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Final Report

THE EFFECT OF MELTING AND CASTING ATMOSPHERES ON THE STRESS-RUPTURE PROPERTIES OF CAST NICKEL-BASE ALLOYS

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SUMMARY

Improvement of cast heat-resistant alloys in the past has depended upon the investigation of new compositions. By contrast, this work is directed toward determining the effect of melting and casting atmospheres upon the properties of existing alloys. The following table of data, obtained for three promising nickel-base alloys, indicates that melting and casting atmospheres can be very important in influencing 100-hour rupture strength and elongation at 1500°F.

Alloy Type	Published Properties		This Investigation (Melted under vacuum, poured under argon)	
	100-Hour Rupture Strength psi	Percent Elongation	100-Hour Rupture Strength psi	Percent Elongation (100 hours)
Guy	49,000	2-5	56,000	7-10
Inco 700	43,000*	10*	42,000	21
GMR 235	39-40,000	6-10	42,000	14-19

*Solution treated and aged, all others as cast.

The strength increases were most pronounced in the high-boron material Guy type, while improvements in ductility were apparent in all cases. The low nitrogen contents, produced by the vacuum-melting technique, may account for the superior strength and ductility.

These data indicate further that the effects of melting and casting atmospheres are of different magnitudes for various alloys. The improvement in ductility of the Guy-type alloy by vacuum melting may have led to the enhanced strength by avoiding rupture in the second stage of creep which may occur in the brittle, air-melted material.

INTRODUCTION

As a result of the conversion to gas-turbine engines by the aircraft industry, alloys for better service at elevated temperatures have become essential. Since the rotor blades in these engines are subjected to the highest temperatures and stresses, the materials used here are critical. Initially, these blades were made of cobalt-base alloys, but the scarcity and expense of cobalt has led to considerable research on nickel-base alloys.[1,2,3] Most of the effort in these researches has been concentrated on variations in chemical analyses correlated with changes in stress-rupture properties at 1500°F.

Most of the nickel-base alloys developed in this manner are titanium or aluminum bearing, or both. Since these elements are known to give casting difficulties through severe metal oxidation and gas solution during melting, it was questioned whether or not the published properties represented the true potential of these materials. Therefore, research concerning the effects of melting and casting atmospheres upon stress-rupture properties appeared necessary to parallel the investigations of compositional effects.

OBJECTIVE

The objective of this investigation, therefore, is to evaluate the effect of melting and casting atmospheres and other processing variables upon the room-temperature and stress-rupture properties of promising nickel-base alloys. This report is concerned chiefly with the effects of the atmosphere during melting and casting.

PROCEDURE

A literature survey indicated that the Guy,[1] Inco 700,[4] and GMR 235[5] alloy types are the most promising nickel-base materials at present. The properties and chemical compositions of these alloys are shown in Table I. All three materials contain considerable percentages of titanium and aluminum and, therefore, as mentioned in the Introduction, melting and casting atmospheres should be significant in determining ultimate properties.

The general procedure followed in this investigation is conveniently discussed under the following headings:

1. Preliminary Experiments
    a. Specimen design
    b. Gating design
    c. Pouring temperatures

2. Standard Procedures
    a. Mold preparation
    b. Melting and pouring
    c. Chemical analysis
    d. Mechanical testing
    e. Metallography

1. PRELIMINARY EXPERIMENTS

Before entering the principal phases of the investigation, it was necessary to establish specimen design, gating design, and pouring practices which would produce acceptable properties, i.e., similar to present published results.

<u>a.</u> <u>Specimen Design.</u>—It was feared that the present standard test bar (.250 diameter, 1-in. gage length) would contain excessive center-line shrinkage in the gage length. Accordingly, two other designs were developed (Fig. 1) which should allow better feeding from the riser.

Specimen Type	Diameter	Gage Length	Length-Diameter Ratio
A	.250	1.0	4:1
B	.250	.50	2:1
C	.125	.25	2:1

To provide a wide range of alloys for evaluation, X-40, GMR 235, and .30%-C steel were selected for the tests. Fortunately, the degree of directional solidification in the standard bar was sufficient to produce radiographically sound bars in all cases. This bar was then used throughout the investigation. Occasional center-line microshrinkage, too fine to affect the radiographs, was encountered. The cooling curves of Fig. 2 indicate a gradient of 100°F/in. at the time of solidification, which is of course the time at which the demand for feed metal is most important.

TABLE I

PROPERTIES AND NOMINAL COMPOSITIONS OF THREE PROMISING CAST NICKEL-BASE ALLOYS

Alloy Type	100-Hour Rupture Strength psi	Percent Elongation at 100 Hours	C	Cr	Mo	Al	Fe	Ti	B	Co	Cb
Guy[1]	48-50,000	3-5	.10	13	6.0	6.0	4.5	---	.50	--	2.0
*Inco 700[4]	41-43,000	10	.10	15	3.0	3.0	.5	2.0	---	28	---
GMR 235[5]	38-40,000	6-10	.15	15.5	5.25	3.0	10.0	2.0	.075	--	---

*Properties from wrought specimens, solution treated 2160°F/2 hours and air-cooled; aged at 1600°F/4 hours and air-cooled.

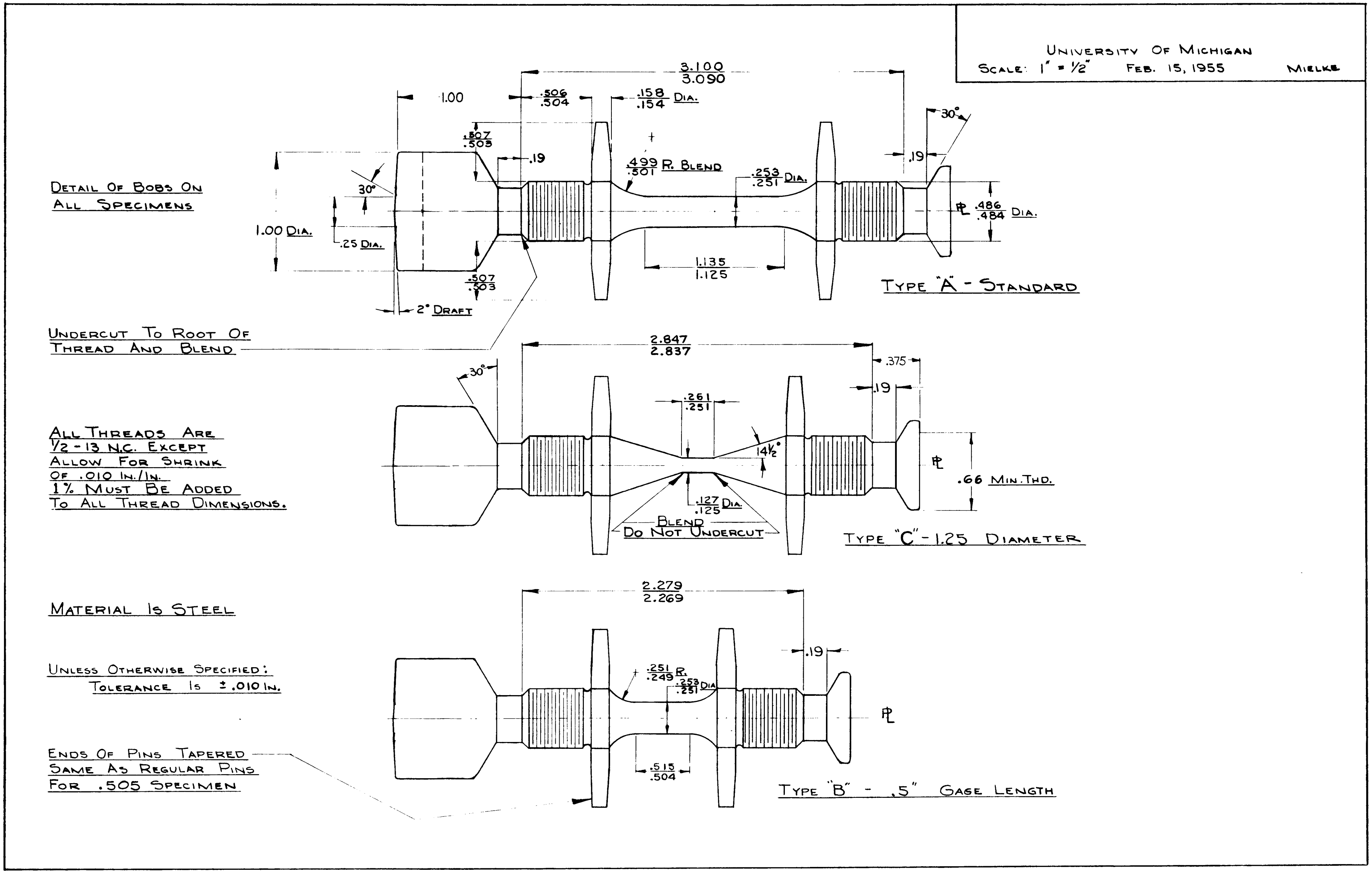


Fig. 1. Cast stress-rupture specimen.

