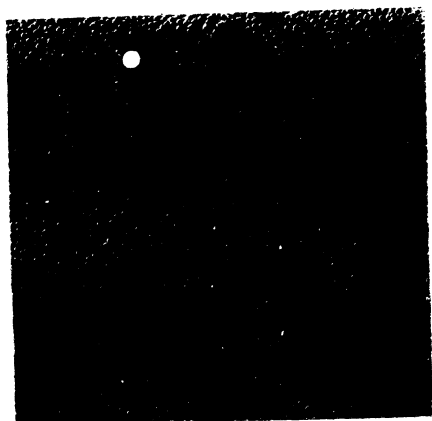


X9000
Waspaloy



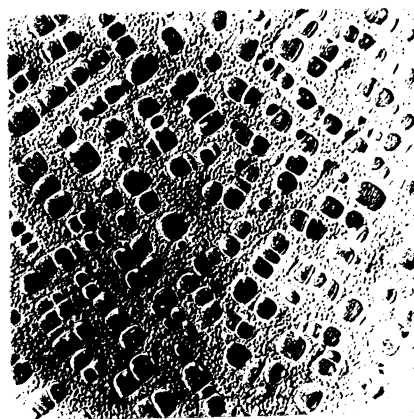
X9000
3Al-3Ti (Heat 1015)

Aged 1000 hrs. at 1200°F 4 hrs. 1975°F, air cool 4 hrs. 1975°F, air cool



X9000
3Al-3Ti (Heat 1015)

4 hrs. 1975°F, air cool



X9000
4Al-4Ti (Heat 1079)

4 hrs. 1975°F, air cool

Figure 21.- Electron micrographs showing progressive change of precipitates in matrix structure of different Ti-Al hardened nickel-base alloys.

