ENGINEERING RESEARCH INSTITUTE
THE UNIVERSITY OF MICHIGAN
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Final Report

THE EFFECT OF PROCESSING VARIABLES AND THE INFLUENCE OF VACUUM MELTING UPON THE STRESS-RUPTURE PROPERTIES OF CAST NICKEL-BASE ALLOYS

C. M. Hammond
R. A. Flinn

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SUMMARY

In the work of the previous year (September 1954-September 1955)[1] it was found that melting in vacuum improved the elevated-temperature properties of three prominent nickel-base alloys (Final Report, Contract No. NOas 55-110c). The objectives of this year's research were (1) to investigate additional processing variables such as melting time and pouring temperature and (2) to explore further the changes in properties associated with vacuum melting.

A. EFFECTS OF ADDITIONAL PROCESSING VARIABLES.

The following variables were investigated:

1. Effect of charge preheat before melting.
2. Pouring temperature.
3. Pouring pressure.
4. Increased time of melt in liquid state (nucleation effect).
5. Mold preheat temperature.

The effect of charge preheat was pronounced, resulting in an increase of 100-hour rupture strength (1500°F) from 40,000 to 50,000 psi for the Guy-type alloy. Variables 2-5 inclusive were without effect (95-percent confidence limits).

B. VACUUM-MELTING EFFECTS.

One of the principal differences in vacuum-melted material is low nitrogen. As nitrogen was increased in vacuum melts by late additions, the elongation of the Guy-type alloy was reduced from 7-10 percent to 2 percent. Strength was unaffected.

Analysis of metal vapors during vacuum melting exhibited surprisingly large quantities of undesirable elements such as lead and calcium. Refining time during vacuum melting reduced the elongation of the Guy-type alloy from 7-10 percent to 3-5 percent.

Electron microscopy, electron diffraction, x-ray diffraction, and spectroscopic investigations of vacuum- vs air-melted material were conducted. The phases Cr_7C_3, Fe_2B, and Cb(CN) were identified along with the matrix lines in both melts. Electron microscopy disclosed the presence of a rod-like precipitate present only in air-melted samples, after testing. Also, a grain-boundary precipitate has tentatively been identified only in air-melted microstructures.

INTRODUCTION

In the past few years, many new heat-resistant alloys have been developed for high-temperature applications, such as turbine blades in jet engines. Nickel-base alloys containing major percentages of chromium, molybdenum and aluminum and varying percentages of boron have been prominent in these development programs. Prior to the initiation of this research, the majority of these alloy programs dealt only with variations of chemical composition which were correlated with stress-rupture properties at 1500° F. The effect of processing variables upon elevated-temperature properties of existing nickel-base alloys in order to demonstrate their ultimate potential had received relatively little attention.

It appeared necessary, therefore, to parallel the investigations of compositional effects with research on the effects of processing variables, such as melting and casting atmospheres, pouring temperatures, etc., upon stress-rupture properties of cast nickel-base alloys.

Previous research on this contract[1] determined the effect of melting and casting atmospheres upon the stress-rupture properties of three commercial nickel-base alloys. Table I and the following discussion briefly review that research.

As shown in Table I, vacuum + argon melting increases the elongation of all three alloys and also significantly raises the 100-hour rupture strength of the Guy-type alloy. Metallographic examination with the light microscope revealed no reasons for the above effects. The only positive difference found between vacuum + argon and air or argon-protected heats was nitrogen content.

OBJECTIVE

Based upon the foregoing data, this year's research had two objectives.

First, the remainder of the processing variables, such as melting practice, pouring temperature, pouring pressure, etc., were to be evaluated to determine their individual effects upon stress-rupture properties.

Second, because of the pronounced influence of vacuum melting upon stress-rupture properties, further investigation to explain the mechanism for these effects was needed.

TABLE I

EFFECT OF MELTING AND CASTING ATMOSPHERES
UPON STRESS-RUPTURE PROPERTIES AT 1500° F.

Alloy Type		Published Properties	Research at The University of Michigan — Atmosphere: Air	Argon-Protected	Vacuum + Argon
Guy	100-hour rupture strength (psi)	49,000	42,000	41,000	56,000
	Percent elongation	2-5	3	1-3	7-10
GMR 235	100-hour rupture strength (psi)	37-40,000	41,000	41,000	42,000
	Percent elongation	6-10	6-9	3-7	14-19
Inco 700	100-hour rupture strength (psi)	43,000	37,000	43,000	42,000
	Percent elongation	10	2-4	3-4	21

PROCEDURE

In order to accomplish the objectives of this research, the following experimental program was planned, involving A , processing variables in general, and B, the effects of vacuum melting:

A. Processing Variables

1. Charge preheat time before melting in air and argon-protected atmospheres.
2. Pouring temperature.
3. Pouring pressure.
4. Holding time of the melt in the liquid state after all additions were made.
5. Mold preheat temperature.

The above variables are considered to be the more important processing variables affecting elevated-temperature properties.

B. Vacuum-Melting Effects

1. Nitrogen additions to vacuum + argon melts.
2. Collection and analysis of metal vapors evolved during vacuum + argon melting.
3. Structural studies to determine the effects of vacuum + argon melting upon metal structure.

The nitrogen additions were made to increase the nitrogen of the vacuum + argon heats to the level of air and argon-protected heats. If nitrogen is an important factor in controlling stress-rupture properties, these additions should lower the strength and/or ductility of vacuum + argon melts to that of air and argon-protected heats.

The vapor analyses were collected to determine whether any trace elements, which might be harmful to elevated-temperature properties, were evolved during vacuum melting.

The objective of the structural studies was to compare the microconstituents (size, shape, number, etc.) of air and vacuum + argon heats.

The detailed procedures for carrying out this program are presented below:

A. DETAILED PROCEDURE--PROCESSING VARIABLES

Two particular alloys of comparable chemical analysis, except for boron content, were selected for studying the effects of the processing variables.

The alloys selected were the Guy type (.50 percent B) and the GMR 235 type (.06 percent B). The processing variables , such as pouring temperature, pouring pressure, etc., were varied individually, maintaining the remainder of the variables consistent with those used in determining the effects of melting and casting atmosphere upon stress-rupture properties.[1] With this experimental design a statistical treatment of the data was possible in order to evaluate the individual effects of each processing variable upon stress-rupture properties at 1500° F.

The processing variables studied and the conditions under which they were investigated are listed in Table II.

The melting and casting techniques for the argon-protected GMR-235-type alloy and the vacuum + argon melted Guy-type alloy were the same as those previously described,[1] while the melting of the Guy-type alloy, under air and argon, was slightly different.

For the air and argon-protected Guy-type alloy, the charge was melted slowly at the beginning of the heat to avoid bridging and, consequently, severe overheating of the molten metal. The time required to melt down the heat increased from five minutes for the conventional practice to twenty minutes using this preheat practice.

Since these alloys contained fairly high percentages of easily oxidized elements, such as Al, Ti, etc., a melting practice which occupied the least time from start-up to pour originally seemed to be the best melting practice. However, one heat of the Guy-type alloy with an air melting atmosphere, which was designed to evaluate the effect of the time of vacuum + argon melting practice in air, displayed a 100-hour rupture strength comparable to vacuum + argon properties. This melting procedure was further investigated and it seemed advisable to use this practice for the remainder of the melts involving the effects of processing variables upon the rupture properties of the Guy-type alloy. The effect of the variables outlined above upon the properties of the GMR-235-type alloy had already been collected and up to the present time these experiments have not been conducted using the slower melting practice.

B. DETAILED PROCEDURE--VACUUM-MELTING EFFECTS

Since vacuum + argon melting affected both the high-temperature strength and ductility of the Guy-type alloy, this alloy was selected in order to study the mechanisms for the vacuum effect.

1. Nitrogen Additions.--Nitrogen was added to vacuum + argon heats in the form of high-nitrogen (1.75 percent N) ferrochrome. This was added just prior to pouring, under an argon pressure of approximately 400 mm of mercury. The application of argon pressure insured reasonable nitrogen recovery in the melt.

TABLE II

PROCESSING VARIABLES INVESTIGATED

Variable	Alloy Type Used	Melting Atmosphere	Heat No.(1)	Range of Variable Employed
1. Charge preheat time before melting	Guy	air and argon	R-285 R-218 R-300 R-213 R-284 R-301	Slow melting at beginning of heat (20 minutes to melt charge vs 5 minutes of previous data)
2. Pouring temperature	Guy	argon	R-212 R-213 R-214 R-302	2660 - 2950° F.
	Guy	vacuum	R-223 R-224 R-225	2660 - 3000° F.
	GMR 235	argon	R-150 R-151 R-152	2660 - 2950° F.
3. Pouring pressure	Guy	argon	R-215 R-216 R-217	0 - 12 psig.
	GMR 235	argon	R-153 R-154 R-155	0 - 15 psig.
4. Holding time	Guy	argon	R-219 R-220	1/2 - 2 hours
	GMR 235	argon	R-221 R-222	1/2 - 2 hours
5. Mold preheat temperature	Guy	air and argon	R-278 R-281 R-279 R-280	1800° F.

(1) Full details given in Appendices I-V.

In order to evaluate the effect of a late chromium addition and argon pressure upon the vacuum + argon Guy-type alloy, a control heat was melted in which standard ferrochrome (essentially 0 percent nitrogen) was added prior to pouring and argon pressure of 400 mm applied to the heat.

The nitrogen additions and appropriate heat numbers are listed in Table III.

TABLE III

NITROGEN ADDITIONS TO VACUUM-MELTED HEATS OF THE GUY-TYPE ALLOY

Heat No.	Percent N_2 Added
R-209	0 (control heat)
R-211	.01
R-210	.03
R-287	.10

2. Vapor Collection and Analysis.--Metal vapors evolved during vacuum melting were collected on a thin sheet of 99.90 percent nickel. The sheet was cleaned in acid and washed thoroughly in water before mounting it above the crucible. The vapors were collected throughout the entire heat and were easily flaked off after completion of the melt.

The analyses of these vapors were conducted spectrographically by the J. H. Herron Company in Cleveland, Ohio.

3. Structural Studies.--The structure of the Guy-type alloy was examined using several different metallurgical techniques:

a. Optical microscopy.
b. X-ray diffraction.
c. Spectroscopy.
d. Electron diffraction.
e. Electron microscopy.

The metallographic specimens were again studied by optical microscopy. The inclusion content of alloys melted in vacuum + argon was compared to air and argon-protected heats. The ratings of the inclusion content of the vacuum + argon heats to which nitrogen was added were also included in this survey.

X-ray diffraction patterns were obtained from solid and powder samples of air and vacuum + argon heats, before and after testing. Minor phases, extracted by a bromine-alcohol mixture[2] from the metal samples of air and vacuum + argon heats, before and after testing, were analyzed by x-ray diffraction.

The same extracts mentioned above were analyzed spectrographically to determine whether any compositional variations were present in the minor phases.