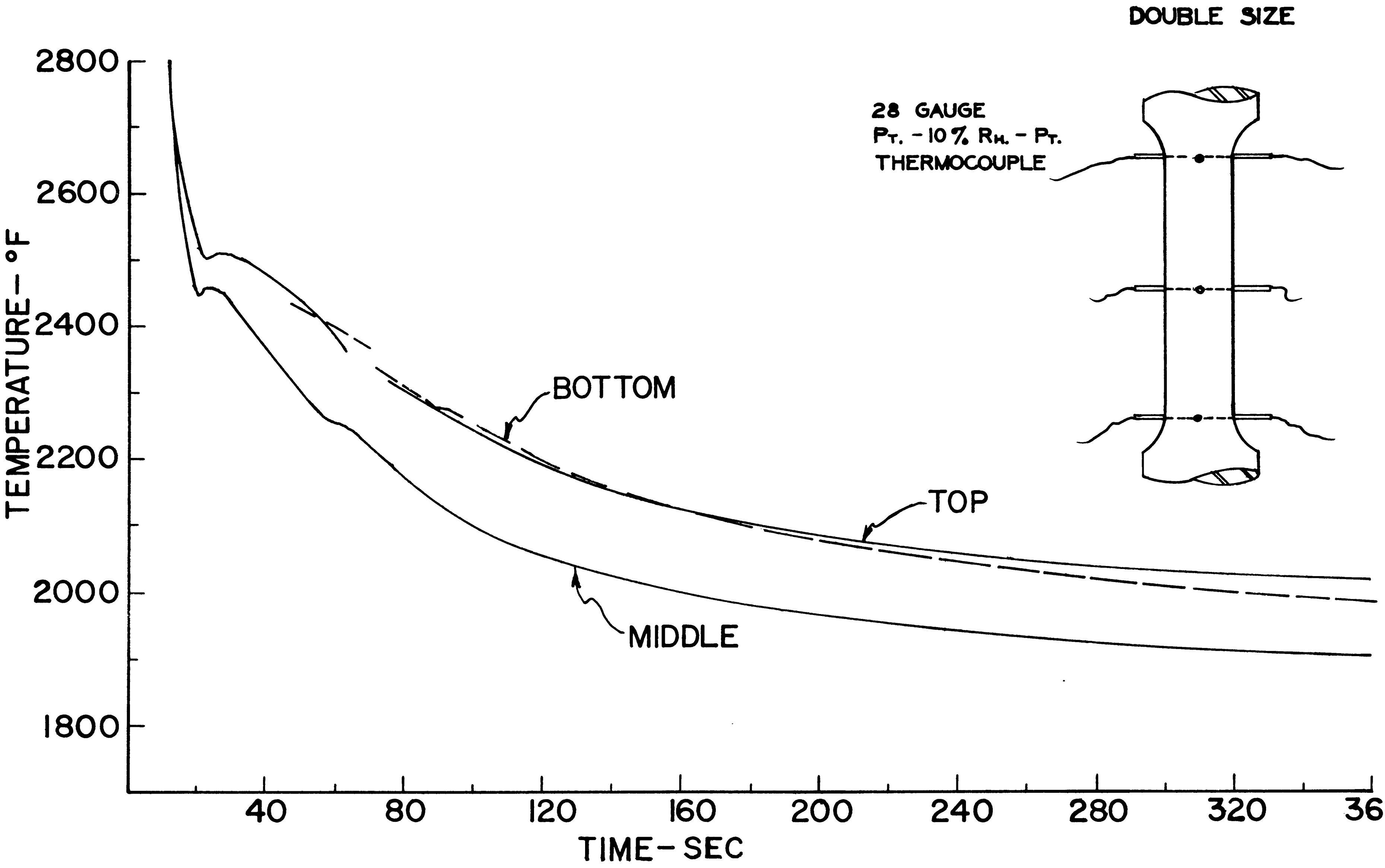


Fig. 2. Test-bar cooling curves.

b. Gating Design.—A new gating system was designed for this investigation for several reasons. The majority of the present systems attempt to deliver molten metal at both ends of the vertically cast bar. It must be recognized that this procedure results in the turbulent meeting of two streams of hot metal in the test bar and can lead to unpredictable variations in drossing and variable thermal gradients during solidification. The principal reason advanced for this type of gating is "to avoid cold metal in the riser."

By contrast, it is generally agreed that a minimum of turbulence is encountered with bottom gating. The General Motors research group[5] has developed a rather elaborate design based on this principle. This mold design, however, still employs an auxiliary runner "to avoid cold riser metal."

It was considered possible by the present investigators that with rapid bottom pouring, a simple bottom gating system might be used, and the design shown in Fig. 3b was developed.

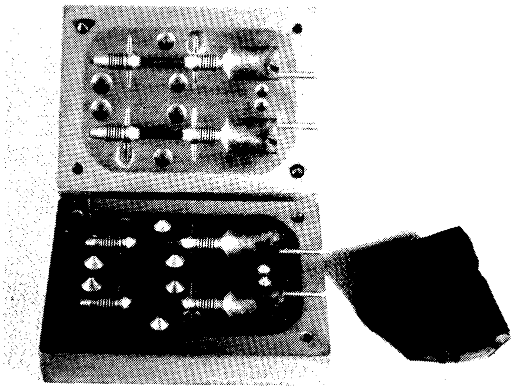
The quantitative data of Fig. 2 indicate that ideal directional solidification with equal feeding contributions from both ends of the test-bar section is obtained with this new arrangement. It should be pointed out, furthermore, that all bars have the same cooling rate because of the symmetrical arrangement about the downsprue. This is not the case in a number of designs now in use in which the bars at interior and corner locations of the cluster have different cooling rates.

c. Pouring Temperatures.—GMR 235 was used as a model to evaluate pouring-temperature effects. Preliminary tests indicated that the influence of temperature was small and that a statistical study of a large sample would be required. (This work is now in progress.) However, to allow the investigation to proceed to the more significant phases, a pouring temperature of 2850° ± 20°F was used for GMR 235 in order to provide satisfactory fluidity and at the same time to insure good casting surfaces. Since it has been established that fluidity is a linear function of the superheat above the liquidus, the other alloys were poured at approximately the same degree of superheat as shown in Table II. These temperatures were maintained for all three

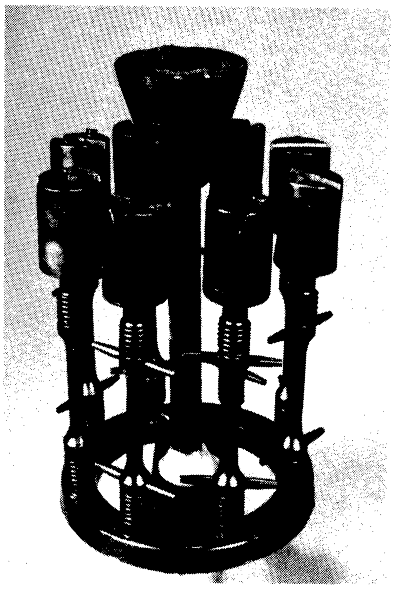
TABLE II

POURING TEMPERATURES OF THE THREE ALLOYS INVESTIGATED

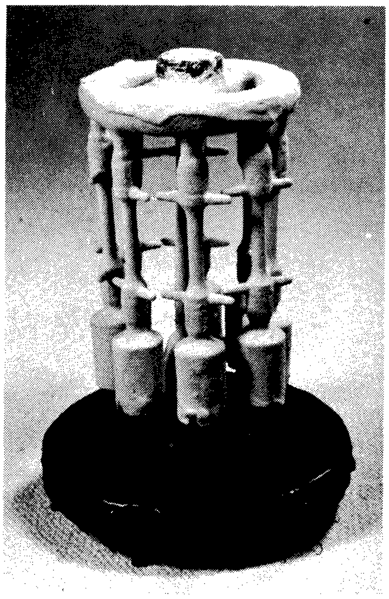
Alloy Type	Liquidus	Pouring Temperature °F	Degree Superheat °F
Guy	~ 2520	2800	280
Inco 700	2530	2800	270
GMR 235	2520	2850	330



a. Test-bar die.

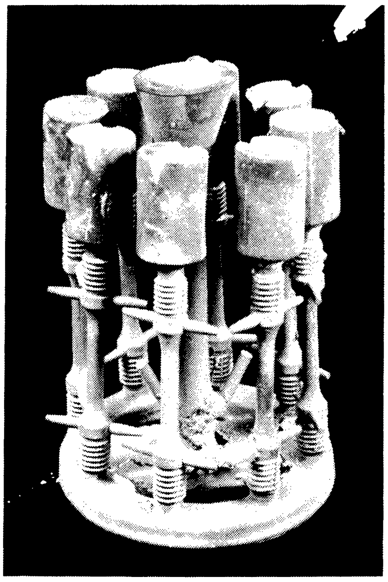


b. Wax cluster.



c. Dipcoated cluster.

d. Mold assembly.



e. Final casting.

Fig. 3. Investment process.

nickel-base alloys throughout the remainder of this investigation. It is recognized that further work to delineate the quantitative effects of pouring temperature, as well as the influence of melting and superheating periods, would be of value and this is now in progress.

2. STANDARD PROCEDURES

While substantial changes in mold design were considered important as just described, a good commercial investment practice was adopted as standard. Accordingly, wax patterns and an ethyl-silicate-base investment were used.

a. Mold Preparation.—This process (Fig. 3) consisted of making wax replicas of the desired final casting, assembling the wax replicas on a wax gating system, dipcoating the replicas with a thin coating of a fine silica slurry, enclosing the assembly in a flask, and finally pouring in the investment material. The mold was allowed to set up at room temperature prior to dewaxing and firing to provide proper green strength for handling.

The wax replicas, in this case wax tensile bars, were made by injecting wax at 80°C into the die shown in Fig. 3a. These bars were then assembled into a cluster shown in Fig. 3b, which was then mounted on a wooden bottom board prior to dipcoating, (Fig. 3c).

The dipcoat mixture is listed below:

Dry materials:	
200-mesh Si flour	10 lb
FeO	.25 lb
Sodium fluoride	13.5 g
Wet materials:	
Nalcoag	875 cc
H_2O	675 cc
Batch A:	
Wetanol	40 g
H_2O	1000 cc
Octyl alcohol	2 drops

The dry materials were mixed thoroughly prior to addition of the wet materials. The Batch-A solution, the wetting agent, was added to the slurry ten minutes prior to dipcoating. To insure removal of adhering air bubbles from the dipcoated cluster, a low-velocity air stream was passed onto the cluster, breaking all bubbles. The dipcoating operation was completed by sprinkling the wet cluster with 40-mesh silica sand and allowing it to dry four hours prior to investing. The coarse 40-mesh silica sand insured good bonding with the back-up investment mix.

Before actually investing, the clusters were surrounded by a stainless-steel flask (Fig. 3d) and the assembly was made water tight by dipping it into wax. A paper extension was sealed to the flask to allow for settling during vibration. The investment mixture is summarized below:

Refractory materials:	
200-mesh Si flour	12.0%
40-80-mesh Si sand	23.5%
G Grog	25 %
P Grog	40 %
MgO (powdered)	.4%

Investment binder:	
Ethyl Silicate 40	2996 cc
Diluter (5% H_2O, 95% Symasol)	2385 cc
Reactor (50% H_2O, 50% Symasol + .75% HCl)	619 cc
	6000 cc

The refractory materials were mixed thoroughly for 30 minutes in a cement mixer. In the preparation of the investment-binder solution, the diluter was added to the ethyl silicate and then the reactor added. The investment binder was added gradually to the refractory materials until the slurry was of the consistency of thin cement, approximately 110 cc/lb. This mixture was then removed from the mixer and poured into the flasks, which were vibrated during pouring. The vibration insured dense packing around the cluster. After approximately four hours at room temperature, the mold possessed enough green strength for handling, dewaxing, and firing.