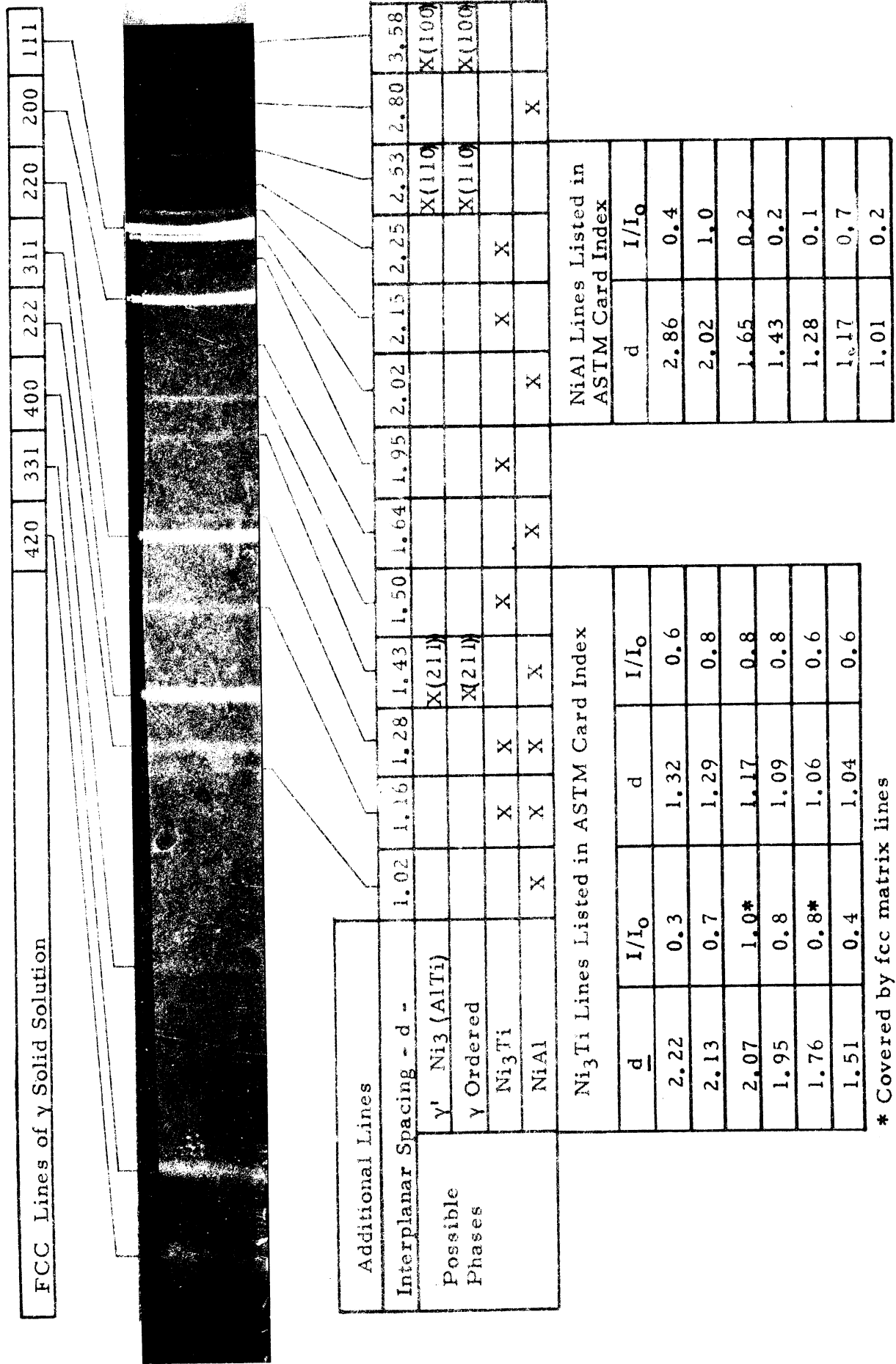


Figure 22. -X-ray diffraction pattern obtained from solid sample of Heat 1095 after solution treatment of 4 hours at 1975°F and air cool. Cu K α radiation. Possible phases are indicated for each diffraction line.