Electron diffraction was used to attempt to identify the Chinese-script phase present in all Guy-type alloys. By directing a low-angle beam of electrons upon the Chinese-script phase, which remains in relief after polishing and etching, positive identification is possible from the diffraction pattern.

Examination of the precipitates and other phases present in the structure and unresolved by optical microscopy was initiated with electron microscopy. The metal surface was prepared in the conventional manner and standard practices, developed by Bigelow and associates,[2] for obtaining shadowed plastic replicas were used.

The samples examined consisted of vacuum + argon and air-melted heats.

DATA AND DISCUSSION OF RESULTS

The presentation of the results of this research is most conveniently discussed under two major headings:

A. The effect of processing variables upon stress-rupture properties.
B. The mechanism of the influence of vacuum melting upon stress-rupture properties.

A. THE EFFECT OF PROCESSING VARIABLES UPON STRESS-RUPTURE PROPERTIES

To evaluate the significance of the effect of the processing variables investigated upon stress-rupture properties, two methods of statistical analyses can be used:

1. 95-percent confidence limits for stress-rupture curves.
2. 95-percent confidence limits for individual rupture tests.

1. <u>95-Percent Confidence Limits for Stress-Rupture Curves.</u>--If a sufficient number of rupture tests are conducted so that a representative stress-rupture curve can be drawn through the data, the first method of statistical analysis can be employed. Essentially, this analysis consists of determining whether the stress-rupture curve for the variable investigated belongs to the same universe as the data to which it is compared. If the rupture curve for the variable studied falls outside the 95-percent confidence

limits of previously established data, the variable has a significant effect upon stress-rupture properties.

2. 95-Percent Confidence Limits for Individual Rupture Tests.--If too few rupture tests are conducted to calculate a representative stress-rupture curve for the particular variable, the second method of statistical analysis must be used. In this case, an individual rupture test is compared to the 95-percent confidence limits for individuals of previously established data. If an individual rupture test for a variable falls outside the confidence limits of the previously established data, the variable has a significant effect upon stress-rupture properties. Otherwise, the effect is not significant.

In this section the results of each processing variable will be discussed separately.

a. Charge Preheat Time.

The effect of charge preheat time upon stress-rupture properties at 1500° F. of the Guy-type alloy, melted under air and argon, is shown in Fig. 1. In order to determine the significance of charge preheat time with regard to stress-rupture properties, both types of statistical analyses were used.

Since the rupture curve for the 20-minute charge preheat time falls outside the 95-percent confidence limits for a preheat time of five minutes, charge preheat time is a significant processing variable. Also, 50 percent of the individual tests fall outside the 95-percent confidence limits for individual rupture tests when only one would show a significant effect. Therefore, it is evident that preheat time is a significant processing variable and considerably affects 100-hour rupture strength (Table IV).

TABLE IV

EFFECT OF CHARGE PREHEAT TIME UPON 100-HOUR RUPTURE STRENGTH OF THE GUY-TYPE ALLOY

Time	Atm	100-Hour Rupture Strength (psi)	Percent Elongation
5	air, argon	40,000	1 - 3
20	air, argon	50,000	2 - 4
20	vacuum	56,000	7 - 10

	C	Cr	Mo	Al	Fe	Cb	B
Compositions of R218 (chrg. for all other heats the same)	.11	11.78	5.80	6.22	4.65	1.78	.43
Analysis range for previous Guy-alloy data	.08-.26	12.83-15.35	4.42-5.87	4.33-7.31	4.36-6.94	1.52-2.30	.28-.48

Heat No.	Atm	Symbol	Heat No.	Atm	Symbol
R285	air	○	R213	argon	●
R218	"	○	R284	"	●
R300	"	○	R301	"	●

Pouring temp. for all heats - 2800°F
Pouring press. for all heats - 5 psig.

Elongation given near point representing rupture test.

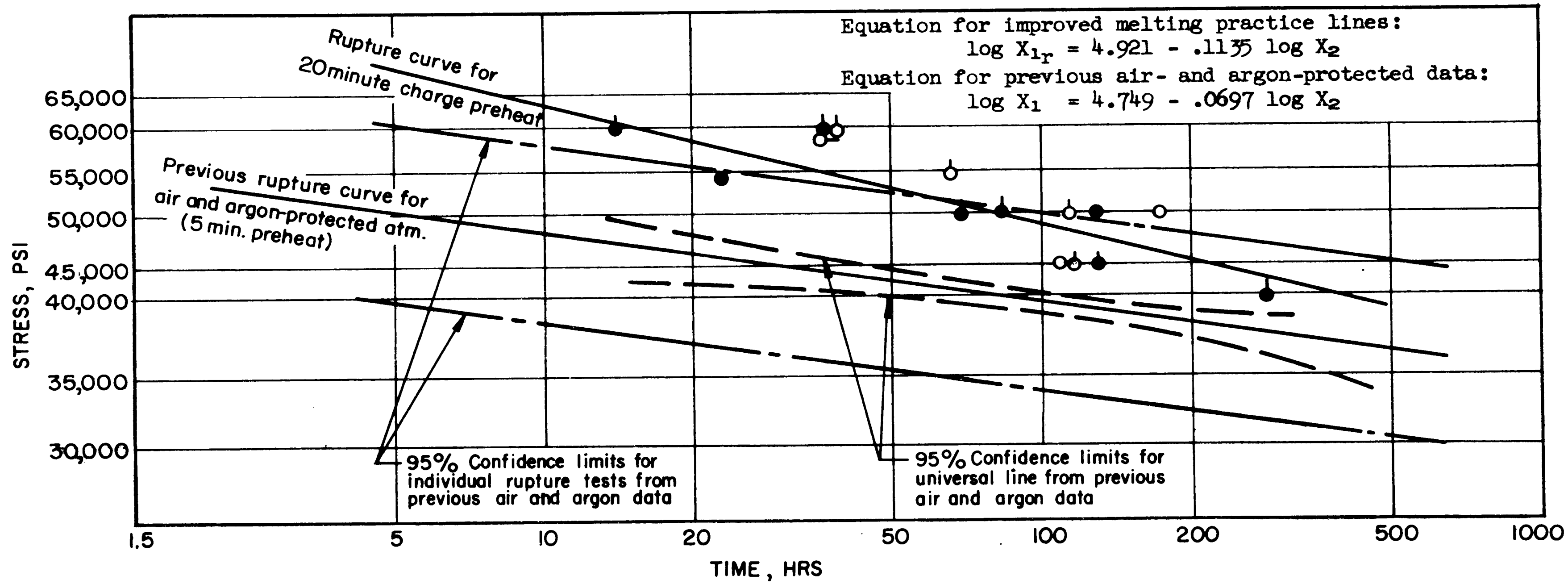


Fig. 1. Effect of charge preheat time upon the stress-rupture properties of air- and argon-protected Guy-type alloys.

It is also of interest to note that the 100-hour rupture strength is nearly comparable to the vacuum + argon melted Guy alloy. The charge preheat time, however, has no significant effect upon elongation.

The influence of this variable upon rupture strength is undoubtedly due to the elimination of severe overheating of the heat during melting.

b. Pouring Temperature.

The Guy-type heats, melted under argon and vacuum + argon atmospheres, were poured at temperatures of 2660°, 2770°, and 2950° F. to evaluate the effect of pouring temperature upon stress-rupture properties. Similarly, the GMR-235-type heats, melted under argon, were poured at 2660°, 2760°, and 2950° F. The stress-rupture results from these experiments are shown in Figs. 2, 3, and 4.

To evaluate the influence of pouring temperature upon rupture properties of the two alloys, the second method of statistical analysis was used. In Figs. 2, 3, and 4 all the rupture tests fall within the appropriate confidence limits for individual tests. Therefore, it can be concluded that pouring temperatures, over the range investigated, do not significantly affect the 100-hour rupture strength of argon-protected and vacuum + argon melted Guy-type alloys or the GMR-235-type alloy melted under argon.

Also, pouring temperature gave no significant change in 100-hour elongation for the Guy alloy. The 100-hour elongation of the GMR 235 alloy increased, compared with the values previously reported[1] (6-12 percent vs 3-7 percent). The effect seemed to be general for all GMR 235 heats regardless of the processing variable investigated. This increase in elongation, however, is possibly due to the lower molybdenum content of the present heats (3.0 percent) compared to higher contents (approx. 5.0 percent) published previously.[1]

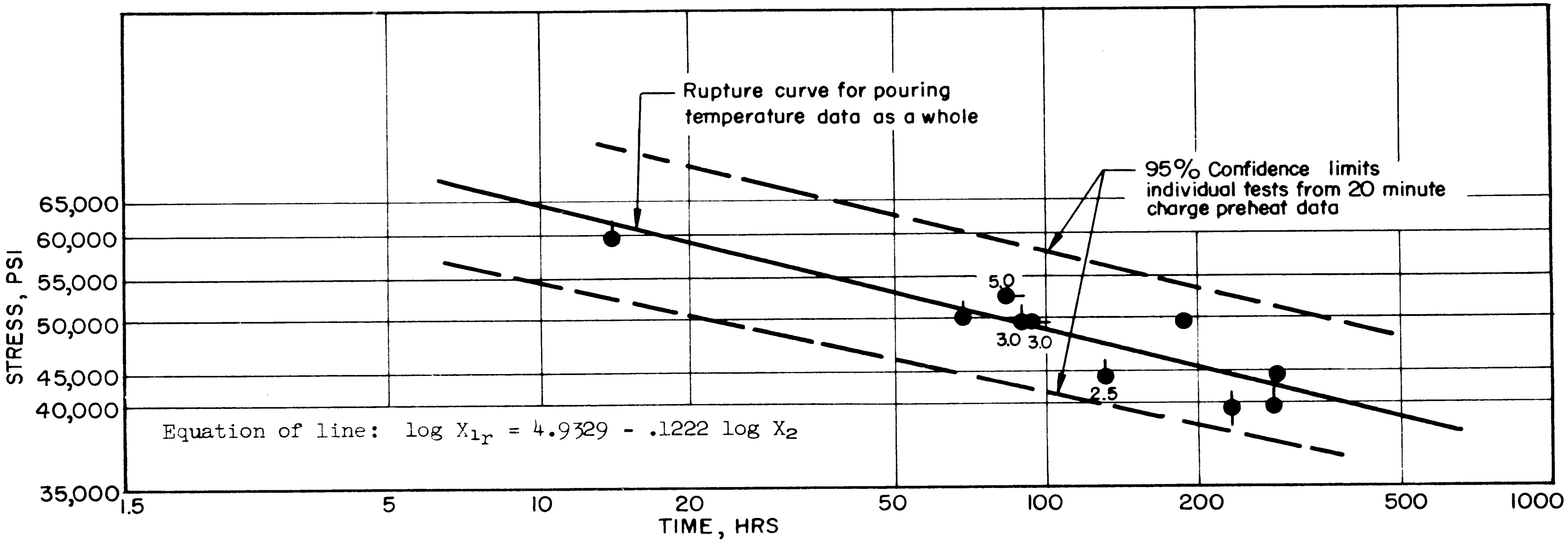
c. Pouring Pressure.