The temperature and time for dewaxing were approximately 225°F and three hours, respectively. The firing cycle was 1600°F for approximately 10 hours. The dewaxing operation removed the bulk of the wax, while firing removed the remainder and preheated the mold to 1600°F. The fired molds were removed from the furnace just prior to pouring. The critical transformation of silica in the temperature range of 1000°-1200°F prohibited cooling the molds below this range after firing. Storage, however, was easily accomplished before or after dewaxing, with the former preferred.

Dimensions were held to ± .0025 in. with this process. The final test-bar casting cluster is shown in Fig. 3e.

b. Melting and Pouring.—The melting and the pouring of the three experimental nickel-base alloy types, Guy, Inco 700, and GMR 235, were conducted under three atmospheric conditions:

(1) Air
(2) Argon-protected
(3) Vacuum + argon

Melts were made of virgin metals, except as noted in the test. The metals and alloys used, their analyses, and suppliers are listed below in Table III.

TABLE III

RAW MATERIALS

Material	Analysis-Percent	Supplier
Ni (electrolytic)	Ni+Co - 99.95; Fe - .01-.04; C - trace; Si - trace.	Inco
Cr (electrolytic)	Cr - 99.3; Fe - .13; O_2 - .50; H_2 - .004; N_2 - .02.	Electromet
Mo (chips)	Mo - 99^+.	Climax Moly
Al (piglets)	Al - 99.99.	Alcoa
NiB	Ni - 80.4; Al < .01; Fe - 1.73; C - .15; B - 17.39; Si - .31.	Electromet
FeCb	Cb - 57.34; C - .25; Si - 7.8; Fe - balance.	Electromet
Ti (rod)	C - .03; N - .023; Fe - .04; Ti - balance.	Titanium Metals Corp. of America
FeC (alloy)	C - 4.22; Si - .28; Mn - .19; S - trace; P - trace; Fe - balance.	Univ. of Mich. foundry
Co	Co - 99.9.	Belmont Smelting and Refining Works

The crucible charge and order of additions were in general the same for all three types of atmospheres. The nickel, chromium, cobalt, molybdenum, and iron were charged to the crucible. The other alloying elements were added after melt down in the following order: aluminum, carbon, titanium or columbium, and boron. Nickel squares were charged with the aluminum and titanium additions to curb their violent exothermic reactions.

Temperatures for air and argon melting were measured by a Pt - 10% Rh - Pt thermocouple enclosed by a fused-silica tube. Optical pyrometer measurements were used to follow temperatures in vacuum. Heating rates in air and argon were determined for each alloy at a constant kilowatt input. These rates were used to calculate the actual pouring temperature, since the thermocouple had to be removed to place the mold on the furnace. The time interval between the last temperature reading and pouring was exactly two minutes. Liquidus temperatures were determined for each alloy investigated.

Any change in the general procedure outlined above is indicated in the sections dealing specifically with each melting atmosphere.

(1) Air Atmosphere.—The roll-over induction furnace used both for air and argon melting is shown in Fig. 4a. The mold was clamped on top of the furnace and the assembly rolled 180°. The position of the mold when partially rolled is shown in Fig. 4b.

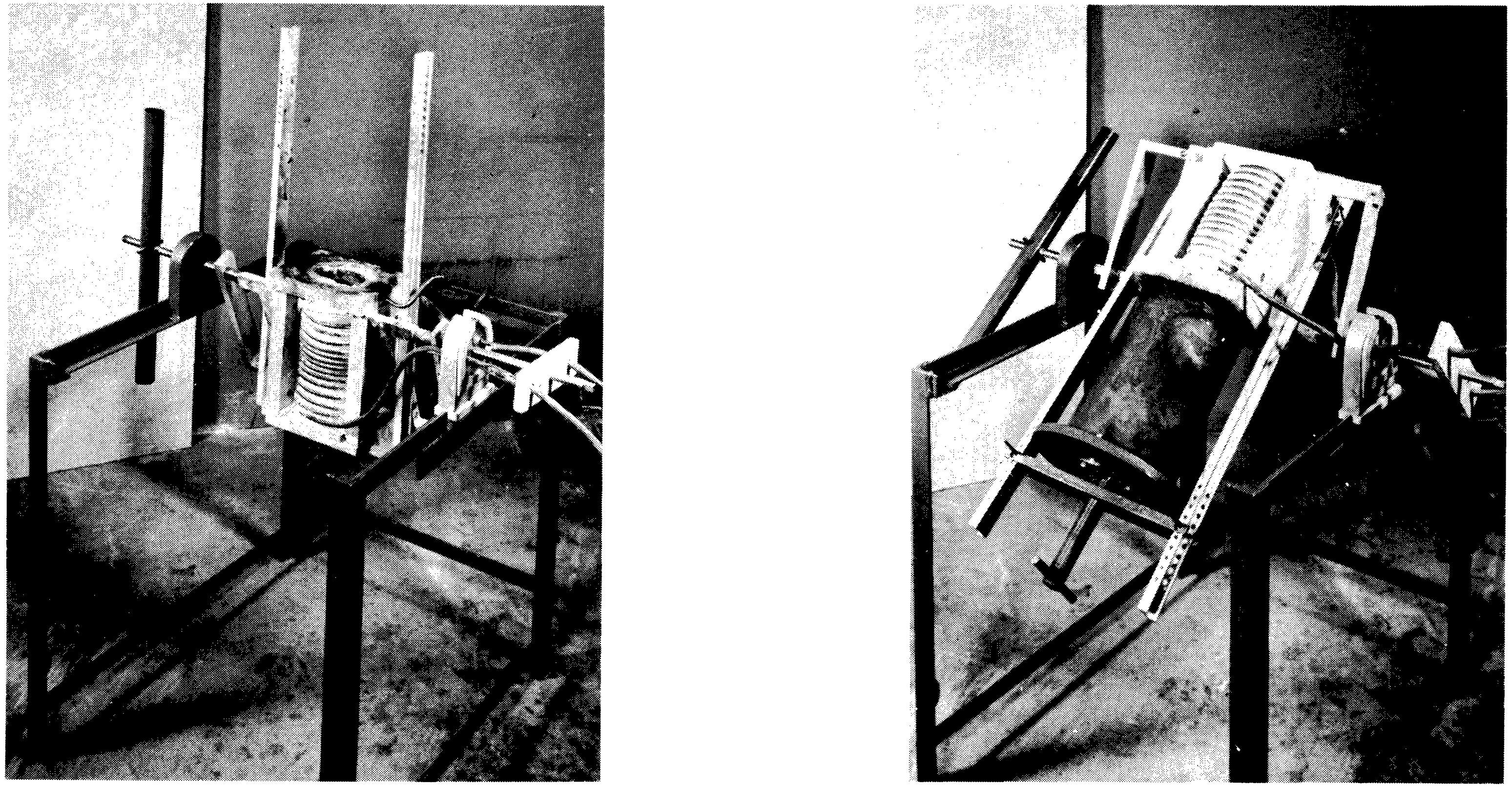
With air melting, the crucible was uncovered, allowing the melt to be exposed to the air. When the heat was poured, the mold was clamped above the furnace and argon pressure was used in pouring. A pressure of five psig was applied after the mold had been rolled approximately 90° of the 180° total.

(2) Argon-Protected Atmosphere.—The same procedure was followed in argon-protected melting as in air melting, except the melt surface was covered with a blanket of argon throughout the heat. The crucible was charged, covered with an asbestos gasket and refractory brick, and flushed with argon. A positive argon pressure was maintained within the crucible during melting. Argon pressure was again used for pouring.

(3) Vacuum + Argon Atmosphere.—The exterior of the vacuum melting unit and control panel is shown in Fig. 5a. The mold assembly within the shell, the crucible, addition buckets and dipper are shown in Fig. 5b. The pressure within the shell during actual melting was below five microns.

The crucible was charged and the buckets filled with the alloy additions in the order previously outlined. The cover was closed and a vacuum of five microns or below was drawn. At this point the power was applied to the crucible. After melt down, the heat was held for 20 minutes at approximately 2700°F to allow refining to take place. After refinement, the alloy additions were made.

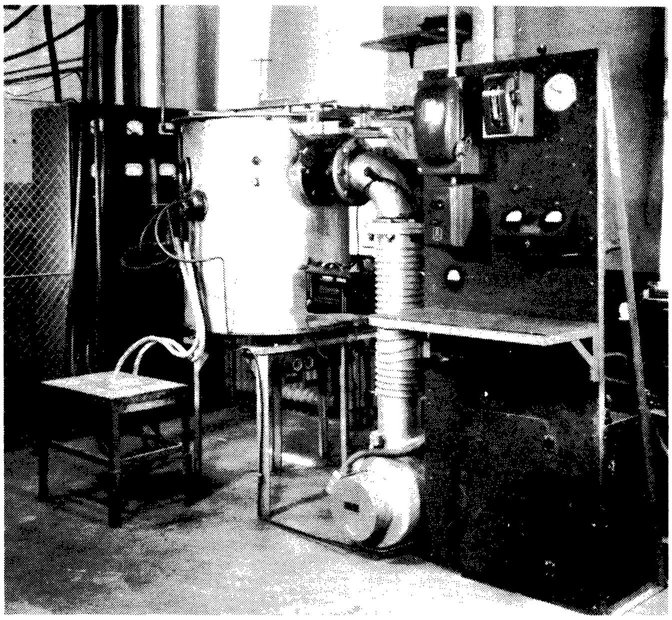
Since a considerable decrease in mold temperature would occur if the mold were placed in the vacuum shell at the outset of the heat, it was left in the



a. Rollover induction melting furnace used for air and argon-protected melts. Capacity, 10 lb.

b. Mold clamping and rollover procedure.