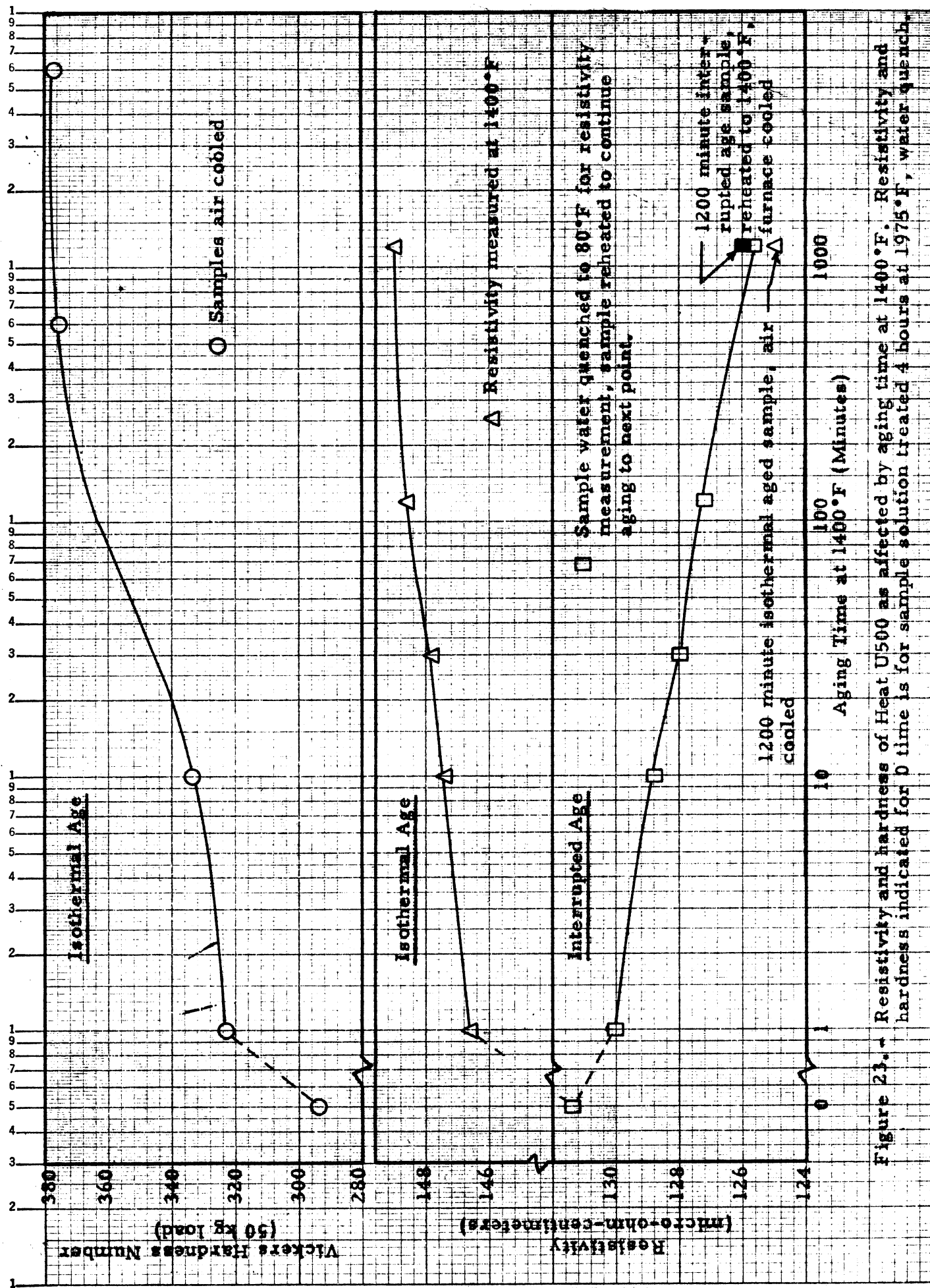


Figure 23. - Resistivity and hardness of Heat U500 as affected by aging time at 1400°F. Resistivity and hardness indicated for 0 time is for sample solution treated 4 hours at 1975°F, water quench.