The Guy-type and GMR-235-type alloys melted under argon were poured at pressures of 0, 8, and 12 psig and 1.25, 10, and 15 psig, respectively. The stress-rupture results are shown in Figs. 5 and 6.

The second statistical method was used to evaluate the effect of pouring pressure. Since the individual rupture tests for all pressures investigated were within the appropriate 95-percent confidence limits for individual tests, pouring pressures, over the range investigated, did not significantly affect 100-hour rupture strength of the Guy- or GMR-235-type alloys melted under argon.

The 100-hour elongation of either alloy was not affected by pouring pressure.

	C	Cr	Mo	Al	Fe	Cb	B
Composition of heat R212 (chrg. of heat R213, R214, and R302 same as R212)	.10	12.86	5.74	6.24	4.94	1.54	.38
Typical composition of data used to calc. 95% confidence limits	.11	11.78	5.80	6.22	4.65	1.78	.43



Heat No.	Atm	Symbol	Pouring Temp.(°F)	Superheat (°F)
R212	argon	●	2950	440
R213	"	●	2770	250
R214	"	●–	2660	150
R302	"	● (with vertical tick)	2660	150

Pouring pressure - 5 psig for all heats

20-minute charge preheat practice used in these heats.

Fig. 2. Effect of pouring temperature upon the stress-rupture properties of argon-protected Guy-type alloys.

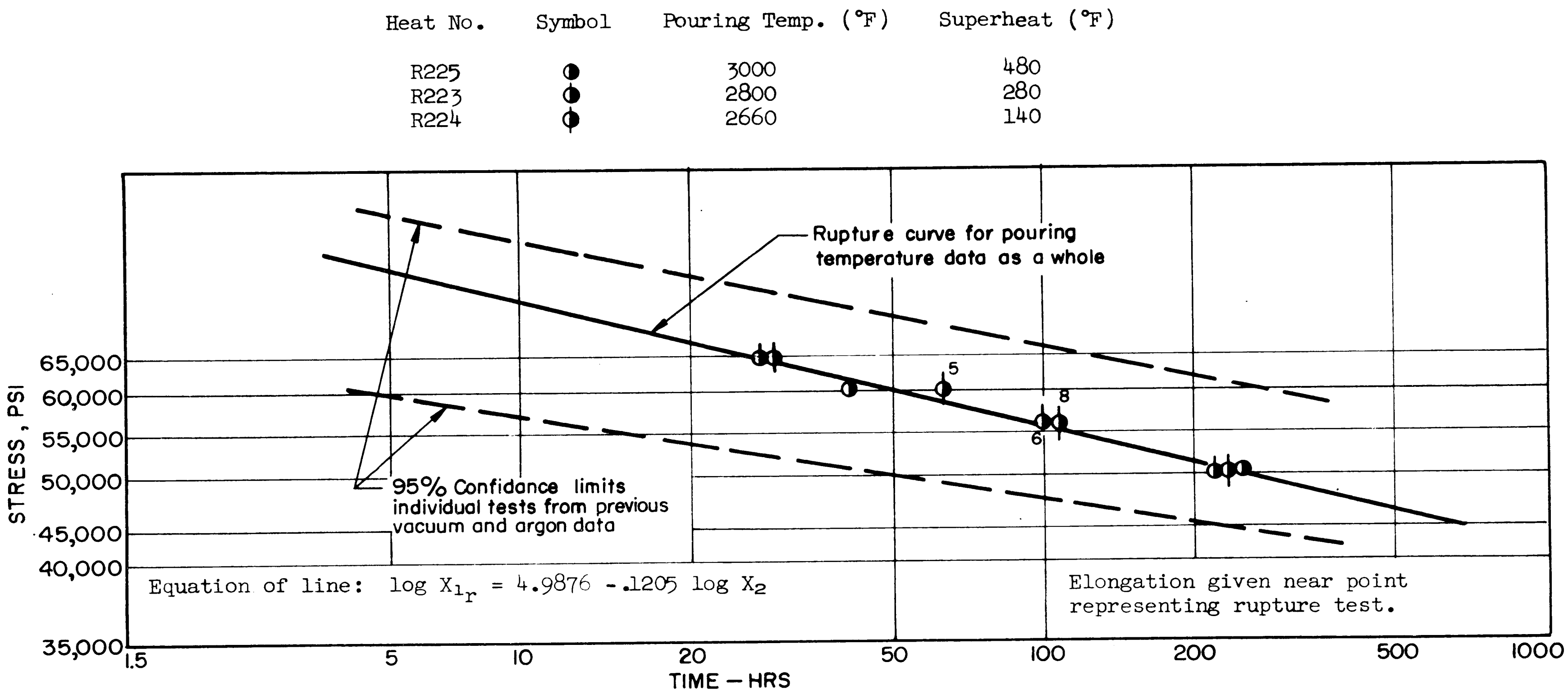


Fig. 3. Effect of pouring temperature upon the stress-rupture properties of vacuum + argon Guy-type alloys.

	C	Cr	Mo	Al	Fe	Ti	B
Composition of heat R150 (chrg. of R151 and R152 same as R150)	.14	14.24	2.85	3.03	9.13	1.56	.06
Analysis range for previous GMR-235-Type heats.	.15-.27	15.03-15.99	4.31-5.39	2.37-4.66	7.65-11.10	1.90-2.30	.06-.12

Heat No.	Atm	Symbol	Pouring Temp (°F)	Superheat (°F)	Pouring Press.
R150	argon	●	2950	440	5 psig
R151	"	●	2760	250	"
R152	"	●	2660	150	"

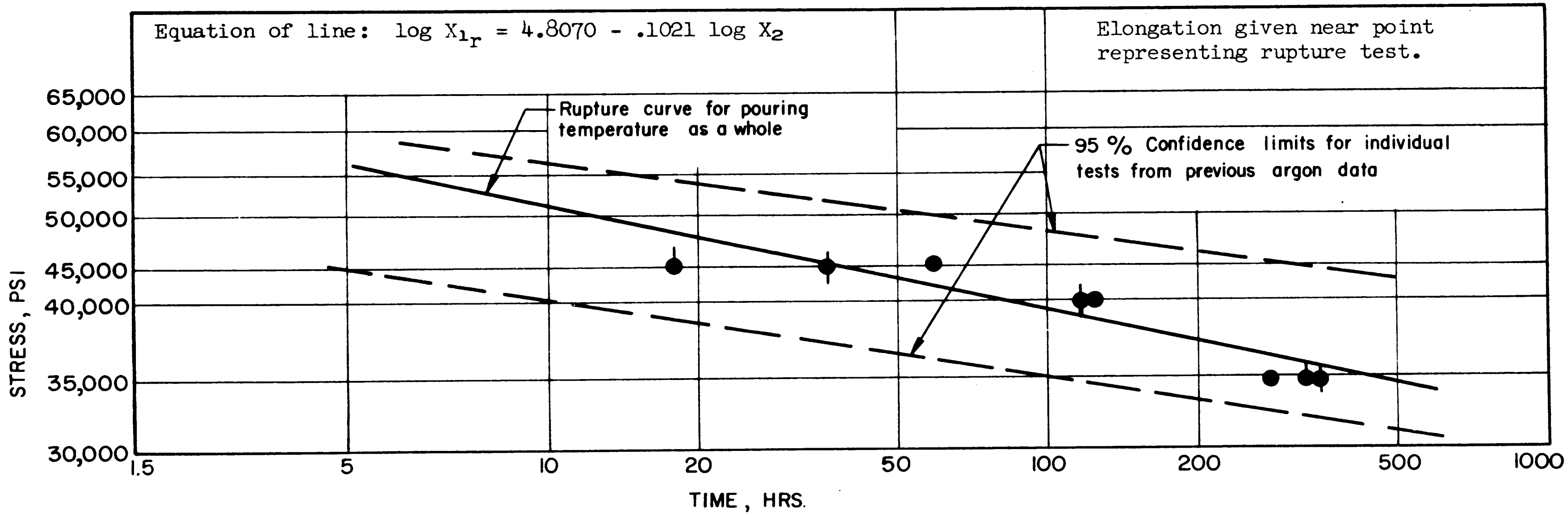


Fig. 4. Effect of pouring temperature upon the stress-rupture properties of argon-protected GMR-235-type alloys.

	C	Cr	Mo	Al	Fe	Cb	B
Composition of Heat R216 (chrg. for R215 and R217 same as R216)	.10	12.24	6.0	6.19	4.75	1.76	.41
Typical composition of data used to calculate 95% confidence limits	.11	11.78	5.80	6.22	4.65	1.78	.43

Heat No.	Atm	Symbol	Pouring Press.(psig)	Pouring Temp.(°F)
R215	argon	●	8	2800
R216	"	● (with vertical bar)	0	"
R217	"	● (with cross)	12	"

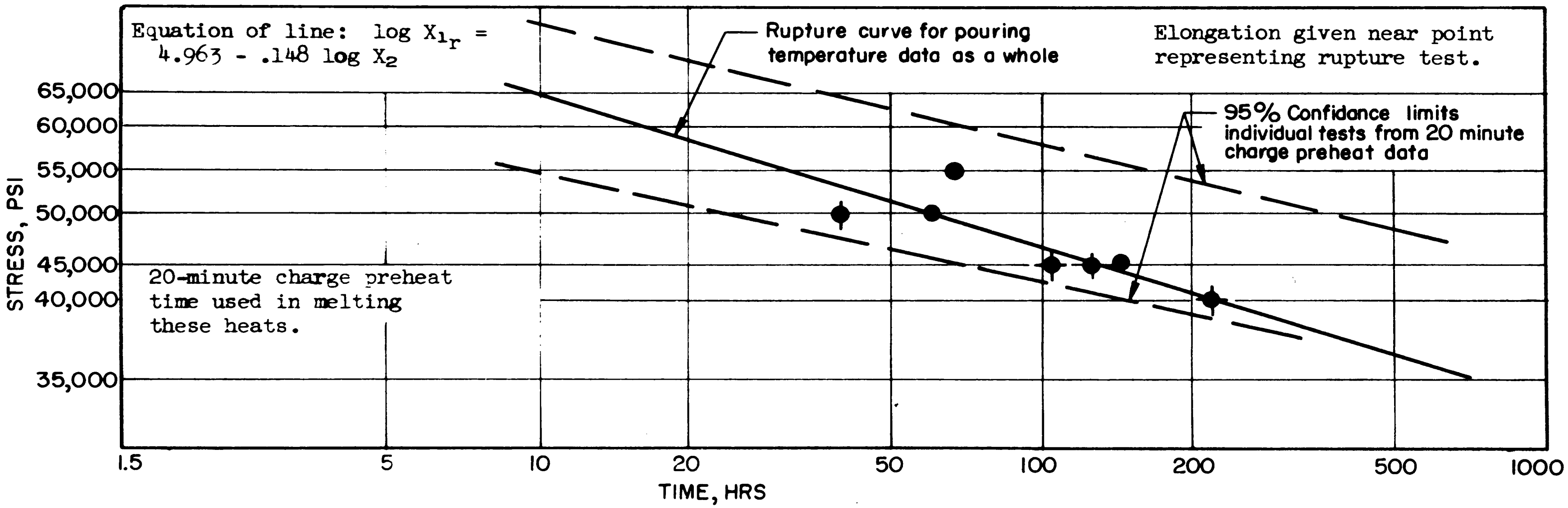


Fig. 5. Effect of pouring pressure upon the stress-rupture properties of argon-protected Guy-type alloys.