Fig. 4. Air and argon-protected melting equipment.



a. Exterior of vacuum melting unit, showing control panel, melting shell, and vacuum pumps.

b. Interior of vacuum shell, showing the induction furnace, mold, charging buckets, and dipper.

Fig. 5. Vacuum + argon melting equipment.

firing furnace until after the alloy additions were made. To protect the melt from air contamination while the mold was inserted, an atmosphere of argon was introduced into the shell and the melting crucible covered. After insertion of the mold, another vacuum was applied and the heat was remelted and heated to the pouring temperature. This entire operation required 20-30 minutes. Argon was bled in during pouring. Heats R-108, R-109, R-114, R-115, and R-160 were poured in this manner.

Even though the sprue temperature was apparently greater than 1200°F after this 20-30—minute period, the actual mold-cavity temperature was unknown. A cooling curve from a mold preheated at 1600°F showed that after 30 minutes the mold-cavity temperature dropped to 900°F. Cooling curves of molds heated at 1800°F indicated that eight minutes were available before the mold-cavity temperature fell to 1600°F.

To meet this time requirement, the melt was kept liquid during the introduction of argon. The power was applied immediately after the mold was inserted and the cover closed. The vacuum was applied to the shell after the power was turned on and a pressure of 30 microns obtained. Argon pressure of one atmosphere was applied during pouring, as in the previous case. Heats R-175 and R-176 were made while using this procedure and a mold temperature of 1600°F attained at pouring (eight minutes after mold withdrawal from the firing temperature of 1800°F).

c. Chemical Analyses.—The majority of the chemical analyses listed in this report were performed by the J. H. Herron Co. in Cleveland, Ohio. The chemical ranges of the elements and the percentages charged for the alloys are listed in Table IV.

Difficulties with Ti, Al, and B analyses arose early. Several checks of these elements were performed by the International Nickel Co.[6] These analyses checks are listed in Appendices I-III.

The validity of the low Al, Ti, and B analyses was questioned, especially with the charged percentages listed above. However, to check the effect of the analysis spread on the stress-rupture properties, the following analysis range was obtained by removing heats which greatly departed from nominal analyses.

Figures 6, 8, and 10 contain all the heats made for the three alloy types. Their wide analysis range is listed in Table IV. Figures 7, 9, and 11 contain the heats with the narrow analysis range listed in Table V.

The effect of the analysis spread upon 100-hour rupture strength is shown in Table VI.

TABLE IV

CHEMICAL ANALYSES—WIDE RANGE

Alloy Type	C	Cr	Mo	Al	Fe	Ti	B	Co	Cb
Guy charged	.10	13.5	7.0	7.0	4.5	---	.60	---	2.0
Range	.08-.26	12.83-15.35	4.42-5.87	4.33-7.31	4.36-6.94	---	.28-.48	---	1.52-2.30
Inco 700 charged	.10	15.5	3.2	3.5	.5	2.0	---	29	---
Range	.09-.34	14.79-15.89	2.10-3.32	1.32-3.4	.17-.99	1.30-3.3	---	27.14-30.5	---
GMR 235 charged	.15	15.5	5.0	3.5	10.0	2.0	.09	---	---
Range	.15-.27	15.03-15.99	4.31-5.39	2.37-4.66	7.65-11.10	1.70-2.30	.06-.12	---	---

TABLE V

CHEMICAL ANALYSES—NARROW RANGE

Alloy Type	C	Cr	Mo	Al	Fe	Ti	B	Co	Cb
Guy*	.10-.15	13.08-15.35	4.42-5.87	5.50-6.80	4.64-6.94	---	.37-.48	---	1.83-2.30
Inco 700**	.12-.16	15.03-15.60	2.10-3.03	2.90-3.52	.37-.69	1.65-2.10	---	27.7-29	---
GMR 235***	.15-.18	15.03-15.99	4.31-5.39	3.02-3.88	8.54-11.10	1.75-2.30	.06-.10	---	---

*Composed of heats R-98, R-119, R-88, R-98, and R-160.

**Composed of heats R-97, R-87, and R-109.

***Composed of heats R-116, R-50, R-84, R-108, and R-114.

	C	Cr	Mo	Al	Fe	B	Cb
Analysis Range	.08-.26	12.83-15.35	4.42-5.87	4.33-7.31	4.36-6.94	.28-48	1.52-2.30

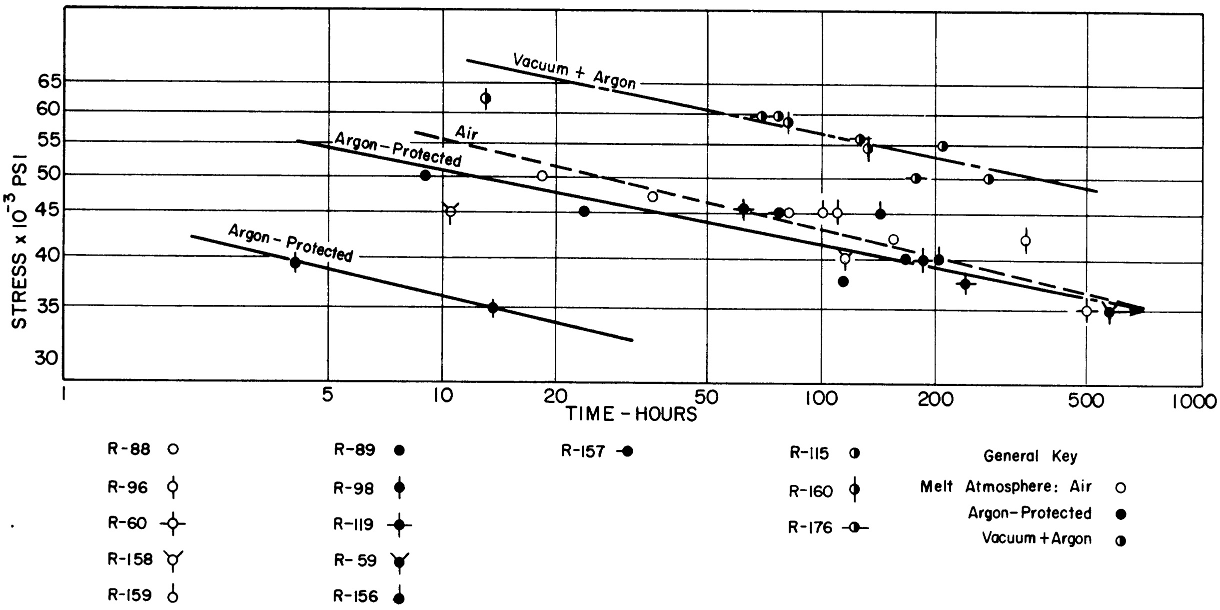


Fig. 6. Effect of melting and casting atmospheres upon stress-rupture properties of Guy-type alloy at 1500°F (wide analysis range).

	C	Cr	Mo	Al	Fe	B	Cb
Analysis Range	.10-.15	13.08-15.35	4.42-5.87	5.50-6.80	4.64-6.94	.37-48	1.83-2.30

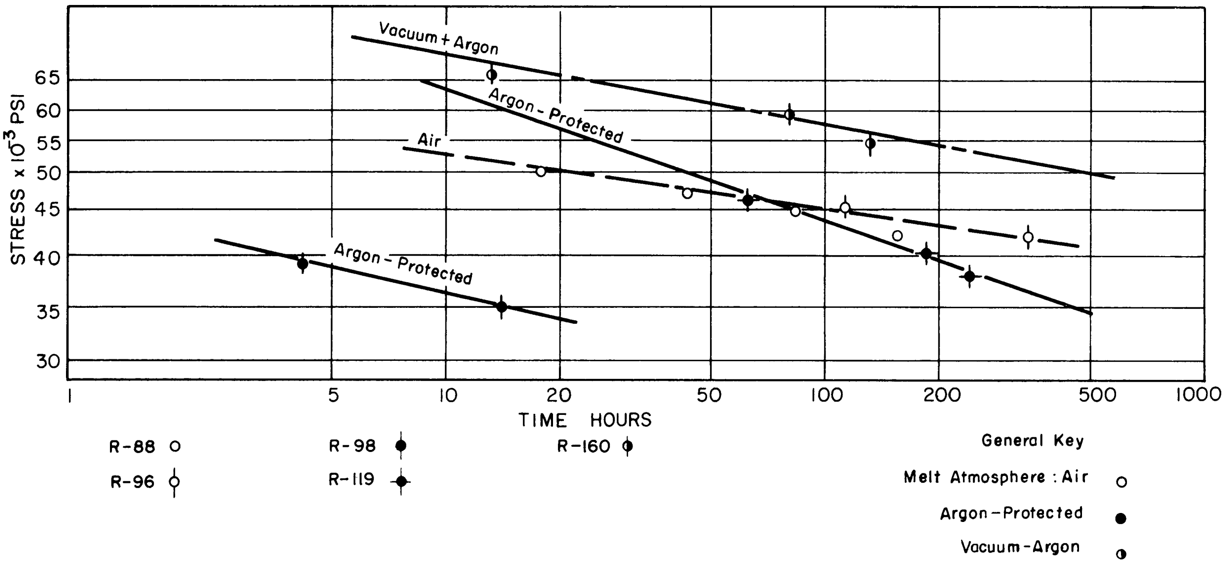


Fig. 7. Effect of melting and casting atmospheres upon stress-rupture properties of Guy-type alloy at 1500°F (narrow analysis range).