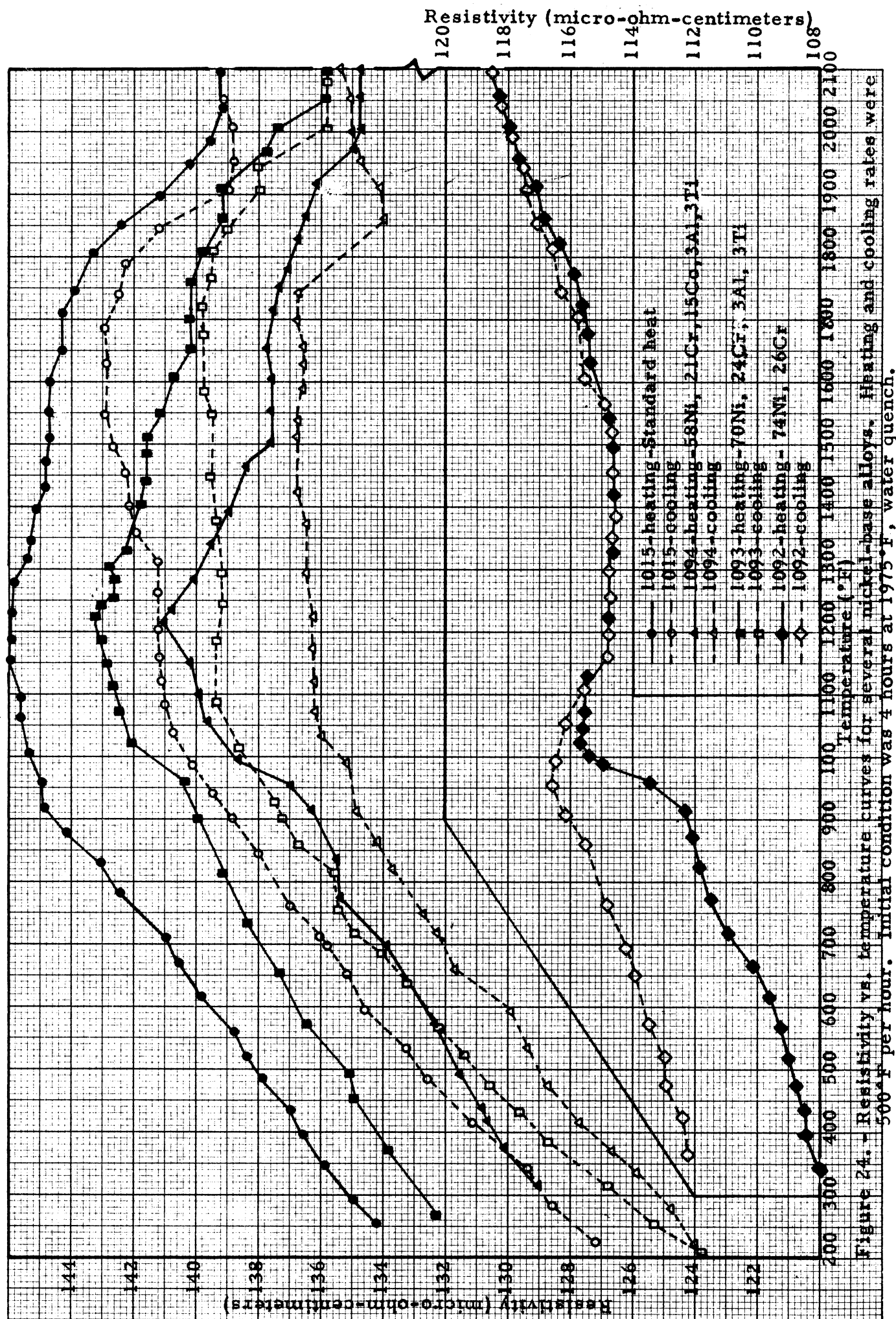


Figure 24. - Resistivity vs. temperature curves for several nickel-base alloys. Heating and cooling rates were 500°F per hour. Initial condition was 4 hours at 1975°F, water quench.