Composition: Charge for heats R153, R154, and R155 same as R150 shown in Fig. 4.

Elongation given near point **representing** rupture test.

Equation of line: $\log X_{1r} = 4.8053 - .1004 \log X_2$

Heat No.	Atm	Symbol	Pouring Press. (psig)	Pouring Temp. (°F)
R153	argon	●	10	2850
R154	"	●	15	2850
R155	"	●	0	2850

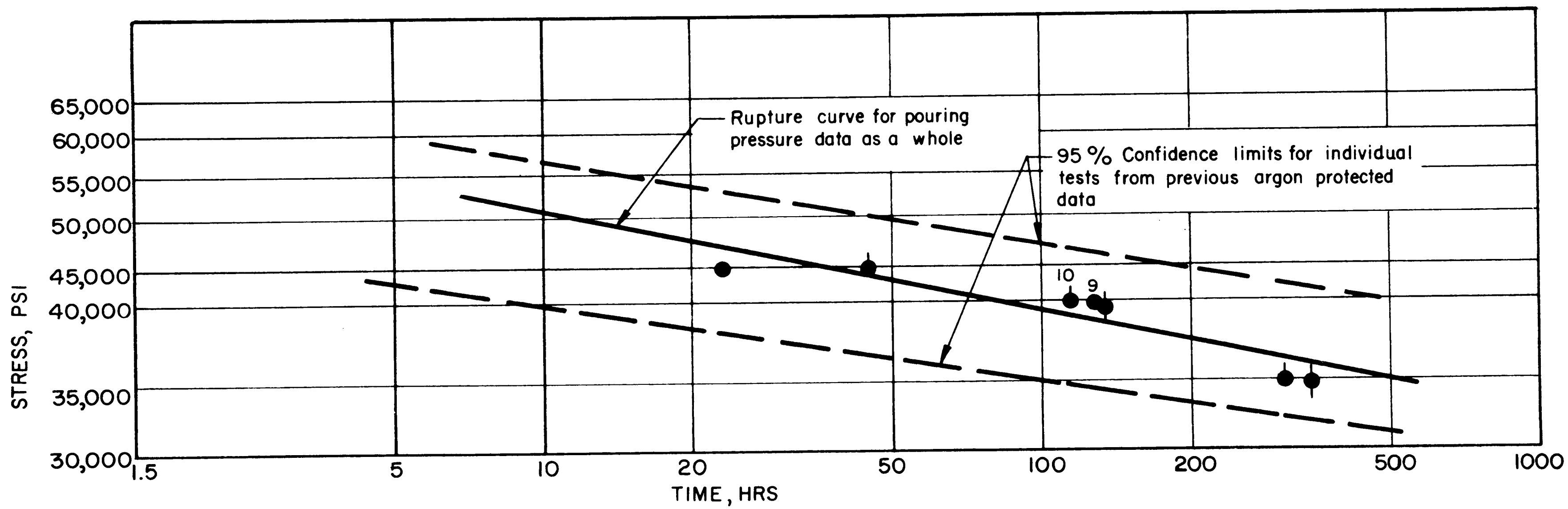


Fig. 6. Effect of pouring pressures upon the stress-rupture properties of argon-protected GMR-235-type alloys.

d. Holding Time.

The Guy-type and GMR-235-type alloys, melted under argon, were maintained in the liquid state for one-half and two hours after all additions had been made. The stress-rupture data are shown in Figs. 7 and 8.

The second statistical method was used to evaluate the data. All the individual rupture tests, for the holding times investigated, were within the appropriate 95-percent confidence limits for individual tests. Therefore, holding time does not significantly affect 100-hour rupture strength. Also, no effect upon 100-hour elongation was observed.

e. Mold Preheat Temperature

The Guy-type alloy, melted under air and argon, was poured into molds preheated at 1800° F. instead of the usual 1600° F. preheat temperature. The stress-rupture data are included in Fig. 9.

The second method of statistical analysis was employed to evaluate the data. All the individual rupture tests fell within the appropriate 95-percent confidence limits for individual tests. Therefore, mold preheat temperature, varied from 1600° to 1800° F., does not significantly affect 100-hour rupture strength of the Guy-type alloy. Also, no effect upon 100-hour elongation is evident from the data.

To summarize the effects of processing variables upon stress-rupture properties of the two alloys investigated, the following statements can be made.

1. The charge preheat time significantly affected the 100-hour rupture strength of the Guy-type alloy.

2. The remainder of the variables did not significantly affect the rupture strength of either alloy.

3. None of the processing variables affected the 100-hour elongation of the Guy- or GMR-235-type alloys.

4. In general, the 100-hour elongation of the GMR-235-type alloy increased, compared with the values previously reported (6-12 percent vs 3-7 percent).

The experimental data for all the variables investigated are included in Appendices I-V.

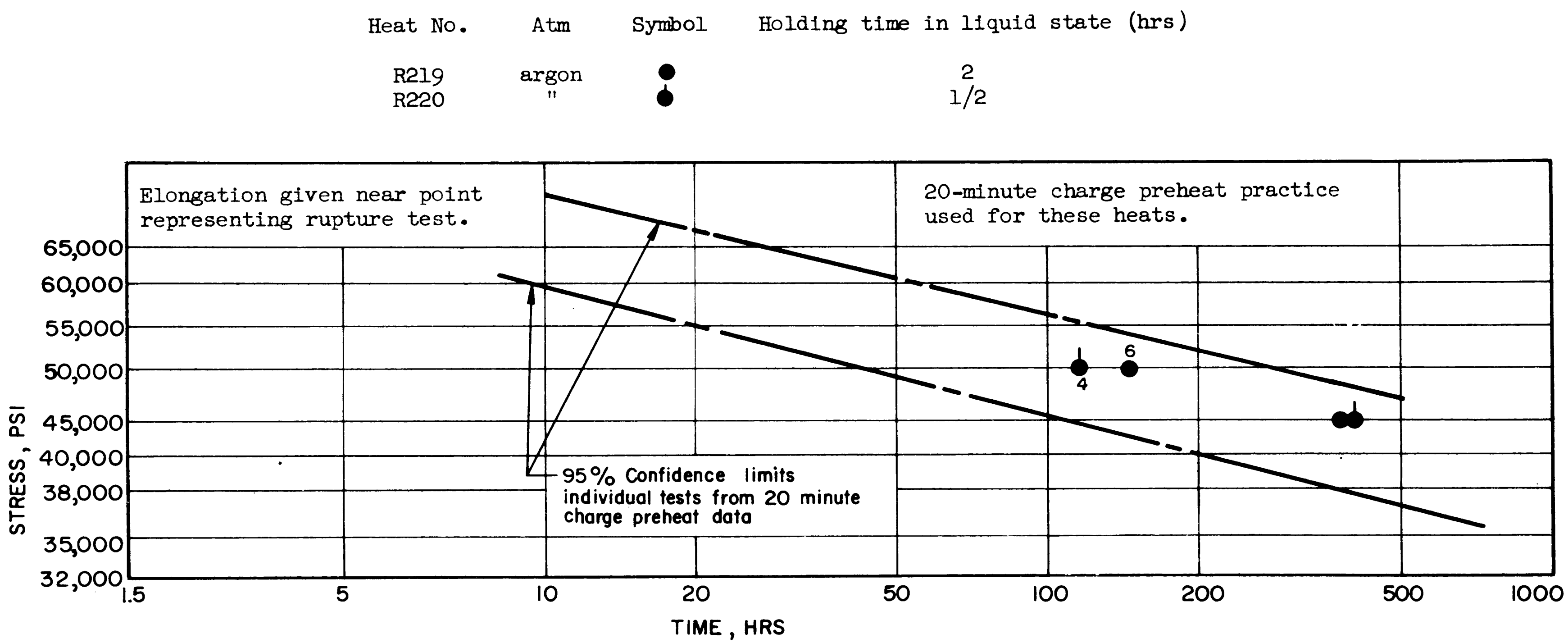


Fig. 7. Effect of holding time in the liquid state upon the stress-rupture properties of argon-protected Guy-type alloys.

	C	Cr	Mo	Al	Fe	Ti	B
Composition of Heat R221 (Same chrg. as R222)	.16	14.86	5.67	3.50	10.38	1.86	.06
Analysis Range for previous GMR-235 data	.15-.27	15.03-15.99	4.31-5.39	2.37-4.66	7.65-11.10	1.90-2.30	.06-.12

Pouring temp. 2850°F—Pouring press. 5 psig

Heat No.	Atm	Symbol	Holding time in liquid state (hrs)
R221	argon	●	2
R222	"	●	1/2

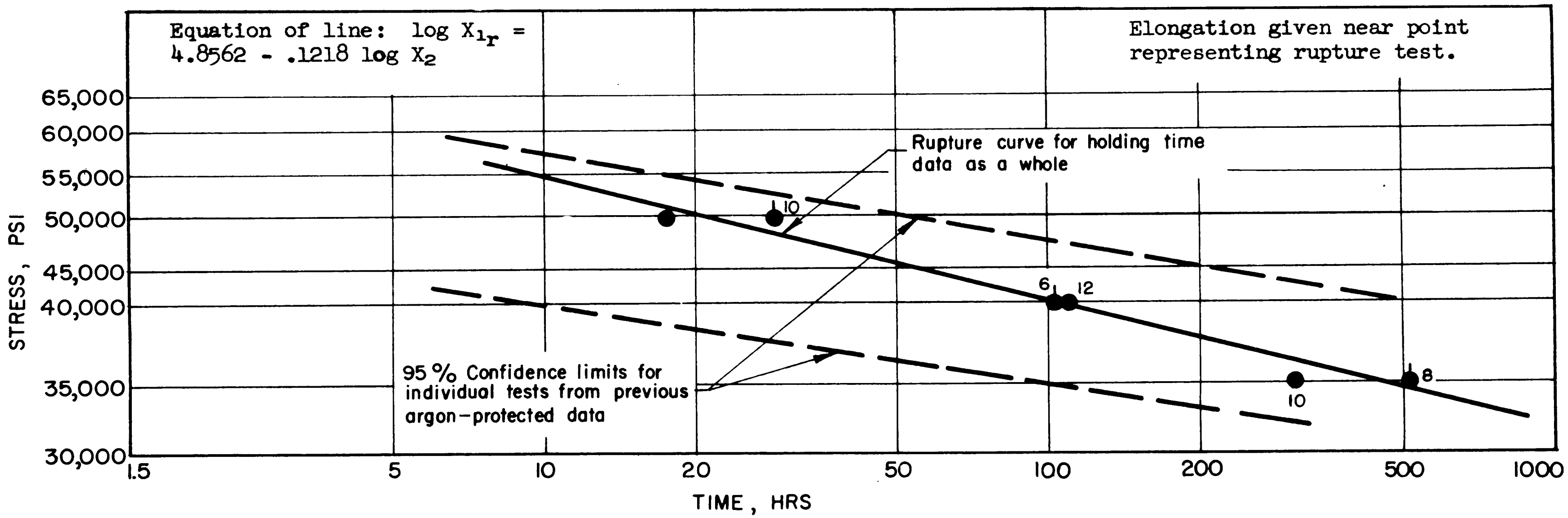


Fig. 8. Effect of holding time in the liquid state upon the stress-rupture properties of argon-protected GMR-235-type alloys.