	C	Cr	Mo	Al	Fe	Ti	Co
Analysis Range	.09-.34	14.79-15.89	2.10-3.32	1.32-3.40	.17-.99	1.30-3.30	27.14-30.5

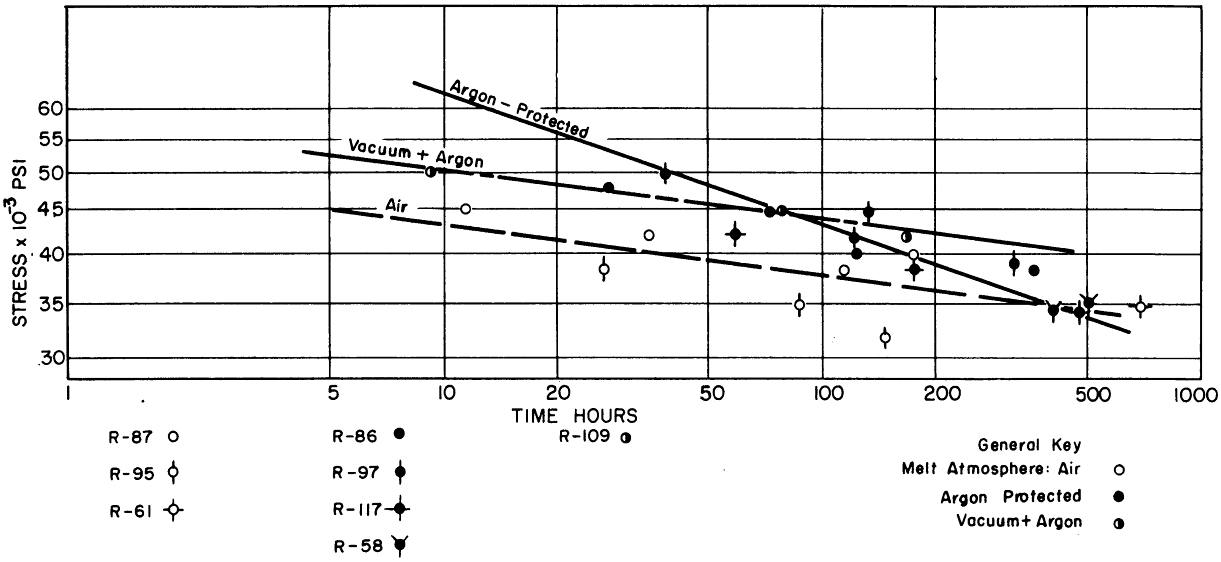


Fig. 8. Effect of melting and casting atmospheres upon stress-rupture properties of Inco-700-type alloy at 1500°F (wide analysis range).

	C	Cr	Mo	Al	Fe	Ti	Co
Analysis Range	.12-.16	15.03-15.60	2.10-3.03	2.90-3.52	.37-.69	1.65-2.10	27.7-29.0

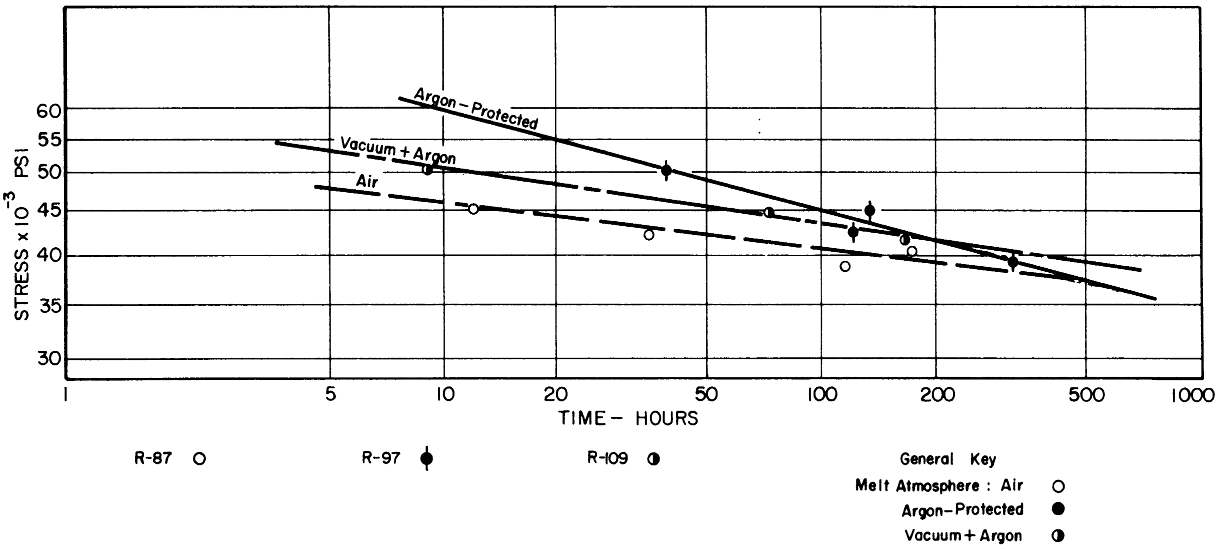


Fig. 9. Effect of melting and casting atmospheres upon stress-rupture properties of Inco-700-type alloy at 1500°F (narrow analysis range).

	C	Cr	Mo	Al	Fe	Ti	B
Analysis Range	.15-.27	15.03-15.99	4.31-5.39	2.37-4.66	7.65-11.10	1.90-2.30	.06-.12

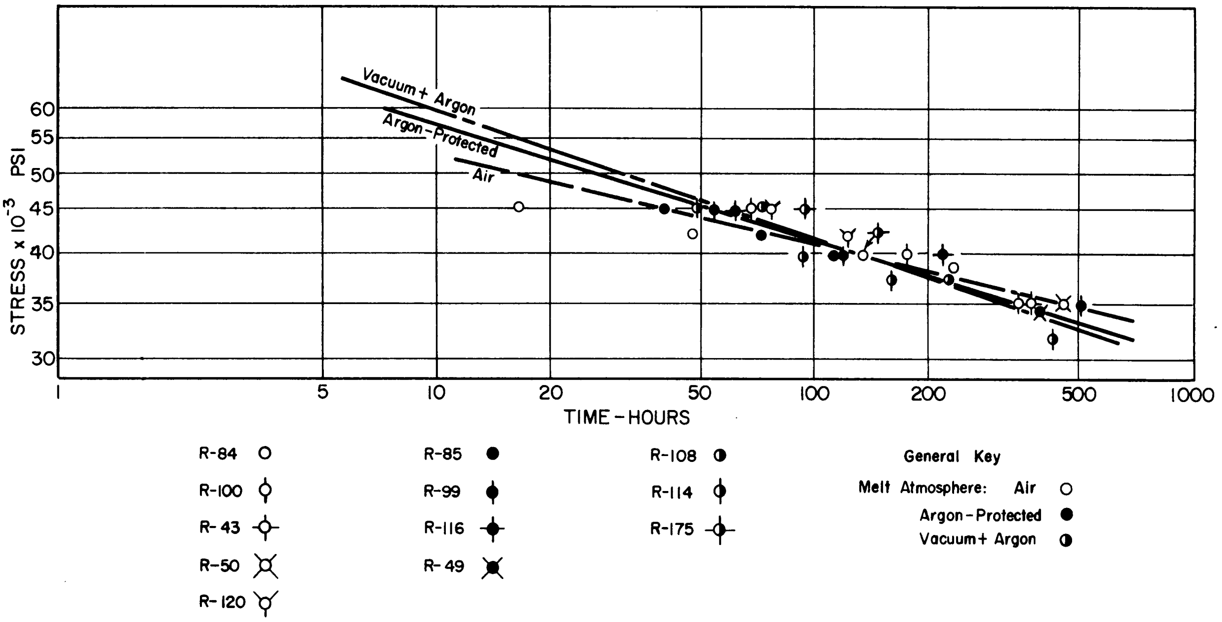


Fig. 10. Effect of melting and casting atmospheres upon stress-rupture properties of GMR-235-type alloy at 1500°F (wide analysis range).

	C	Cr	Mo	Al	Fe	Ti	B
Analysis Range	.15-.13	15.03-15.99	4.31-5.39	3.02-3.88	8.54-11.10	1.75-2.30	.06-.10

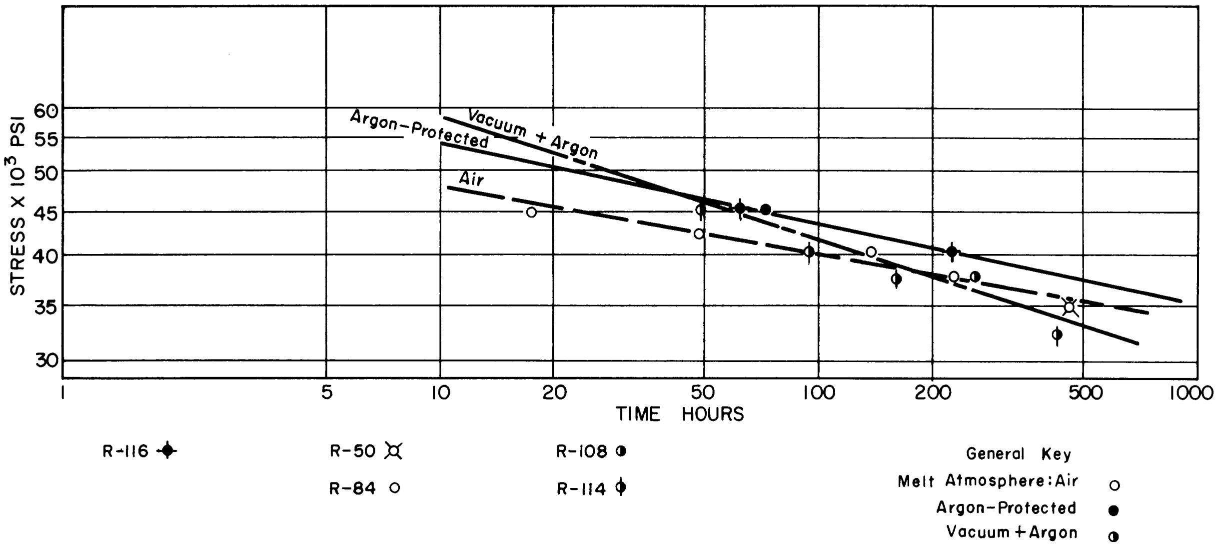


Fig. 11. Effect of melting and casting atmospheres upon stress-rupture properties of GMR-235-type alloy at 1500°F (narrow analysis range).