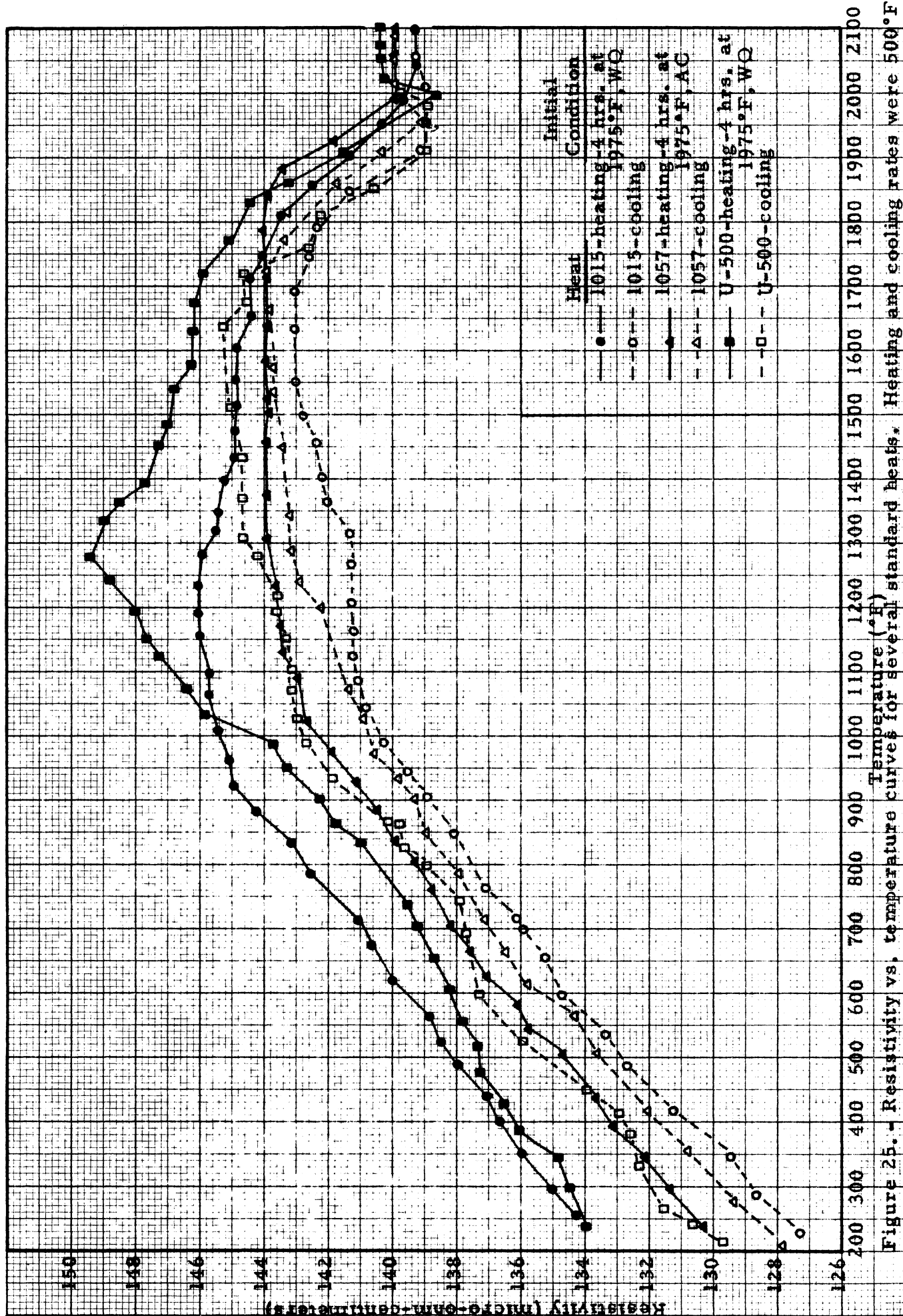


Figure 25. - Resistivity vs. temperature curves for several standard heats. Heating and cooling rates were 500°F per hour.