Heat No.	Atm	Symbol	Mold Temp.
R278	air	○	1800
R281	"	○	"
R279	argon	●	"
R280	"	●	"

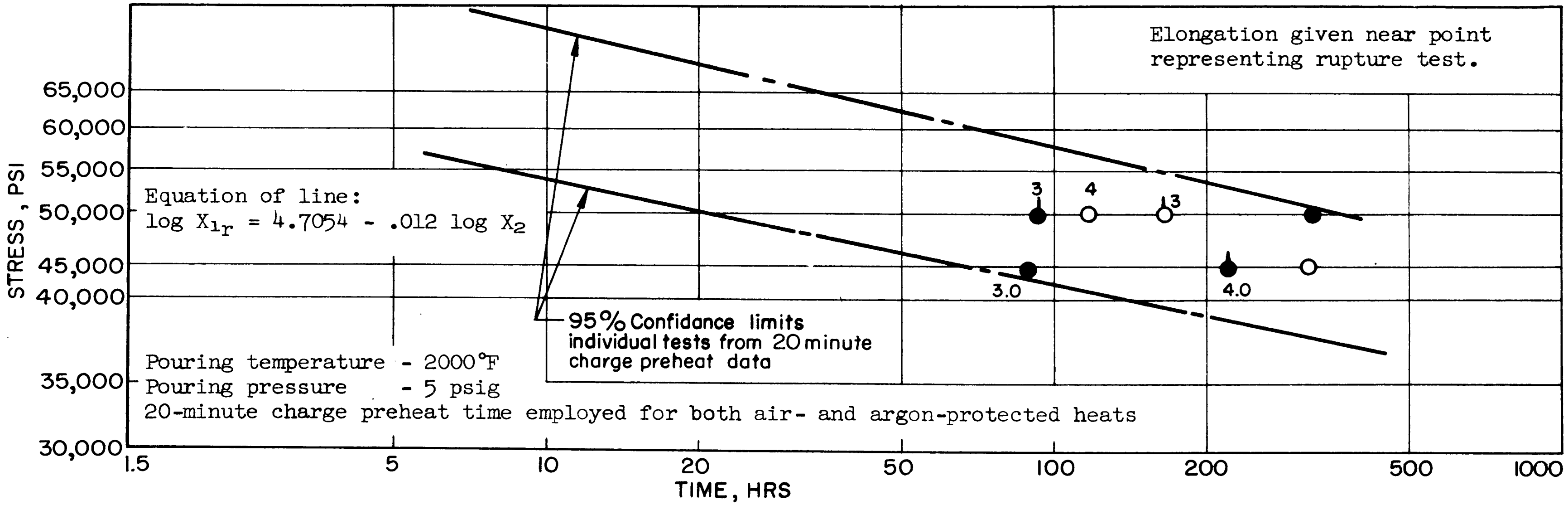


Fig. 9. Effect of mold preheat temperature upon the stress-rupture properties of air- and argon-protected Guy-type alloys.

B. THE MECHANISM OF THE INFLUENCE OF VACUUM MELTING UPON STRESS-RUPTURE PROPERTIES

The results of this segment of the research are conveniently discussed under the following headings:

1. Nitrogen additions to vacuum + argon melts.
2. Vapor collection and analysis.
3. Structural studies.

The Guy-type alloy was selected for the above analysis because of the effect of vacuum + argon melting upon 100-hour strength and ductility. The stress-rupture properties were obtained at 1500° F.

1. Nitrogen Additions to Vacuum + Argon Melts.--Nitrogen reduces ductility and does not significantly affect the high-temperature strength of the Guy-type alloy (Table V and Fig. 10).

TABLE V

THE EFFECT OF NITROGEN CONTENT OF THE GUY ALLOY UPON 100-HOUR ELONGATION

Percent Nitrogen Added	Percent Nitrogen Analyzed	Percent Elongation (100 Hour)
0	.01	6 - 7
.01	.02	5 - 6
.03	.03	4 - 4.5
.10	---	2

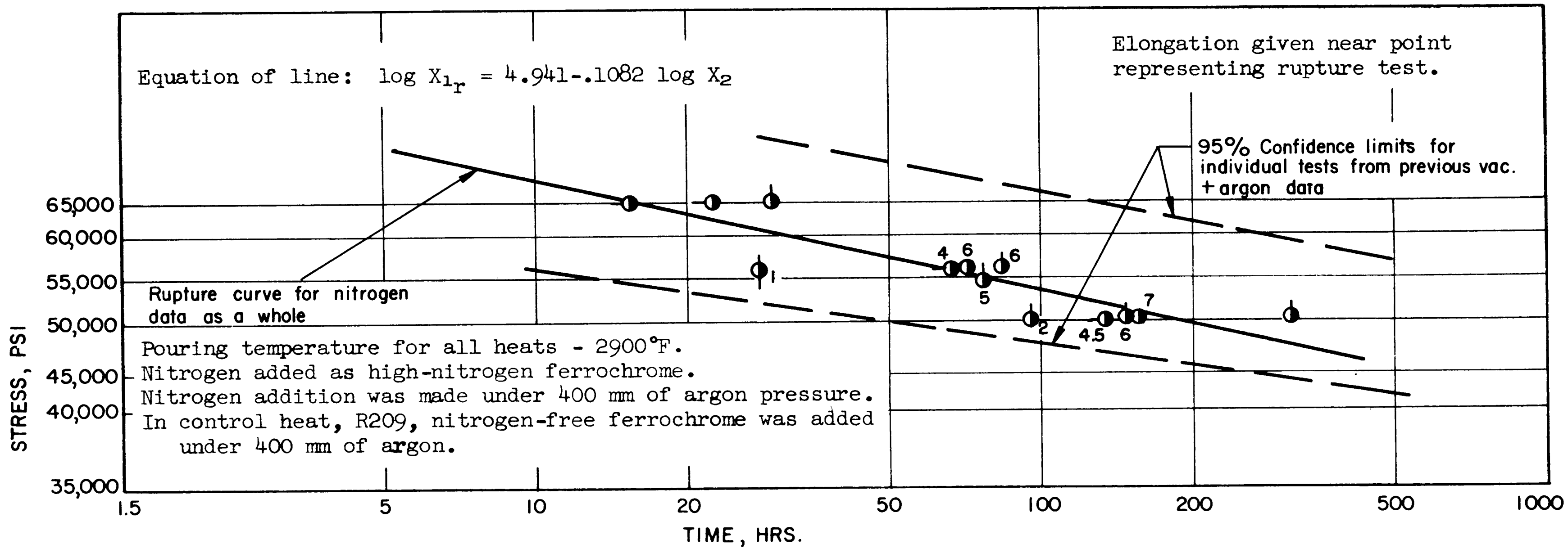
The second method of statistical analysis was used in determining the insignificant effect of nitrogen content upon stress-rupture properties of the Guy-type alloy. All individual rupture tests are within the appropriate 95-percent confidence limits for individual tests.

2. Vapor Collection and Analysis.--The analyses of the metal vapors collected during vacuum + argon melting are shown in Table VI. Two different refining times were used to determine the importance of this procedure.

More metal vapor was collected during the 20-minute refining period. It is evident from the data that considerable refining of trace elements occurs during vacuum melting.

Analysis

Heat No.	C	Cr	Mo	Al	Fe	Cb	B	N_2
R209	.16	12.43	6.00	5.40	5.36	2.11	.43	.01
R211	.13	12.43	5.96	5.45	5.36	1.92	.43	.02
R210	.14	12.27	6.09	5.93	5.06	2.0	.43	.03



Heat No.	Atm	Symbol	% N_2 added	% N_2 (analysis)	% Elong., 100 hr
R209	vac	◑	0	.01	6-7
R211	"	◑	.01	.02	5-6
R210	"	◑	.03	.03	4-4.5
R287	"	◑	.10		2

Fig. 10. Effect of nitrogen content upon the stress-rupture properties of vacuum-melted Guy-type alloys.

TABLE VI

ANALYSES OF VAPOR COLLECTED FROM VACUUM-MELTED HEATS OF THE GUY-TYPE ALLOY

Elements Present	Estimated Percentage Range	
	Zero Refining Time	20-Minute Refining Time
Cr	.10 - 1.0	.001 - .01
Mn	.10 - 1.0	.01 - .10
Ni	over 10 percent (major constituent)	over 10 percent (major constituent)
Al	.10 - 1.0	.01 - .10
Cu	.10 - 1.0	.01 - .10
Pb	.01 - .10	.01 - .10
Ca	.01 - .10	.01 - .10
Mg	.01 - .10	.01 - .10
Mo	.01 - .10	.01 - .10
Si	.10 - 1.0	.10 - 1.0
B	.001 - .01	.001 - .01

The effect of refining time upon stress-rupture properties at 1500° F. is shown in Fig. 11. The second statistical method was used to evaluate the effect of refining time. All the rupture tests fall within the appropriate 95-percent confidence limits for individual tests and, therefore, refining time does not affect the 100-hour rupture strength of the Guy-type alloy.

However, zero refining time results in lower ductility at 100 hours as shown in Table VII.