TABLE VI

ANALYSIS RANGE VS 100-HOUR RUPTURE LIFE

Alloy Type	Analysis Range	100-Hour Rupture Life Air	 Argon-Protected	 Vacuum + Argon
Guy	wide	43,000	41,000	57,000
	narrow	45,000	44,000	58,000
Inco 700	wide	37,000	43,000	44,000
	narrow	41,000	45,000	44,000
GMR 235	wide	41,000	42,000	42,000
	narrow	41,000	43,000	42,000

It is evident from the above table that compositional variations, to the extent given in this report, have little effect upon 100-hour rupture strengths of the three alloys. Some small changes occur in the slopes of the stress-rupture lines.

d. Mechanical Testing.—To evaluate the effect of melting and casting atmospheres upon ambient- and elevated-temperature properties, the following were used as indices:

Ambient-Temperature Properties	Elevated-Temperature Properties
(1) Tensile strength	(1) Stress-rupture strengths at varying rupture times
(2) Yield strength (.2% offset)	(2) Percent elongation
(3) R_c hardness	(3) Percent reduction of area
(4) Percent elongation	
(5) Percent reduction of area	

Before testing, each specimen was radiographed and visually inspected for any flaws. Only specimens which were radiographically sound and which possessed good surface quality were submitted.

The room-temperature and stress-rupture tests were performed at The University of Michigan by the staff of Prof. J. W. Freeman, using well-established techniques. The general procedure for room-temperature testing was as follows:

(1) the load was applied hydraulically
(2) strain measurements were recorded by a Martens-type extensometer system.

Stress-rupture testing was conducted as follows:

(1) three covered thermocouples were tied to the specimen along its gage length

(2) the specimen was placed in a cold furnace and brought to temperature at no load

(3) the specimen was loaded when the temperature distribution along the gage length was within ± 5°F (at 1500°F).

e. Metallography.—All metallographic specimens were taken transversely from the center of the gage length. The specimens were mounted in bakelite, ground on dry emery papers, and polished on a diamond lap. Electrolytic etching was performed in a solution composed of 5% hydrofluoric acid, 10% glycerine, and 85% absolute alcohol.

Vacuum-fusion analyses of representative heats of the three alloys melted and cast under the three atmospheres were performed with the National Research Vacuum-Fusion Apparatus at the University. Nitrogen, hydrogen, and oxygen contents were determined.

DATA AND DISCUSSION OF RESULTS

The effects of melting and casting atmospheres upon the properties of the Guy-, Inco-700-, and GMR-235 types of alloys are illustrated in Figs. 6-11 and Appendices I-IV.

In the following section the results for each alloy will first be discussed separately, followed by a general comparison. The discussion covers only the elevated-temperature properties because of their importance in design variation with atmosphere. The ambient-temperature properties for all alloys are summarized in Appendix IV. No atmosphere effect is evident.

1. GUY-TYPE ALLOY

The effect of melting atmospheres upon the stress-rupture properties of this alloy is shown in Figs. 6 and 7 and Appendix I. In the case of the Guy-type, the elevated-temperature elongation and strength level are raised considerably by vacuum + argon melting, as shown in Table VII.

A separate curve is drawn for the argon-protected heat, R-98 in Figs. 6 and 7 because large amounts of a foreign phase appeared in the microstructure. This foreign "white phase" is illustrated in Fig 12c, and minor

TABLE VII

EFFECT OF MELTING AND CASTING ATMOSPHERES UPON 100-HOUR RUPTURE STRENGTH AND ELONGATION OF GUY-TYPE ALLOY AT 1500°F

Atmosphere	100-Hour Rupture Strength psi	Percent Elongation at 100-hour Rupture
Air	42,000	3
Argon-protected	41,000	1-3
Vacuum + argon	56,000	7-10

amounts were also observed in the following heats only: R-119 (5%), R-60 (5%), and R-89 (1%). In general, these heats exhibit stress-rupture properties below average for their specific melting conditions. A survey of the analyses does not indicate any correlation with chemical composition. Apparently, the appearance of this phase is undesirable for high-temperature strength.

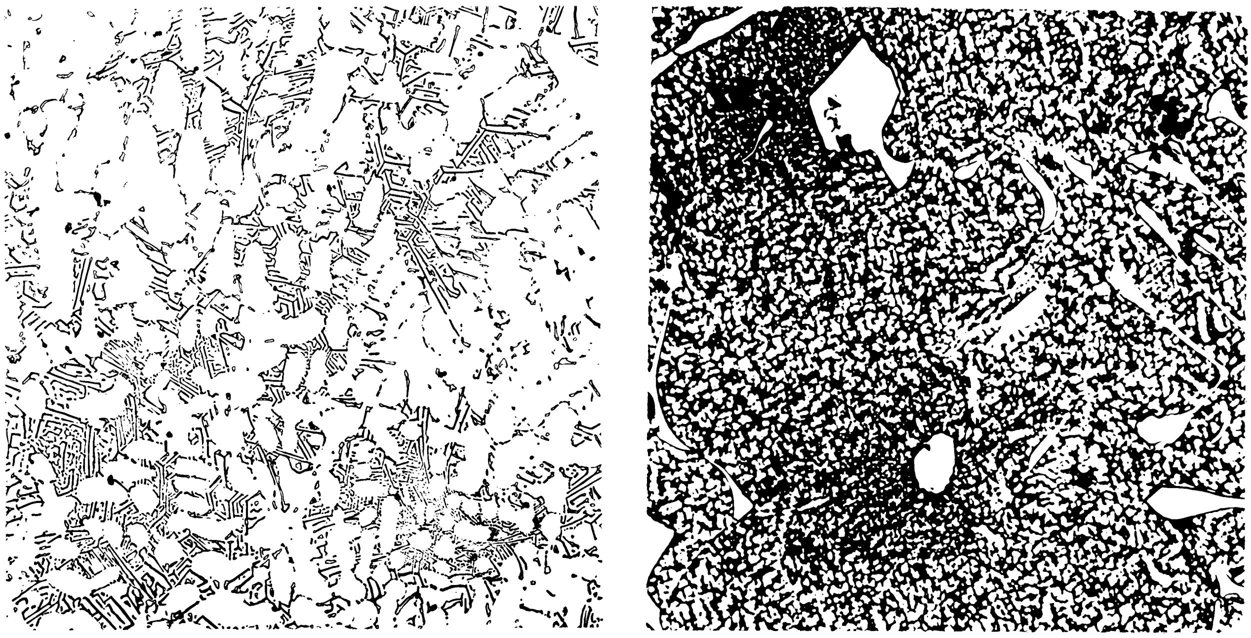
No optical microstructural changes are evident in the vacuum + argon melted heats to explain the increase in rupture strength or elongation. The structure of this alloy is shown in Fig. 12a. General precipitation occurs throughout the matrix, as shown in Fig. 12b.

Vacuum-Fusion Analyses—Nitrogen Effect

Vacuum + argon melting and casting decrease the amount of nitrogen in the metal compared with air or argon-protected atmospheres, as shown in Table VIII. Apparently, nitrogen may be involved in explaining the marked increases in elongation and strength at 1500°F.

The difference in nitrogen evolved at the 1550°C and 1850°C temperatures is of interest. Since 1850°C is necessary to decompose TiN or CbN, the difference between the 1850° and 1550°C analyses may indicate the nitrogen combined in this manner. It is interesting to note that the ratio of nitrogen evolved at 1550°C to that evolved at 1850°C is comparable for all atmospheres.

Work is in progress to investigate the effect of deliberate nitrogen additions to vacuum-melted material.



100X

a. General structure of Guy-type alloy.
Heat R-88: air atmosphere.

1000X

b. Matrix structure of Guy-type alloy.
Heat R-88: air atmosphere.
Fine precipitate in matrix is $Ni_3(AlTi)$.

500X

c. Unidentified white phase in low-strength Guy-type alloy.
Heat R-98: argon-protected atmosphere.

Fig. 12. Microstructures of cast Guy-type alloy; electrolytic etch, 5% HF, 10% glycerine, and 85% alcohol (specimens from center of test-bar gage length).

TABLE VIII

VACUUM-FUSION ANALYSES FOR NITROGEN

Alloy Type	Heat No.	Melting and Casting Atmosphere	Crucible Temperature		Percent N_2 at 1850°C minus Percent N_2 at 1550°C	Percent of 1850°C Nitrogen Evolved at 1550°C
			1550°C	1850°C		
Guy	R-96	Air	.0442	.0154	?	
	R-98	Argon-protected	.0098	----		
	R-115	Vacuum + argon	.0010	.0021	.0011	52.
	R-160	Vacuum + argon	.0029	----		
	R-176	Vacuum + argon	----	.0036		
Inco 700	R-95	Air	.0108	----		
	R-117	Argon-protected	.0045	.0113	.0068	60.
	R-109	Vacuum + argon	.0010	.0018	.0008	44.
GMR 235	R-120	Air	.0043	.0258	.0215	83.
	R-99	Argon-protected	.0053	----		
	R-108	Vacuum + argon	.0006	.0025	.0019	76.
	R-175	Vacuum + argon	----	.0041		

Oxygen was .0003-.02% and hydrogen .00008-.0004% at 1850°C. No consistent variation with type of melting was obtained. The role of dross in the case of oxygen and of storage time of the samples in the case of hydrogen can affect the results seriously.

2. INCO-700-TYPE ALLOY

The effect of melting atmospheres upon the elevated temperature properties of this alloy is shown in Figs. 8 and 9 and in Appendix II.

Vacuum + argon melting and casting atmospheres exert a significant effect upon elevated-temperature ductility and improve strength somewhat as shown in Table IX.

TABLE IX

EFFECT OF MELTING AND CASTING ATMOSPHERES UPON 100-HOUR RUPTURE STRENGTH AND ELONGATION OF INCO-700-TYPE ALLOY AT 1500°F

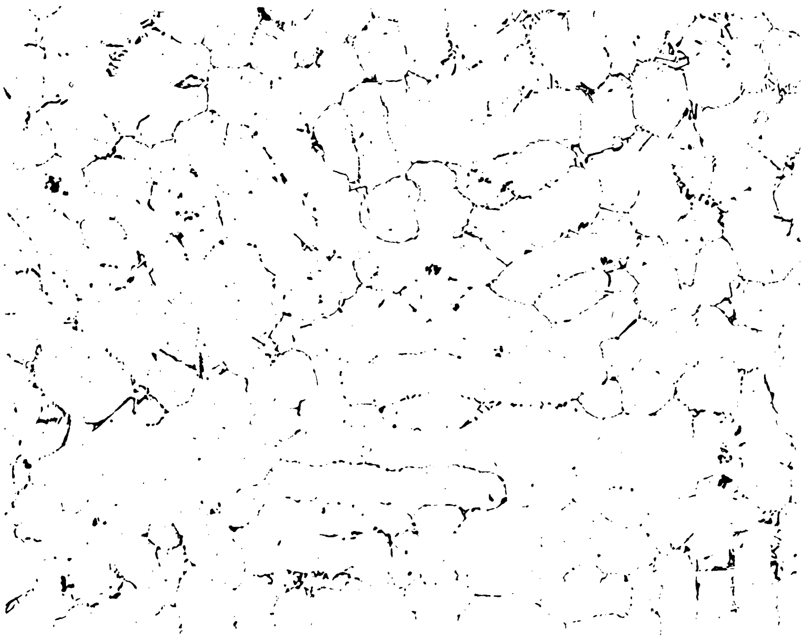
Atmosphere	100-Hour Rupture Strength psi	Percent Elongation Range at 100 Hours
Air	37,000	2-4
Argon-protected	42,000	3-4
Vacuum + argon	42,000	21

Optical microstructural examinations give no clues to the reasons for strength or ductility variations with melting and casting atmospheres. The general cast microstructure of the Inco-700 type is shown in Fig. 13a. Figure 13b shows in detail the primary precipitate. This precipitate is probably chromium carbide with dissolved molybdenum and titanium.