Accumulated Overheat Time at Indicated Temperature (Minutes)

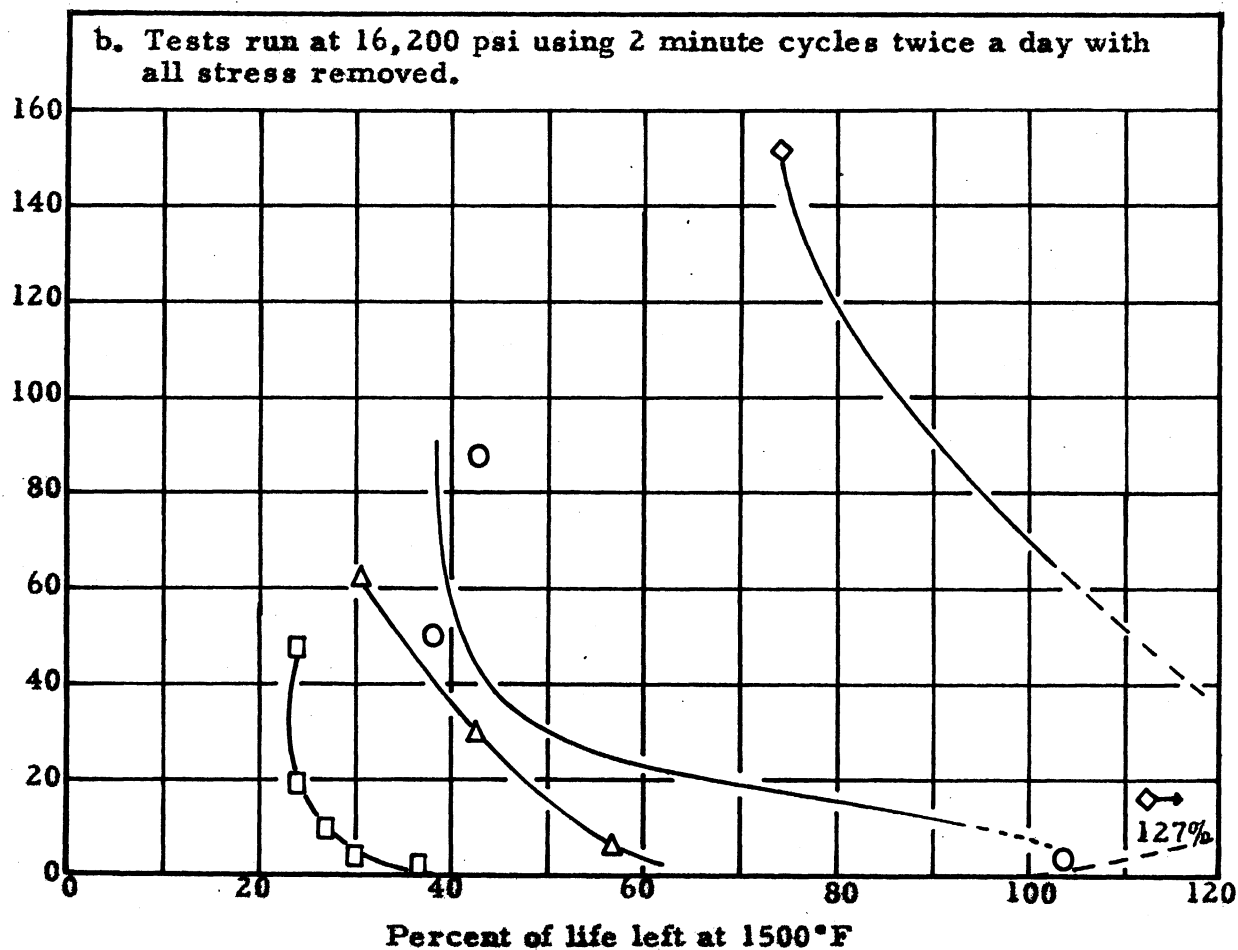