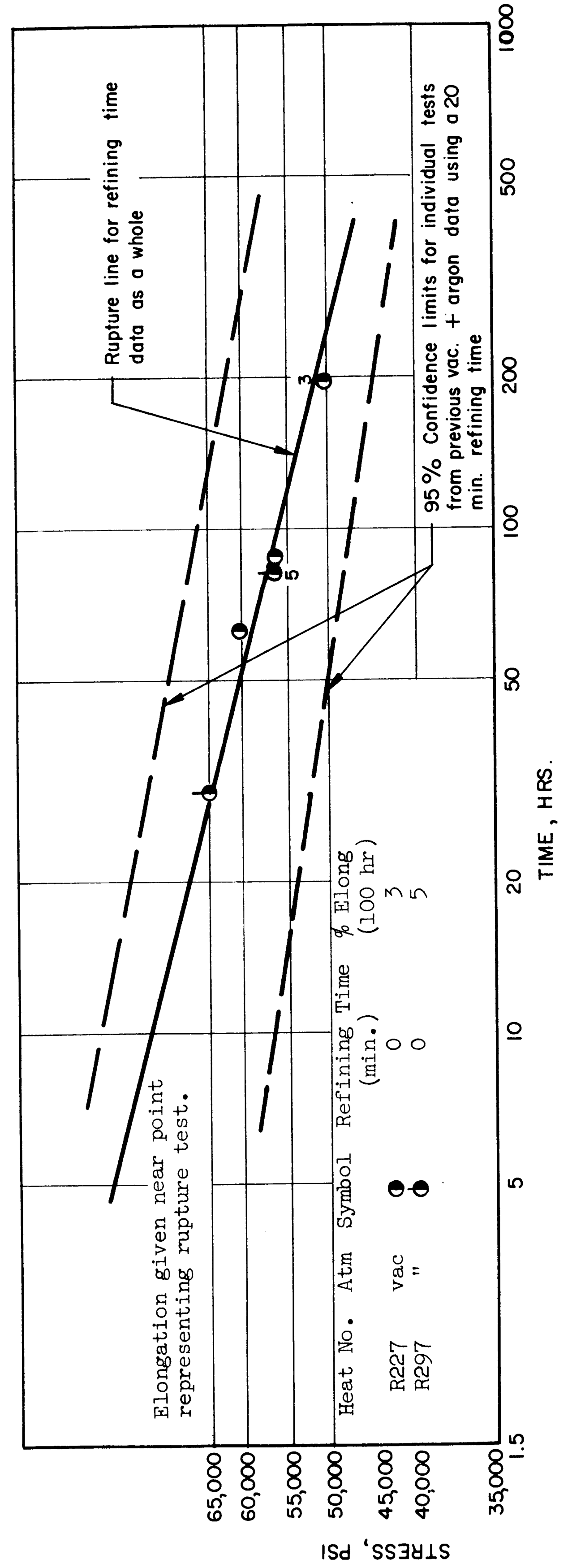


Fig. 11. Effect of refining time upon the stress-rupture properties of vacuum-melted Guy-type alloy.

TABLE VII

EFFECT OF REFINING TIME UPON 100-HOUR ELONGATION OF THE GUY-TYPE ALLOY

Refining Time (minutes)	Percent Elongation
20	7 - 10
0	3 - 5

Therefore, removal during vacuum melting of the trace elements listed in Table VI affects elongation in the same manner as nitrogen.

3. Structural Studies.--In order to determine whether any structural differences existed as a result of air vs vacuum + argon melting of the Guy-type alloy, these structures, before and after rupture testing, were examined by optical microscopy, x-ray diffraction, spectroscopy, electron diffraction, and electron microscopy. The results are conveniently discussed under the above headings.

a. Optical Microscopy.

The microstructures of air, vacuum + argon, and vacuum + argon heats to which nitrogen was added were examined for inclusion content. The inclusion ratings are listed in Table VIII.

TABLE VIII

INCLUSION CONTENT OF THE GUY-TYPE ALLOY AS A FUNCTION OF MELTING ATMOSPHERE

Atmosphere	Inclusion Type (ASTM Inclusion Designation for Steels, Globular-Type Oxides)
vacuum + argon	D - 1
vacuum + argon (nitrogen additions)	D - 4
air	D - 4

Figures 12 and 13 exemplify the ASTM designations given in Table VIII. It is evident from the table and figures that a smaller number of inclusions result from vacuum + argon melting compared with air or nitrogen additions to vacuum + argon melts.

Fig. 12. Inclusion content typical of vacuum + argon heats of the Guy-type alloy (250X, unetched).

Remarks: No inclusions present in entire cross section of test bar.

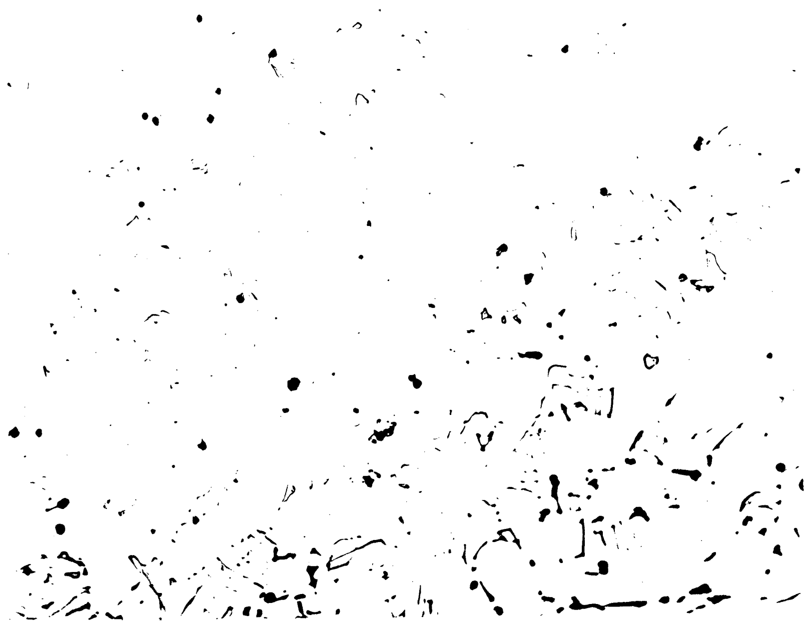


Fig. 13. Inclusion content typical of the Guy-type alloy melted under vacuum + argon (with nitrogen additions) and air atmospheres. (250X, unetched)

Remarks: Considerable amount of inclusions present, micrograph representative of entire cross section of test bar.

b. X-ray Diffraction.

X-ray diffraction patterns have been obtained from minor-phase extracts, powder samples, and solid samples of the Guy-type alloy for both air and vacuum + argon melting atmospheres. Since the phases present in the as-cast condition may disappear and new phases appear during rupture testing at 1500° F., as-cast and after-rupture-testing patterns were obtained for each of the above atmospheres.

The diffraction results are shown in Appendices VII-X. The phases Cr_7C_3, Fe_2B, and Cb(CN) have been identified along with the F. C. C. matrix lines. No appearance or disappearance of any of these phases occurs with melting atmosphere or with the as-cast or after-rupture-testing condition of the specimen.

Other diffraction data indicate the presence of either a precipitate based on the $Ni_3Al(\gamma')$ phase or the existence of a superlattice formation within the alloy matrix.

c. Spectroscopy.

To insure the presence of the above elements (Cr, B, Fe) and to measure their relative concentrations, the minor-phase extracts were analyzed spectrographically. The lines for the above three elements were definitely present in the spectrum of the vacuum + argon, as-cast, and after-rupture-testing extracts and the air, as-cast, and after-rupture-testing extracts.

To measure the relative concentrations of Fe and B in the four samples, the intensities of a sensitive iron line, one of the lines of the boron doublet, and a chromium line, whose intensity remained relatively constant for all four samples, were measured. (Chromium was the major element present in the extracts.) The intensity ratios Fe/Cr and B/Cr were calculated from the above measurements and are shown in Table IX.

These data show that air heats contain a greater percentage of boron and a smaller percentage of iron than vacuum + argon melted heats.

As a result of the data in Table IX, it is apparent that Fe_2B is not a pure stoichiometric compound but actually a solid solution with small boundary limits.

d. Electron Diffraction.

Electron diffraction patterns have been obtained from etched surfaces of the four different specimens (air melted, before and after testing; vacuum melted, before and after testing), using reflection diffraction techniques.[2] The patterns obtained to date have been generally of rather poor quality, consisting of weak, diffuse spots and having rather heavy backgrounds. Because of their spotty character, accurate measurements of interplanar spacings have not been possible. It appears, however, that the patterns are essentially the same for all four specimens.

TABLE IX

RELATIVE CONCENTRATIONS OF IRON AND BORON IN MINOR-PHASE EXTRACTS OF THE GUY-TYPE ALLOY

Melting Atm	Condition	Fe/Cr	B/Cr
vacuum + argon	as-cast	.89	1.58
vacuum + argon	after testing	.89	1.57
air	as-cast	.76	1.98
air	after testing	.64	2.21

e. Electron Microscopy.

To date, microstructure examination of electron microscopy has yielded one definite difference between air- and vacuum-melted structures. In all air-melted heats, a rod-like precipitate appears in the microstructure of after-tested specimens. This precipitate is shown in Fig. 14. The rod-like phase appears to precipitate along definite crystallographic planes.

The general structure of the γ' (based on Ni_3Al) precipitate within the matrix is shown in Fig. 15. No difference in size, shape, etc., of the γ' precipitate is evident for air- or vacuum-melted samples.

The etchant used in the above work is designed to delineate the over-all microstructure. Several other etchants, which bring out grain boundaries, grain-boundary precipitates, and selectively etch the γ' phase, have only been tried on a preliminary basis. These preliminary tests indicate the possibility of a grain-boundary precipitate present in air-melted but not vacuum-melted samples.

The research on the mechanism of the influence of vacuum melting upon stress-rupture properties is summarized below:

1. Nitrogen and zero refining time during vacuum melting reduces ductility of vacuum + argon melts to air-melted values.

Fig. 14. Electron micrograph of rod-like precipitate found in air-melted specimens after testing (Guy-type alloy). (10,000 X)

Remarks: Etched electrolytically in HF, glycerine, and alcohol mixture.

Fig. 15. Electron micrograph of the general structure of the matrix and γ' precipitate (Guy-type alloy). (10,000 X)

Remarks: Etched electrolytically in HF, glycerine, and alcohol mixture.

2. Vacuum melting removes many elements which might be harmful to stress-rupture properties.

3. The phases Cr_7C_3, Fe_2B, and Cb(CN) have been identified in both air- and vacuum-melted heats.

4. A rod-like precipitate is present only in the microstructure of air samples, after testing.

CONCLUSIONS

Following the arrangement of the report, the conclusions are best given under two divisions: A, Effects of Additional Processing Variables, and B, Vacuum-Melting Effects.

A. EFFECTS OF ADDITIONAL PROCESSING VARIABLES

1. Charge preheat time, that is, the time during which the charge is visibly heated before melting, has a major effect. An increase from a preheat period of 5 to a period of 20 minutes improves the 100-hour, 1500° F. rupture strength of the Guy-type alloy from 40,000 to 50,000 psi.

2. Pouring temperatures of 2660°, 2770°, and 2950° F. for the Guy-type alloy and of 2660°, 2760°, and 2950° F. for the GMR-235-type alloy resulted in no change in stress-rupture properties at 1500° F. (95-percent confidence limits).

3. Variations in pressure from 0 - 15 psig during pouring were without effect upon the stress-rupture properties (1500° F.) of Guy- and GMR-235-type alloys.

4. Variations in the time during which the melt was in the liquid state (under argon) up to two hours were without effect upon the stress-rupture properties (1500° F.) of Guy- and GMR-235-type alloys.

5. Preheat temperature of the mold was raised to 1800° F. in place of the usual 1600° F. without effect upon the stress-rupture properties (1500° F.) of Guy-type alloy.