Figure 14a shows the structure of this alloy after testing at 1500°F. This precipitation around existing phases is typical of all air and argon heats. Figure 14b shows the structure of the vacuum + argon melted Inco-700-type alloy after testing. A radical change has occurred as a result of testing.

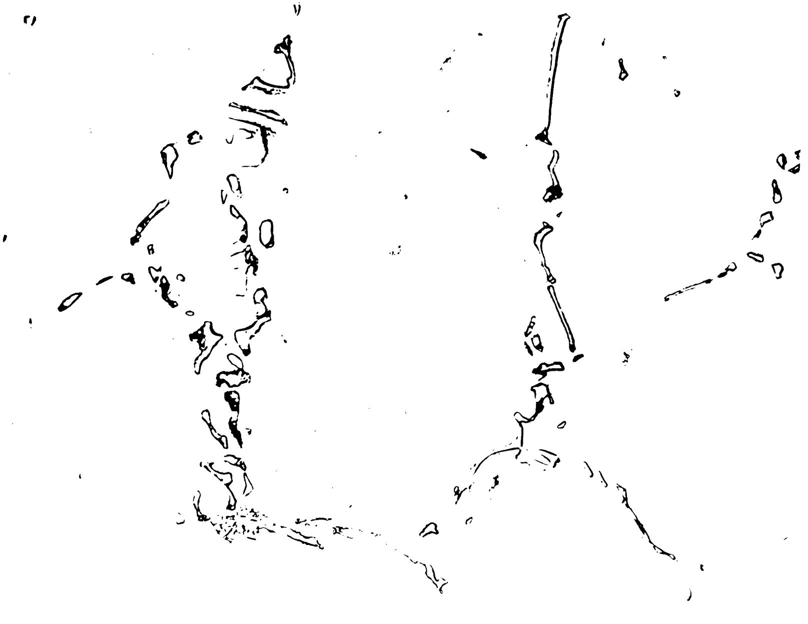
Vacuum-Fusion Analyses—Nitrogen Effect

The vacuum-fusion analyses for Inco-700-type alloys shown in Table VIII show that vacuum + argon melting produces the lowest nitrogen content.



100X

a. General structure of cast Inco-700-type alloy.
Heat R-86: argon-protected atmosphere.



500X

b. Detail of precipitate in cast Inco-700-type alloy.
Heat R-86: argon-protected atmosphere.

Fig. 13. Microstructures of cast Inco-700-type alloy; electrolytic etch, 5%HF, 10% glycerine, and 85% alcohol (specimens from center of test-bar gage length).

500X

a. Precipitation around primary precipitate after testing. Typical of heats melted under air and argon-protected atmospheres. Heat R-86: argon-protected atmosphere.

500X

b. Structure of vacuum + argon melted Inco-700-type alloy after testing. Heat R-109: vacuum + argon atmosphere.

Fig. 14. Microstructures of Inco-700-type alloy after testing at 1500°F; electrolytic etch, 5% HF, 10% glycerine, and 85% alcohol (specimens from center of test-bar gage length).

3. GMR-235-TYPE ALLOY

Elongation at 1500°F is strongly affected by melting and casting atmospheres, as indicated in Table X and in Figs. 10 and 11 and Appendix III.

TABLE X

EFFECT OF MELTING AND CASTING ATMOSPHERES UPON 100-HOUR RUPTURE STRENGTH AND ELONGATION OF GMR-235-TYPE ALLOY AT 1500°F

Atmosphere	100-Hour Rupture Strength psi	Percent Elongation Range at 100 Hours
Air	41,000	6-9
Argon-protected	41,000	3-7
Vacuum + argon	42,000	14-19

Examination using the light microscope does not explain variations. Figure 15a shows the general microstructure of this alloy. Inclusion clusters are evident. At higher magnifications (Fig. 15b), some of the primary precipitate appears as a eutectic. General precipitation within the matrix is obvious in this figure.

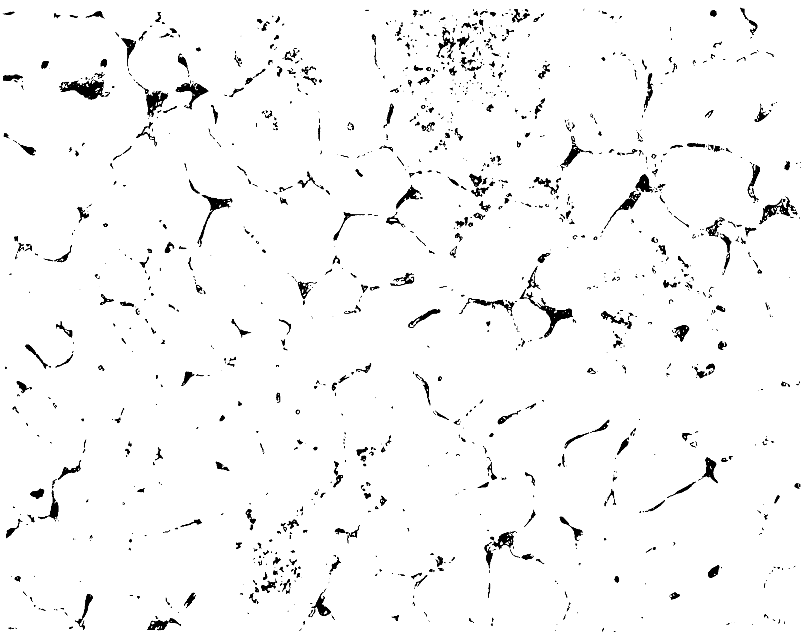
The eutectic is apparently Ni-B or Ni-B-C, since in the Inco-700-type alloy, which has similar chemistry except for boron, no eutectic appears. A similar eutectic also appears in boron steels. The inclusion clusters are titanium carbonitrides.[7] In Fig. 15b, general precipitation is evident in the matrix. This phase is $Ni_3(AlTi)$.[8]

Vacuum-Fusion Analyses—Nitrogen Effect

The vacuum-fusion analyses for the GMR-235-type alloy, shown in Table VIII, indicate that the increase in elongation at 1500°F by vacuum + argon melting could result from differences in gas content.

GENERAL COMPARISON

The data just discussed may be summarized best by Table XI.



100X

a. General structure of cast GMR-235-type alloy.
Heat R-85: argon protected atmosphere.
Clusters of inclusions are TiN + TiC.
Eutectic is Ni-B-C.

1000X

b. Detail of matrix, inclusion cluster and eutectic.
Heat R-85: argon-protected atmosphere.
Precipitate in matrix is $Ni_3(AlTi)$.

Fig. 15. Microstructures of GMR-235-type alloy; electrolytic etch, 5% HF, 10% glycerine, and 85% alcohol (specimens from center of test-bar gage length).

TABLE XI

SUMMARY OF STRESS-RUPTURE PROPERTIES OF THE THREE ALLOYS

Alloy Type		Published Properties	This Investigation		
			Atmosphere		
			Air	Argon-Protected	Vacuum + Argon
Guy	100-hour rupture strength (psi)	49,000[1]	42,000	41,000	56,000
	Percent elongation (100 hours)	2-5[1]	3	1-3	7-10
Inco 700	100-hour rupture strength (psi)	*43,000[4]	37,000	43,000	42,000
	Percent elongation (100 hours)	10*	2-4	3-4	21
GMR 235	100-hour rupture strength (psi)	37-40,000[5]	41,000	41,000	42,000
	Percent elongation (100 hours)	6-10	6-9	3-7	14-19

*Solution treated and aged.

CONCLUSIONS

1. In all three alloy types, melting under vacuum and casting under argon atmospheres improves ductility two- to tenfold under stress-rupture test conditions at 1500°F.

2. Stress-rupture strength at 1500°F is improved in the Guy-type alloy by 38% and in the Inco-700 type by 13% with the argon + vacuum melting technique compared with air melting. No change occurs in the GMR-235 type.

3. The air-melted and argon-protected heats contain from five to ten times the nitrogen of the vacuum + argon heats.

FUTURE WORK

Additional research may profitably include the following:

1. Investigation of the effect of pouring temperature and pressure upon elevated-temperature properties of the three alloys.
2. Effect of grain size upon ambient- and elevated-temperature properties.
3. Effect of nucleation upon elevated-temperature properties of the three alloys.
4. Effect of nitrogen additions to vacuum-melted, Guy-type alloy to determine the atmosphere effect.
5. Evaluation of properties after solution treatment and aging to produce uniform structures.
6. Exploration of the effect of higher stress-rupture test temperatures.
7. Examination of vacuum- and air-melted heats with the electron microscope to explore differences in structure.