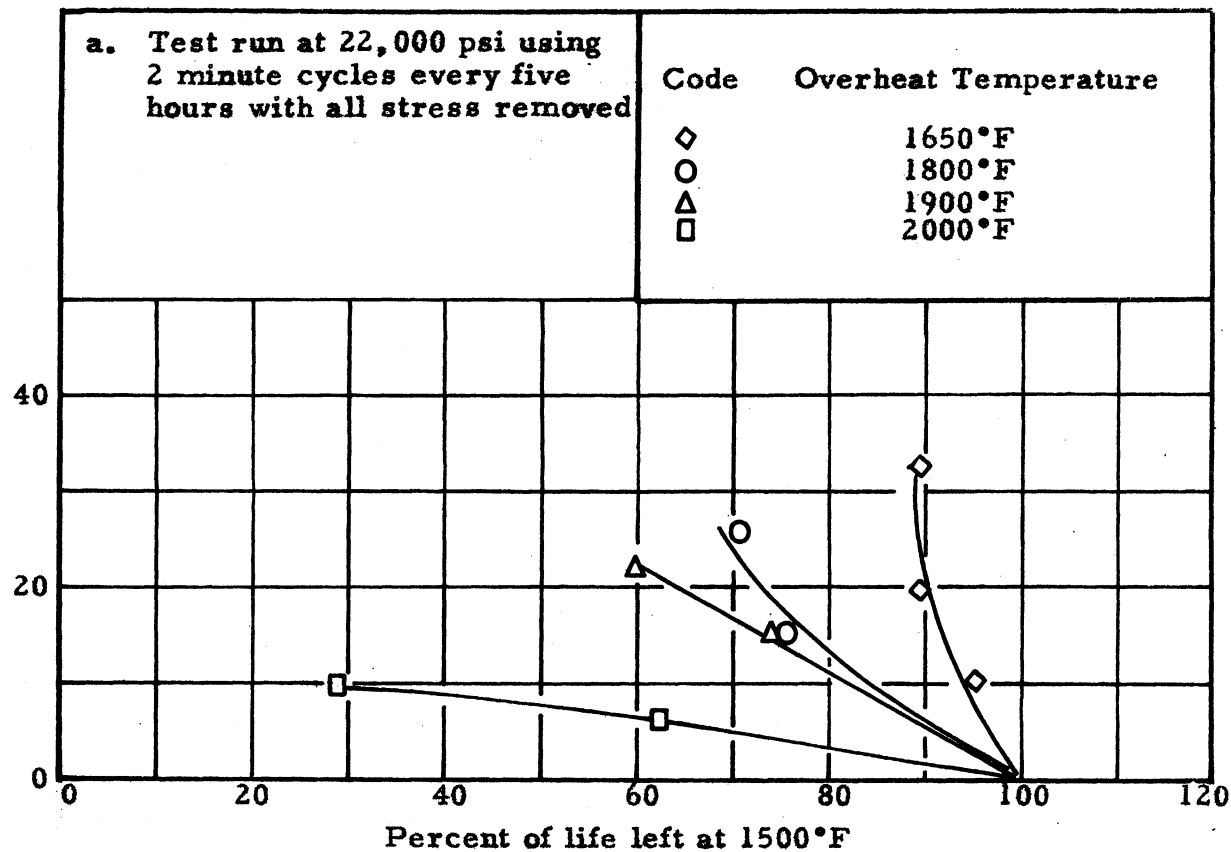
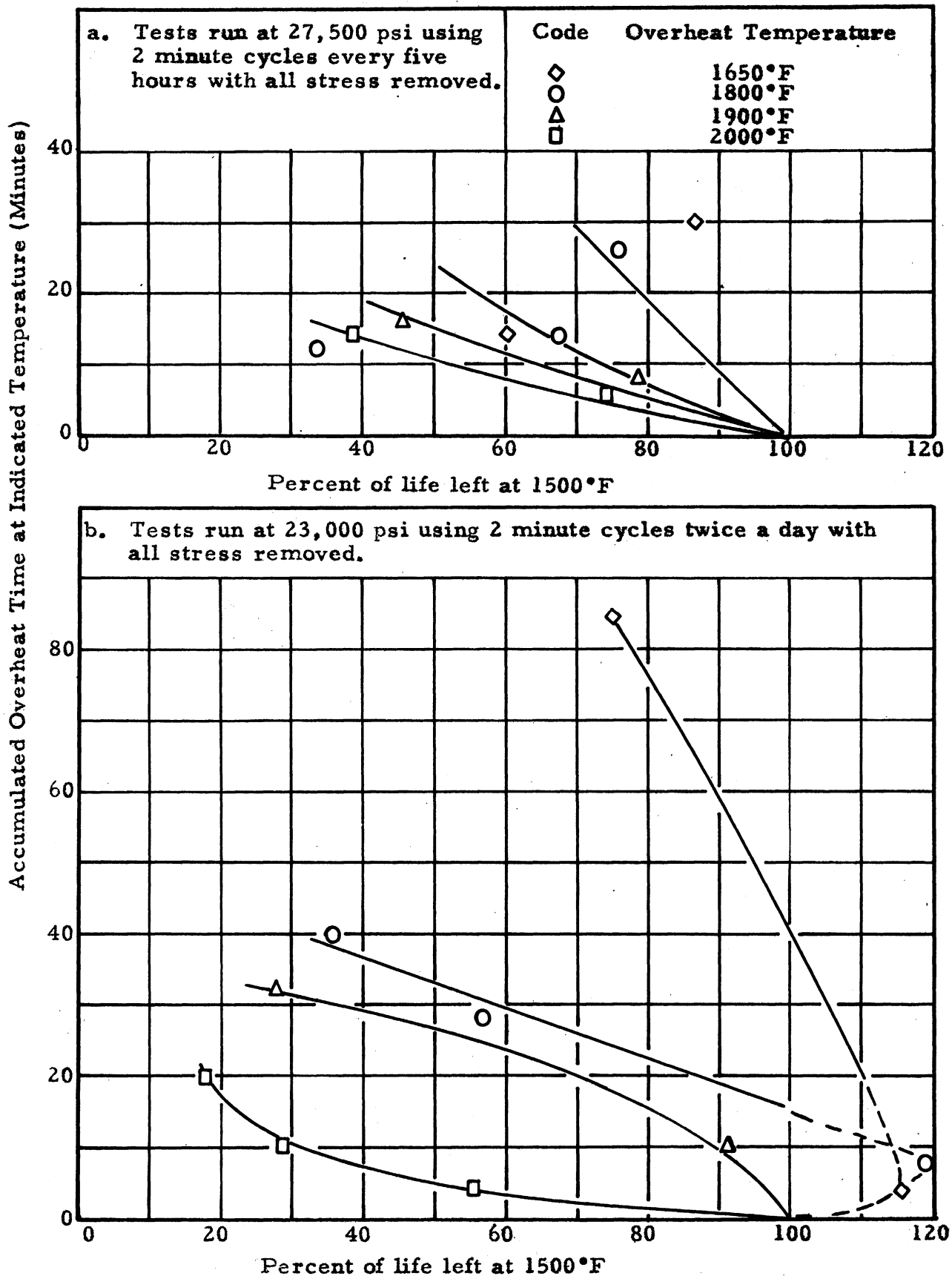