B. VACUUM MELTING

1. Late nitrogen additions from 0 to .10 percent N to vacuum melts of Guy-type alloy decreased the elongation regularly from 7-10 percent to 2

percent elongation (at 1500° F., 100-hour). No effect upon strength was noted.

2. Zero refining time during vacuum melting decreased the elongation from 7-10 percent to 3-5 percent in Guy-type alloys. No effect upon strength was noted.

3. Vapor samples collected during vacuum melting showed appreciable amounts of deleterious elements, such as lead.

4. Structural studies employing electron diffraction, spectroscopic, and x-ray diffraction techniques identified the phases Cr_7C_3, Fe_2B, and Cb(CN). These three phases were present in air- and vacuum-melted samples.

5. Electron microscopy disclosed a rod-like precipitate which appeared only in air-melted samples, after testing.

SUGGESTIONS FOR FUTURE WORK

The work of the past two years has demonstrated that vacuum melting produces very definite improvement in the 100-hour elongation of nickel-base alloys. It is also evident that the 100-hour elongation of vacuum heats is decreased to the elongation of air-melted heats by nitrogen additions and zero refining time. In view of the above discussion, the following future research would greatly add to the understanding of the effects of vacuum melting upon stress-rupture properties:

A. Further examination of vacuum-melted microstructures by electron microscopy.

B. An investigation of the physical chemistry involved during vacuum melting.

C. The investigation of the effect of ordering upon stress-rupture properties.

A. EXAMINATION OF VACUUM-MELTED MICROSTRUCTURES

The preliminary investigations with different selected etchants revealed the possibility of a grain-boundary precipitate present only in air-melted heats. Since this type of precipitation is known to decrease ductility, the presence of such a grain-boundary phase could explain the difference in ductility of air- and vacuum-melted heats. Therefore, it is suggested that research along these lines be continued to explain the elongation differences in vacuum- vs air-melted heats of the Guy-type alloy.

B. PHYSICAL CHEMISTRY OF VACUUM MELTING

Preliminary analysis of vapors collected during vacuum melting disclosed important amounts of deleterious elements such as lead. This work indicates that further quantitative analyses of the purification during vacuum melting by evolution of undesirable elements might also explain the improvements in properties which have been obtained. The entire field of vacuum melting is of such widespread interest and importance that the development, in this way, of basic data might constitute major progress.

It is proposed, therefore, first, that vapor samples be collected over melts prepared under various conditions and that the vapor analysis be correlated with elevated-temperature properties.

Second, the elements which seem to have major effects could be reintroduced separately into vacuum melts and individual effects upon elevated-temperature properties determined. At the same time, the evolution of deleterious elements from the melts should be followed quantitatively along the classical lines of physical chemistry, developing activity data for the important harmful trace elements.

C. INVESTIGATION OF THE EFFECT OF ORDERING UPON STRESS-RUPTURE PROPERTIES

It is suggested that a small amount of research be devoted to the exploration of the effect of ordering upon the elevated-temperature properties of these nickel-chromium-aluminum alloys. A high-temperature x-ray camera operating with a bent calcium fluoride monochromat is now available and could be used to determine ordering temperatures for several alloys. The changes in elevated-temperature properties accompanying ordering could be developed.

In addition to the foregoing, there is a portion of work to be completed involving other processing variables. A number of unsuccessful attempts were made to evaluate the effect of grain size. A procedure has now been developed at Michigan to produce very fine grained material using graphite molds and it is recommended that representative specimens be tested.

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APPENDICES

APPENDIX I

EFFECT OF CHARGE PREHEAT TIME UPON THE STRESS-RUPTURE PROPERTIES OF AIR AND ARGON-PROTECTED GUY-TYPE ALLOY

Pouring Temperature, 2800°F.
Pouring Pressure, 5 psig.

Testing Temperature, 1500°F.

Heat	Atm	Chemical Composition (1)							Stress	Rupture Life (Hours)	Percent Elongation (2)
		C	Cr	Mo	Al	Fe	Cb	B			
R-218	air	.11	11.78	5.80	6.22	4.65	1.78	.43	60,000	39.3	5
									55,000	67.2	5
									50,000	111	3
									45,000	116	3
R-285	air	charge same as R-218							60,000	7.5	1.5
									50,000	173	2.5
R-300	air	charge same as R-218							60,000	36.9	2
									45,000	117	-
R-213	argon	charge same as R-218							60,000	14.2	3
									50,000	69.3	2
									45,000	128	2.5
									40,000	282	5
R-284	argon	charge same as R-218							50,000	130	4
R-301	argon	charge same as R-218							60,000	37.7	3
									50,000	83.7	2

(1) Balance Ni
(2) Percent elongation in one inch measured after fracture.

APPENDIX II

EFFECT OF POURING TEMPERATURE UPON THE STRESS-RUPTURE PROPERTIES OF ARGON-PROTECTED AND VACUUM + ARGON MELTED GUY-TYPE AND ARGON-PROTECTED GMR-235-TYPE ALLOYS

Pouring Pressure Under Argon Protection, 5 psig.
Improved Melting Practice Used Only For Argon-protected Guy Alloy
Standard Vacuum Procedure Employed

Type Alloy	Heat	Pouring Temp.	Atm.	Chemical Composition (1) C	Cr	Mo	Al	Fe	Cb	B	Stress	Rupture Life (Hours)	Percent (2) Elongation
Guy	R-212	2950	argon protected	.10	12.86	5.74	6.24	4.94	1.54	.38	50,000	190	6
											45,000	286	5
Guy	R-213	2770	argon protected	charge same as R-212							60,000	14.2	3
											50,000	69.3	2
											45,000	128	2.5
											40,000	282	5
Guy	R-214	2670	argon protected	charge same as R-212							55,000	86.3	5
											50,000	95	3
Guy	R-302	2670	argon protected	charge same as R-212							50,000	92.8	3
											40,000	231	2
Guy	R-223	2800	vacuum + argon	charge same as R-212							65,000	27.5	3.5
											60,000	50.7	5
											56,000	100	6
											50,000	219	8
Guy	R-224	2660	vacuum + argon	charge same as R-212							65,000	28.4	4
											60,000	61.5	5
											56,000	103	8
											50,000	249	7
Guy	R-225	3000	vacuum + argon	charge same as R-212							50,000	242	10
GMR235	R-150	2950	argon protected	.14	14.24	3.0	3.03	9.13	1.56	.06	45,000	58.9	10
											40,000	126	10
											35,000	278	6
GMR235	R-151	2760	argon protected	same charge as R-150							45,000	19	7
											35,000	329	7
GMR235	R-152	2660	argon protected	same charge as R-150							45,000	35.6	15.5
											40,000	118	9
											35,000	346	12

(1) Balance Ni
(2) Percent elongation in one inch measured after fracture.

APPENDIX III

EFFECT OF POURING PRESSURE UPON THE STRESS-RUPTURE PROPERTIES OF ARGON-PROTECTED GUY- AND GMR-235-TYPE ALLOYS

Pouring Temperature of Guy Alloy 2800° F.
Pouring Temperature of GMR 235 2850° F.

Improved Melting Practice Employed Only For Guy Alloy

Type Alloy	Heat	Pouring Pressure	Chemical Composition C	Cr	Mo	Al	Fe	Cb	B	Stress	Rupture Life (Hours)	Percent Elongation
Guy	R-216	0	.10	12.24	6.0	6.19	4.25	1.76	.41	50,000	39.3	2.5
										45,000	128	2.5
Guy	R-215	8	charge same as R-216							55,000	68.1	3
										50,000	61.4	3
										45,000	141	4
Guy	R-217	12	charge same as R-216							45,000	105	---
										40,000	219	3
			C	Cr	Mo	Al	Fe	Ti	B			
GMR 235	R-154	15	.13	14.09	2.82	3.10	9.05	1.50	.08	45,000	45	8
										40,000	113	10
										35,000	304	9
GMR 235	R-153	10	charge same as R-154							45,000	23	6.5
										40,000	128	9
GMR 235	R-155	0	charge same as R-154							40,000	130	6
										35,000	348	13

APPENDIX IV

EFFECT OF HOLDING TIME IN THE LIQUID STATE UPON THE STRESS-RUPTURE PROPERTIES OF ARGON-PROTECTED GUY- AND GMR-235-TYPE ALLOYS

Pouring Temperature for Guy 2800° F.
Pouring Temperature for GMR 235 2850° F.

Improved Melting Practice Employed For Both Alloys
Pouring Pressure for both alloys 5 psig

Type Alloy	Heat	Holding Time (Hours)	Chemical Composition C	Cr	Mo	Al	Fe	Cb	B	Stress	Rupture Life (Hours)	Percent Elongation
Guy	R-219	2	.10	12.40	5.67	6.19	4.95	1.48	.40	50,000	149	6
										45,000	366	6
Guy	R-220	1/2			charge same as R-219					50,000	114	4
										45,000	400	6
			C	Cr	Mo	Al	Fe	Ti	B			
GMR 235	R-221	2	.16	14.86	5.67	3.50	10.38	1.86	.06	50,000	17.4	14
										40,000	107	12
										35,000	309	10
GMR 235	R-222	1/2			same charge as R-221					50,000	28.9	10
										40,000	102	6
										35,000	459	8

APPENDIX V

EFFECT OF MOLD PREHEAT TEMPERATURE UPON THE STRESS-RUPTURE PROPERTIES OF AIR AND ARGON-PROTECTED GUY ALLOY

Improved Melting Practice Used For Both Air and Argon-Protected Heats
Pouring Temperature 2800° F.
Pouring Pressure 5 psig

Heat	Atm	Mold Temp. (°F)	Chemical Composition C	Cr	Mo	Al	Fe	Cb	B	Stress	Rupture Life (Hours)	Percent Elongation
R-278	air	1800								50,000	119	4
										45,000	317	1
R-281	air	1800								50,000	164	3
R-279	argon	1800								50,000	326	3
										45,000	88.7	3
R-280	argon	1800								50,000	91.6	3
										45,000	220	4

APPENDIX VI

EFFECT OF NITROGEN CONTENT UPON THE STRESS-RUPTURE PROPERTIES OF VACUUM + ARGON MELTED GUY-TYPE ALLOY

Pouring Temperature 2900° F.

Nitrogen Added As High-Nitrogen Ferrochrome
Nitrogen Addition Was Made Under 400 mm. Of Argon Pressure
In Control Heat, R-209, Nitrogen-Free Ferrochrome Was Added Under 400 mm. Of Argon

Heat No.	Percent N_2 Added	C	Cr	Mo	Al	Fe	Cb	B	N_2	Stress	Rupture Life (Hours)	Percent Elongation
		Chemical Composition										
R-209	0	.16	12.43	6.0	5.40	5.36	2.11	.43	.01	65,000	22.1	7
										56,000	75.2	7
										56,000	67.2	5
										50,000	199	8
										50,000	107	6
R-211	.01	.13	12.43	5.96	5.45	5.36	1.92	.43	.02	65,000	29.5	6
										56,000	83.3	6
										55,000	75.1	5
										50,000	311	5
										50,000	154	5
R-210	.03	.14	12.27	6.09	5.93	5.06	2.0	.43	.03	65,000	15.2	3
										56,000	68.1	4
										50,000	148	4
										50,000	120	5
R-287	.10								.07	56,000	27.7	1.0
										50,000	95.2	2.0

APPENDIX VII

EFFECT OF REFINING TIME UPON THE STRESS-RUPTURE PROPERTIES OF VACUUM + ARGON MELTED GUY-TYPE ALLOY

Pouring Temperature 2900° F.