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APPENDIX I

EFFECT OF ATMOSPHERE ON STRESS-RUPTURE PROPERTIES OF GUY-TYPE ALLOY AT 1500°F

Heat	Atm	Chemical Composition(1)									R_C(10)		Stress	Rupture Life (Hours)	Percent Elongation(2)	Percent R.A.(3)
		C	Cr	Mo	Al	Fe	Ti	B	Co	Cb	B.T.	A.T.				
R-59	Argon	.26	14.04	4.78	6.12	5.96	-	.32	-	1.99	38.5	31	35,000	643(4)	-	-
													35,000	508(4)(5)	-	-
													35,000	547(4)	-	-
													35,000	383	2	2
R-89	Argon	.13	14.63	5.3	7.31	4.51	-	.31	-	1.89	36	40	50,000	9.0	2	4
													45,000	24(6)	1	-
													37,000	115	1	3
R-98(8)	Argon	.12	15.35	5.50	6.14	5.45	-	.44	3.68	2.22	43	44	39,000	4.3	2	2
													35,000	14	2	4
R-119(8)	Argon	.11	13.08	4.42	5.77	6.94	-	.38	-	2.30	39	31.5	46,000	62	3	4
													40,000	188	4	5
													37,000	240	3	5
R-60	Air	.26	13.75	4.99	4.33	5.91	-	.30	-	1.52	37	30	35,000	544	5	6
													35,000	485	5	7
													35,000	511	6	9
													35,000	189(6)	4	4
R-88	Air	.12	13.99	5.87	6.80	4.64	-	.42	-	1.97	38.5	33	50,000	18	< 1	< 1
													47,000	43	2	2
													45,000	83(7)	3	3
													42,000	155	3	4
R-96(8)	Air	.15	14.17	4.84	5.91	4.86	-	.37	4.24	1.83	42	29	45,000	.33(6)	1	3
													45,000	111	3	4
													42,000	345	4	8
R-115	Vacuum	.20	12.83	5.54	6.54 6.42(9)	5.40	-	.35 .60(9)	-	1.96	-	-	60,000	77	7	17
													55,000	210	6	9
													50,000	273(6)	9.0	15
R-160	Vacuum	.10	13.44	5.56	5.50	5.06	-	.48	-	2.06	-	-	65,000	13.2	3.0	6.5
													58,000	80.5	10	15
													54,000	132	8	11
R-176*	Vacuum										-	-	60,000	70	4.0	5.0
													55,000	131	7.0	8.0
													50,000	178	5.0	6.5
R-156*	Argon												40,000	199	3	3
													45,000	145	1.0	1.0
R-157*	Argon												45,000	79	2	2.5
													40,000	168	2.0	2.5
R-158*	Air												45,000	10.2(6)	-	-
													40,000	117	3	4
R-159	Air	.08	13.10	5.71	6.04	4.36	-	.28	-	1.86			45,000	102	2	1.5

(1) Balance Ni.
(2) Percent elongation in one inch measured after fracture.
(3) Percent R.A. measured after fracture.
(4) Test discontinued after stated number of hours.
(5) Overheated to 1800°F after 508 hours.
(6) Casting defect in fracture.
(7) Broke in fillet.
(8) Ti and Al analyzed by specifications in the Analysis Manual of the International Nickel Co.[6]
(9) Analysis checks performed by the International Nickel Co.
(10) B.T., A.T. - before testing, after testing.
* Analyses not completed.

APPENDIX II

EFFECT OF ATMOSPHERE ON STRESS RUPTURE PROPERTIES OF INCO-700-TYPE ALLOY AT 1500°F

Heat	Atm	Chemical Composition (1)									Rc(9)		Stress	Rupture Life (Hours)	Percent Elongation(2)	Percent R.A.(3)
		C	Cr	Mo	Al	Fe	Ti	B	Co	Cb	B.T.	A.T.				
R-58	Argon	.34	15.89	2.48	2.56	.60	3.11	-	28.48	-	38.5	31	35,000 35,000 35,000 35,000	408 544(4) 474 500(4)	4 - 4 -	5 - 3 -
R-86	Argon	.27	15.18	3.05	3.4	.17	1.55	-	2[illegible]	-	32.5	28	47,500 45,000 40,000 38,000	27 73 123 261	3 3 3 3	6 3 8 4
R-97	Argon	.12	15.50	2.49	1.32 3.52(8)	.69	1.30 2.06(8)	-	28.50	-	31	29	50,000 45,000 42,000 39,000	39 132 122 319	3 4 3 5	6 7 2 7
R-117(7)	Argon	.13	15.32	3.32	2.13	.99	2.11	-	30.02	-	-	34.5	42,000 38,000 34,000	60 175 480	3 3 3	2 7 5
R-61	Air	.23	14.79	2.61	2.12	.60	3.3	-	27.14	-	31	33	35,000 35,000 35,000 35,000	571(4) 545(4) 523(4) 693	- - - 7	- - - 18
R-87	Air	.16	15.6	3.03	2.90	.37	1.65	-	27.7	-	31	30	45,000 42,000 40,000 38,000	12 35 173 114(5)	4 2 2 -	3 3 3 -
R-95	Air	.09	15.35	2.49	1.49	.69	1.55	-	30.50	-	27	31	41,000 38,000 35,000 32,000	8(6) 32 88 147	3 2 2 4	4 3 2 5
R-109	Vacuum	.15	15.03	2.10	1.32 3.17(8)	.60	1.73 2.10(8)	-	29.0	-	33	37	50,000 45,000 41,000	9 73 166	24 21 4	48 25 4

(1) Balance Ni.
(2) Percent elongation in one inch measured after fracture.
(3) Percent R.A. measured after fracture.
(4) Test discontinued after stated number of hours.
(5) Inclusion in fracture.
(6) Probable defect in fracture.
(7) Ti and Al analyzed by specifications in the Analysis Manual of the International Nickel Co.[6]
(8) Analysis checks performed by the International Nickel Co.
(9) B.T., A.T. - before testing, after testing.

APPENDIX III

EFFECT OF ATMOSPHERE ON STRESS RUPTURE PROPERTIES OF GMR-235-TYPE ALLOY AT 1500°F

Heat	Atm	Chemical Composition(1)									R_c(11)		Stress	Rupture Life (Hours)	Percent Elongation(2)	Percent R.A.(3)
		C	Cr	Mo	Al	Fe	Ti	B	Co	Cb	B.T.	A.T.				
R-49	Argon	.18	15.33	5.10	2.89	8.37	2.26	.11			30	29	35,000 35,000 35,000	366 177 382	6 5 6	9 11 9
R-85	Argon	.16	15.18	4.83	4.66	8.45	1.70	.11			-	25	45,000 42,000 40,000 38,000	40 72 113(4) 13(5)	4 5 7 -	4 10 13 -
R-99(9)	Argon	.17	15.27	4.98	2.95 3.62(10)	10.91	1.90	.08 .12(10)			32	34.5	45,000 40,000 35,000	55 119 510	6 3 18	14 8 21
R-116(9)	Argon	.17	15.43	4.31	3.24	10.51	2.17	.07	-	-	32	33	45,000 40,000	61 223	6 11	11 11
R-43	Air	.17	15.34	4.71	3.12	7.65	2.26	.12	-	-	30	-	35,000 35,000 35,000	397 317 384	9 10 13	14 16 15
R-50	Air	.18	15.99	5.39	3.34	8.54	2.27	.08	-	-	31	31.5	35,000 35,000 35,000 35,000	516 460 399 456	7 5 7 5	14 8 8 14
R-84	Air	.16	15.64	4.46	3.88	10.44	1.75	.10	-	-	-	-	45,000 42,000 40,000 36,000	17 49(6) 137 .5(7)	1 2 6 1	4 8 13 2
R-100(9)	Air	.15	15.11	5.06	2.61	11.01	1.90	.07	-	-	32	27	45,000 40,000 36,000	68 177 347	7 9 11	15 15 21
R-120(9)	Air	.27	15.43	4.38	2.37	10.41	2.05	.06			32.8	32.5	45,000 42,000 38,000	78 122 233	7 9 3	11 13 8
R-108(9)	Vacuum	.16	15.03	5.26	3.01 3.02(10)	10.91	2.15	.06 .066(10)			30.5	30	45,000 37,200	72 260(8)	19 19	22 31
R-114	Vacuum	.15	14.98	5.06	3.76	11.10	2.30	.06	-	-	26	32	45,000 40,000 37,000 32,000	49 94 160 429	18 14 12 13	25 40 27 25
R-175	Vacuum										-	-	40,000 45,000	136 96.5	13 17	24 29

(1) Balance Ni.
(2) Percent elongation in one inch measured after fracture.
(3) Percent R.A. measured after fracture.
(4) Broke in gage mark.
(5) Blow hole in fracture.
(6) Broke near fillet.
(7) Fracture shows casting defect.
(8) Stripped out of adapter after 175 hours, brought back up under load.
(9) Ti and Al analyzed by specifications in the Analysis Manual of the International Nickel Co.[6]
(10) Analysis checks performed by the International Nickel Co.
(11) B.T., A.T. - before testing, after testing.

APPENDIX IV

AMBIENT-TEMPERATURE PROPERTIES OF GMR-235-, INCO-700-, AND GUY-TYPE ALLOYS

Alloy Type	Heat	Atm	R_c (1)	Tensile Strength	Yield Strength (4)	Percent Elongation	Percent R.A.
GMR-235	R-10	Argon	33	114,000	98,000	4	5.5
	R-14	Argon	-	96,300	96,300 (3)	2	0
	R-99	Argon	32	116,000	99,000	3	5
	R-116	Argon	32	122,300	-	4	4
	R-120	Air	33	110,000	97,000	2	3.5
	R-100	Air	32	100,000	100,000 (3)	2	2.5
	R-108	Vacuum	31	106,000	95,500	3	6
	R-114	Vacuum	26	97,000	85,000	3	6
Inco-700	R-117	Argon	-	91,700	-	5	8
	R-58	Argon	31	118,000	104,500	4	4
	R-86	Argon	33	111,000	102,000	3	4
	R-97	Argon	31	110,500	99,000	4	8.5
	R-61	Air	31	90,500	90,000	2	7.5
	R-95	Air	27	106,000	97,000	4	9
	R-109	Vacuum	33	109,000	95,000	6	13
Guy Type	R-98	Argon	43	104,000	104,000 (3)	1	-
	R-119	Argon	39	127,000	127,000 (3)	1	-
	R-88	Air	39	106,800 (2)	106,800 (3)	-	-
	R-96	Air	42	130,100	122,000	2	1
	R-115	Vacuum	-	138,000	118,000	2	1

(1) Average of three readings.

(2) Defect in fracture.

(3) Tensile strength same as yield strength.

(4) .2 percent offset.