Figure 26. - Effect on S816 of accumulated overheat time in the absence of stress on the percent life left at 1500°F.



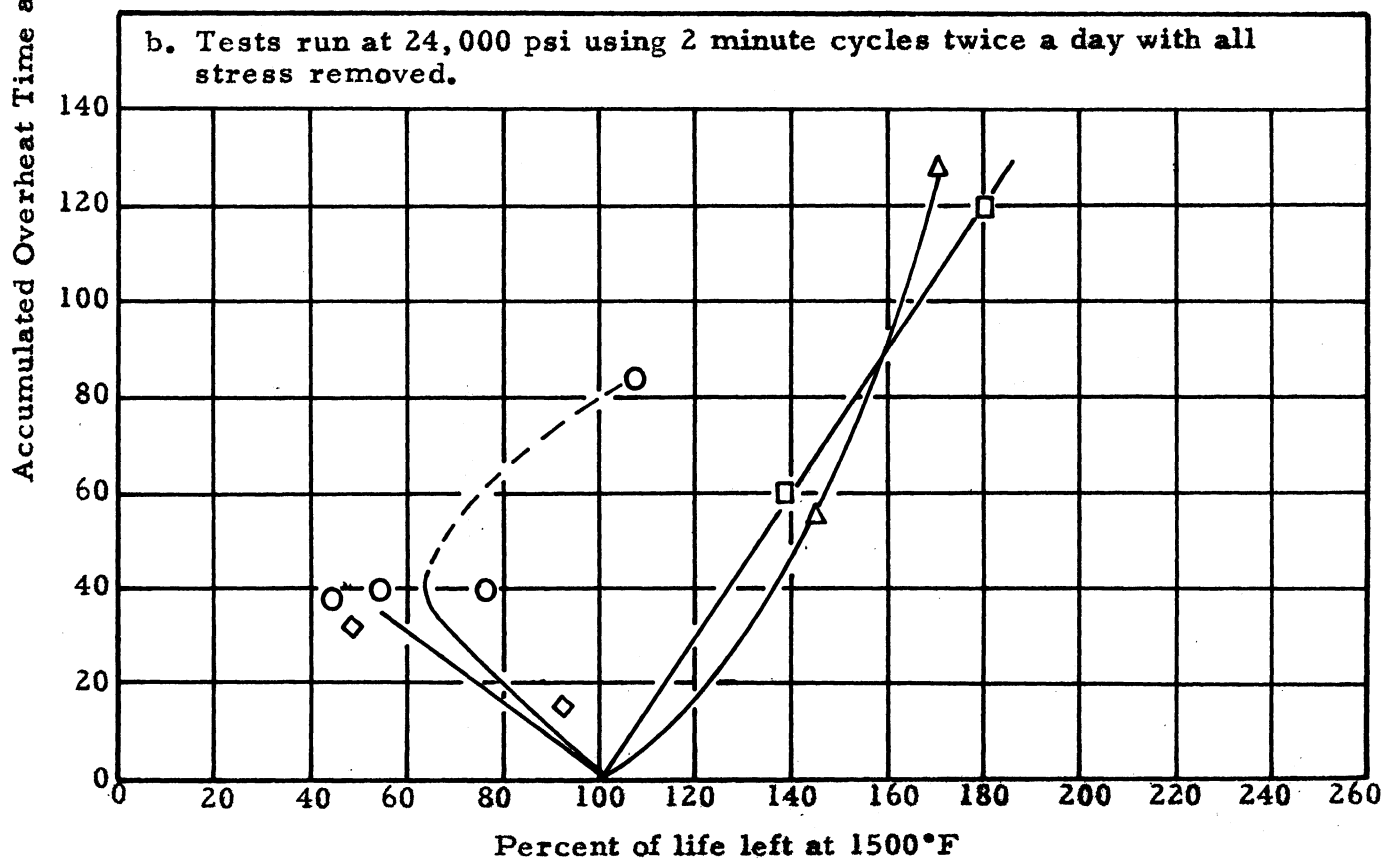
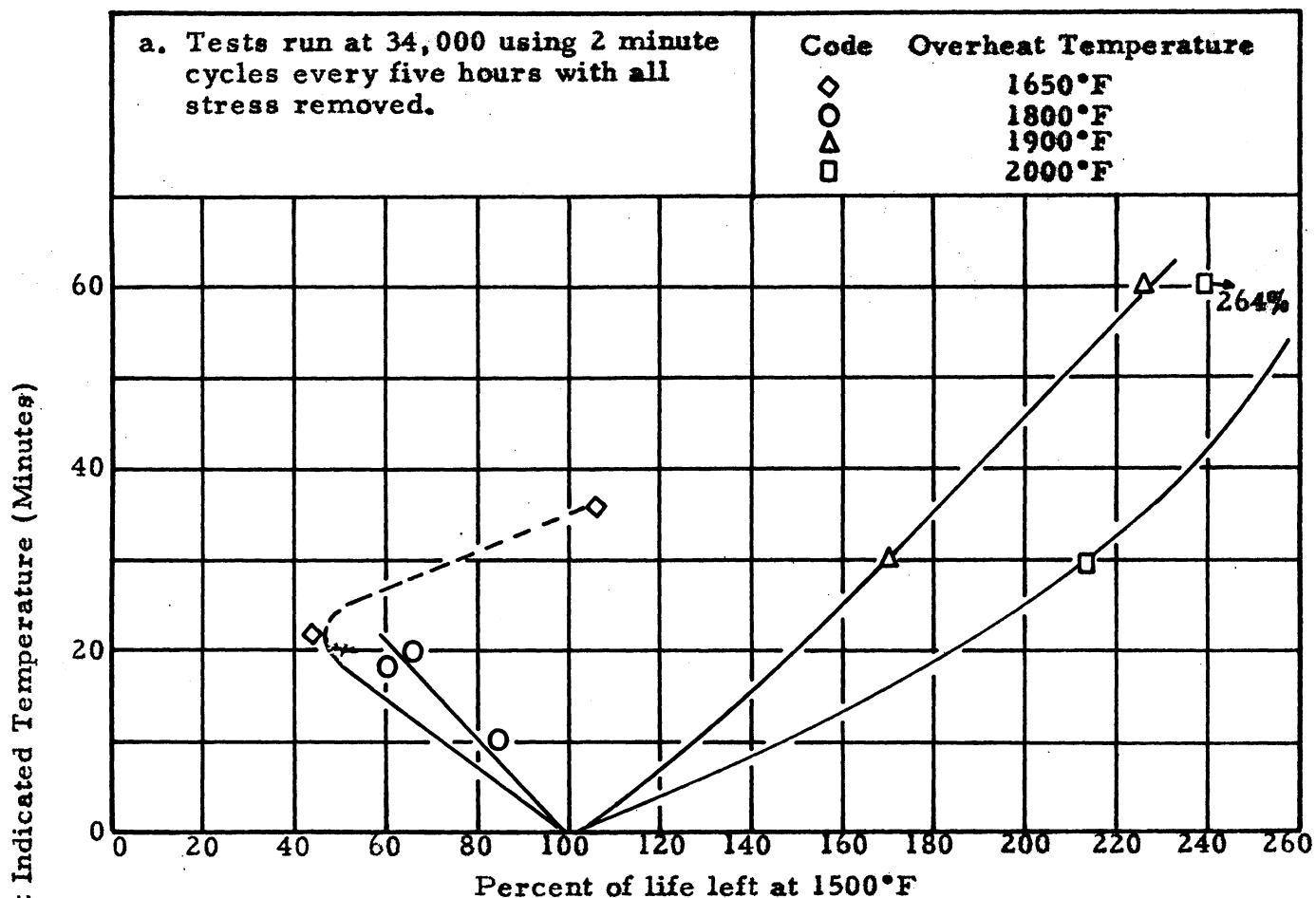


Figure 28. - Effect on M 252 of amount of accumulated overheat time in the absence of stress on the percent life left at 1500°F.

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