Heat	Refining Time (Min.)	Chemical Composition							Stress	Rupture Life (Hours)	Percent Elongation
		C	Cr	Mo	Al	Fe	Cb	B			
R-227	0								60,000	61.1	4
									56,000	88.1	3
									56,000	25	2.5
									50,000	191.2	3
R-297	0								65,000	30	4.5
									56,000	83.8	5
R-298 Control Heat	20								56,000	78.4	4

APPENDIX VIII

X-RAY DIFFRACTION RESULTS FOR THE GUY-TYPE ALLOY MELTED IN VACUUM + ARGON, AS-CAST

Cu Rad. Extract Pattern		Cr Rad. Powder Pattern		Cu Rad. Solid Sample Pattern		Cr_7C_3		Fe_2B		CbC CbN		Cr_3C_2		γ' Precipitate Or Ordered F.C.C. Matrix		F.C.C. Matrix	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
								3.62	vw								
														3.56			
				3.22	vw	3.22	mw										
												3.14	.4				
				3.12	w												
3.09	m																
						3.01	mw										
2.87	w																
												2.74	.7				
						2.70	w										
						2.65	w										
				2.59	m												
2.56	s																
								2.55	wm	2.55	vs	2.55	.8				
														2.52			
				2.48	mw	2.48	mw					2.48	.5				
2.46	m											2.46	.5				
						2.38	m										
												2.30	1.0				
						2.26	w					2.26	.6				
												2.23	1.0				
						2.22	w										
2.20	w									2.20	s						
		2.16	w														
						2.15	w										
								2.13	m								
						2.12	s										
2.10	s			2.10	mw							2.10	.7				
		2.07	m	2.07	vs									2.07		2.07	s
						2.04	s										
2.03	wm																
		2.02	vs														
								2.01	s								
				2.00	mw												
												1.99	.8				
1.98	s																
						1.96	m										
						1.86	s					1.86	1.0				
		1.85	w														
						1.84	s	1.84	w								
1.82	w			1.82	mw	1.82	mw	1.82	w			1.82	1.0				
		1.79	w											1.79		1.79	s
						1.78	w					1.78	.7				
						1.76	m					1.76	.7				
						1.71	m										
												1.66	.4				
								1.64	mw								
								1.62	mw			1.62	.5				
						1.61	mw										
														1.60			
												1.58	.5				
1.56	m			1.56	vw					1.56	ms						
1.54	vw																
												1.53	.8				
		1.52	w														
						1.51	m										
												1.50	.8				
						1.46	w							1.46			
												1.44	.5				
		1.43	s			1.43	s										
												1.42	.4				
												1.41	1.0				
1.40	vw			1.40	vw												
						1.38	w					1.38	.4				
1.36	vw																
						1.35	s										
1.34	ms			1.34	vw			1.34	ms								
		1.33	w							1.33	ms	1.33	.4				
								1.28	wm								
1.27	ms	1.27	s	1.27	ms					1.27	mw			1.27		1.27	m
						1.26	m					1.26	.8				
1.24	vw	1.24	vw														
														1.19			
														1.13			
																1.09	m
				1.09	m												
														1.08			
				1.04	mw									1.04		1.04	
				.892	w											.892	
				.83	m											.83	

APPENDIX IX

X-RAY DIFFRACTION RESULTS FOR THE GUY-TYPE ALLOY MELTED IN VACUUM + ARGON, AFTER RUPTURE TESTING*

Powder Pattern		Solid Sample Pattern		Cr_7C_3		Fe_2B		CbC CbN		Cr_3C_2		γ' Precipitate Or Ordered F.C.C. Matrix		F.C.C. Matrix	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
						3.62	vw								
												3.56			
				3.22	mw										
		3.14	vw							3.14	.4				
				3.01	mw										
										2.74	.7				
				2.70	w										
				2.65	w										
		2.61	m												
		2.58	w												
2.57	vw														
						2.55	wm	2.55	vs	2.55	.8				
												2.52			
		2.50	mw												
										2.49	.5				
				2.48	mw										
										2.46	.5				
				2.38	m										
										2.30	1.0				
				2.26	w					2.26	.6				
										2.23	1.0				
				2.22	w										
								2.20	s						
2.17	w														
				2.15	w										
		2.13	ms			2.13	m								
				2.12	s										
2.11	wm														
										2.10	.7				
		2.07	ms									2.07		2.07	s
2.06	vs														
				2.04	s										
2.02	vs														
						2.01	vs								
		2.00	ms												
										1.99	.8				
				1.96	m										
				1.90	w					1.90	1.0				
1.87	w														
				1.86	s					1.86	1.0				
1.85	w														
				1.84	s	1.84	w								
				1.82	mw	1.82	w			1.82	1.0				
1.81	w														
		1.79	vs									1.79		1.79	s
1.78	s			1.78	w					1.78	.7				
				1.76	m					1.76	.7				
				1.71	m										
										1.66	.4				
		1.65	vw												
						1.64	mw								
						1.62	mw			1.62	.5				
		1.61	vw	1.61	mw										
												1.60			
										1.59	.5				
		1.56	vw					1.56	ms						
										1.53	.8				
				1.51	m										
										1.50	.8				
				1.46	w							1.46			
										1.44	.5				
1.43	s			1.43	s										
										1.41	1.0				
		1.40	vw												
				1.38	w	1.38	w			1.38	.5				
				1.35	s										
		1.34	vw			1.34	m								
1.33	w							1.33	ms	1.33	.4				
						1.28	wm								
1.27	vs	1.27	vs					1.27	mw			1.27		1.27	m
				1.26	s					1.26	.8				
												1.19			
												1.13			
														1.09	m
		1.09													
												1.08			
		1.04										1.04		1.04	
		.892												.892	
		.83												.83	

* Same extract pattern as shown in Appendix VII

APPENDIX X

X-RAY DIFFRACTION RESULTS FOR THE GUY-TYPE ALLOY MELTED IN AIR, AS-CAST*

Powder Pattern d	I	Solid Sample Pattern d	I	Cr_7C_3 d	I	Fe_2B d	I	CbC CbN d	I	Cr_3C_2 d	I	γ' Precipitate Or Ordered F.C.C. Matrix d	I	F.C.C. Matrix d	I
		3.62	mw			3.62	vw								
												3.56			
				3.22	mw										
										3.14	.4				
		3.13	w												
				3.01	mw										
		2.88	w												
										2.74	.7				
				2.70	w										
				2.65	w										
		2.59	m												
								2.56	vs	2.56	.8				
						2.55	wm								
		2.54	mw												
												2.52			
		2.49	mw												
				2.48	mw					2.48	.5				
				2.38	m										
										2.30	1.0				
				2.26	w					2.26	.6				
										2.23	1.0				
		2.22	w	2.22	w										
								2.20	s						
						2.13	m								
		2.12	mw	2.12	s										
										2.10	.7				
		2.07	vs									2.07		2.07	s
2.06	vs														
				2.04	s										
2.02	vs														
						2.01	vs								
		1.99	m							1.99	.8				
				1.96	m										
				1.90	w					1.90	1.0				
				1.86	s					1.86	1.0				
				1.84	s	1.84	w								
				1.82	mw	1.82	w			1.82	1.0				
		1.79	m									1.79		1.79	s
				1.78	w					1.78	.7				
1.76	m			1.76	m					1.76	.7				
										1.75	.7				
				1.71	m										
										1.66	.4				
						1.64	mw								
				1.62	mw					1.62	.5				
						1.61	mw			1.59	.5				
								1.56	ms						
										1.53	.8				
				1.51	m										
										1.50	.8				
				1.46	w										
										1.44	.5				
				1.43	s										
										1.42	.4				
										1.41	1.0				
										1.39	.4				
						1.38	w								
										1.37	.5				
				1.35	s	1.35	s								
		1.34	w												
1.33	w							1.33	ms						
										1.30	.3				
						1.28	wm			1.28	.5				
1.27	vs	1.27	m					1.27	mw			1.27		1.27	m
				1.26	s					1.26	.8				
												1.19			
												1.13			
		1.09												1.09	m
												1.08			
		1.04										1.04		1.04	
		.892												.892	
		.83												.83	

*Same extract pattern as shown in Appendix VII.

APPENDIX XI

X-RAY DIFFRACTION RESULTS FOR THE GUY-TYPE ALLOY MELTED IN AIR, AFTER RUPTURE TESTING*

Powder Pattern		Solid Sample Pattern		Cr_7C_3		Fe_2B		CbC CbN		Cr_3C_2		γ' Precipitate Or Ordered F.C.C. Matrix		F.C.C. Matrix	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
		3.68	mw												
						3.62	vw								
												3.56			
		3.44	vw												
				3.22											
										3.14	.4				
				3.01	mw										
										2.74	.7				
				2.70	w										
				2.65	w										
		2.61	m												
		2.56	mw							2.56	.8				
						2.55	wm	2.55	vs						
												2.52			
		2.48	mw	2.48	mw					2.48	.5				
				2.38	m										
		2.32	w												
										2.30	1.0				
				2.26	w					2.26	.6				
										2.23	1.0				
								2.20	s						
						2.13	wm								
		2.12	m	2.12	s										
										2.10	.7				
		2.07	vs									2.07		2.07	s
2.06	s														
				2.04	s										
2.02	wm														
						2.01	vs								
		1.99	m							1.99	.8				
				1.96	m										
				1.90	w					1.90	1.0				
				1.86	s					1.86	1.0				
				1.84	s										
		1.82	mw	1.82	mw					1.82	1.0				
		1.80	m												
1.79	s											1.79		1.79	s
				1.78	w					1.78	.7				
				1.76	m					1.76	.7				
				1.71	m										
										1.66	.4				
						1.64	mw								
				1.62	mw	1.62	mw			1.62	.5				
		1.60	w												
										1.59	.5				
		1.56	w					1.56	ms						
										1.53	.8				
				1.51	m										
										1.50	.8				
				1.46	w										
										1.44	.5				
1.43	m			1.43	s										
										1.42	.4				
										1.41	1.0				
										1.39	.4				
						1.38	w	1.38	ms						
										1.37	.5				
				1.35	s	1.35	s								
		1.33	vw												
										1.30	.3				
						1.28	wm			1.28	.5				
1.27	vs	1.27	vw					1.27	mw			1.27		1.27	m
				1.26	s					1.26	.8				
												1.19			
												1.13			
		1.09												1.09	
												1.08			
		1.04										1.04		1.04	
		.892												.892	
		.83												.83	

*Extract pattern same as Appendix VII.