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First Quarterly Technical Narrative Report
August 25 Through November 30, 1980

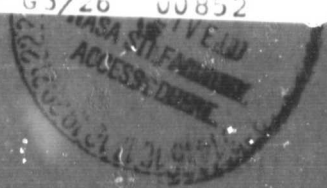
CAST Fe-BASE CYLINDER/REGENERATOR HOUSING ALLOY

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December 10, 1980

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Prepared by:

F. Larson
L. Kindlimann ✓

Prepared for

NASA - Lewis Research Center
Cleveland, OH 44135

Contract No. DEN 3-234 ✓



AIRESEARCH CASTING COMPANY
TORRANCE, CALIFORNIA

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1. INTRODUCTION

The objective of this program is the development of an iron-base alloy that can meet the requirements of automotive Stirling engine cylinders and regenerator housings. Specific goals are:

Cast alloy

Stress for 5000-hr rupture life of 200 MPa (29 ksi) at 775°C (1427°F)

Oxidation/corrosion resistance comparable to that of N-155

Compatibility with hydrogen

Alloy cost less than or equal to that of 19-9DL

To meet program requirements, the following tasks were established:

Task I, Selection of Alloy Approach, Preliminary Screening Study, Evaluation of Ten Alloys (Selection of Three Alloys).

Task II, Casting Optimization (Selection of One Alloy)

Task III, Establishment of Preliminary Data Base

Task IV, Reporting



2. TECHNICAL PROGRESS SUMMARY

In the initial three-month period of the program, the alloy approach has been selected and work has proceeded on the preliminary screening study.

2.1 TASK 1a, SELECTION OF ALLOY APPROACH

2.1.1 Literature Review

In this section, the results of the historical search are presented. The search was keyed primarily to the effects of alloying elements on high temperature strength of iron-based materials, with secondary emphasis on high temperature environmental resistance, i.e., oxidation/hot corrosion, and hydrogen embrittlement. Computer searches were conducted by Defense Technical Information Center (DTIC), NASA, and Savage Information Services in Rancho Palos Verdes, California (Covering Metadex, Chemical Abstracts, Scisearch, Weldasearch, Compendex, and NTIS). A total of 1035 citations were generated, many with abstracts, from which approximately forty were selected for complete review. In addition, personal discussions were conducted with Dr. David Sponseller of Climax Molybdenum Co., Dr. George Aggen of Allegheny Ludlum Steel Co., and Mr. Fred Hagen of Chrysler Corporation.

Since the primary goal is rupture strength (5000-hr life at 775°C under a 200 MPa stress), this review will concentrate on alloying for strength, and will touch just briefly on environmental resistance. Additional information on oxidation, hot corrosion, and hydrogen embrittlement will be presented in subsequent quarterly reports as these areas are investigated and test results are discussed. In particular, this document will concentrate on developing the basis for selection of the starting chemistries of materials for the preliminary stress rupture screening study.

2.1.2 Alloy Design Philosophy

There are literally reams of literature on the philosophy and techniques of designing metal alloys for high-temperature service. One can, however, readily reduce these to a few basic concepts: solid-solution strengthening, precipitation strengthening, composites (dispersion-strengthened, fiber-reinforced, etc.) and combinations of any of these techniques. For a low-cost application requiring casting, composites are virtually ruled out as being either not technically feasible or too complex to fit within cost guidelines. This points to the combination of solid solution and precipitation strengthening as the only viable approach.



Historically, the development of solid-solution-strengthened alloys for elevated temperature service under high stresses has progressed from relatively simple materials containing chromium, nickel, and iron, e.g., Type 310 stainless steel, through the more complex materials such as Vitallium, N-155, S-816 etc., to the current relatively simple systems exemplified by materials such as Hastelloy X, Inconel alloys 617 and 625, and Haynes 188. Many of the more complex intermediate alloys contain substantial carbon for additional strengthening by carbide precipitation. Included in this group is the 19Cr-9Ni series (DL, DX, etc.) based on Type 304 stainless, and many of the casting alloys. The use of high carbon in high-temperature casting alloys is still very much in practice today, as evidenced by materials such as PK (cast 310 stainless) and X-40 (HS-31).

As temperature and strength requirements increased, the development of precipitation-hardenable alloys based on gamma-prime $\text{Ni}_3(\text{Al}, \text{Ti})$ evolved from the simple upgraded stainless steel, A-286, to the complex cast alloys like IN 738 and IN 792. To drive the precipitation reaction effectively, it is necessary to have a considerable amount of nickel in the alloy--generally at least 25 percent.* Further, this level must be increased as the Ti + Al content is raised above approximately 2 percent. Thus, the stronger alloys are predominantly Ni-based, with little iron; as such, they are quite high in alloy cost, compared to the 19-9 DL target set for this program.

One of the more interesting features of the Ni-based, high-temperature alloys is the generally negative effect of chromium on high-temperature creep strength (ref 1). For years, researchers worked on the tradeoffs of strength and oxidation/hot corrosion resistance by lowering chromium and adjusting the Al/Ti ratio. For gas turbine service, especially turbine blades, some form of coating has invariably been found necessary for the high-strength alloys. A similar phenomenon exists in stainless steels, e.g., creep strength in the 400-Type ferritic grades decreases as one progresses from an 11-percent Cr alloy (Type 409), to a 17-percent Cr alloy (Type 430), to a 26-percent Cr alloy, Type 446, (ref 2). While it is difficult to draw a similar parallel in the austenitic 300-Type grades because of the need to increase nickel level simultaneously, it is worthy of note that creep properties of materials like types 309 and 310 stainless steels, RA330, and Incoloy alloy 800 are not markedly different from those of Types 316 and 347 stainless steels, in spite of the higher alloy content of the former.

It seems logical, then, to discuss the function of alloying elements in high temperature alloys. Because of the cost restraints (develop an alloy with cost similar to or less than that of 19-9 DL), the emphasis is necessarily tailored to base materials of relatively low alloy content i.e., upgraded stainless steel, or downgraded lower-cost superalloys.

*Alloy compositions are given in weight percent.



2.1.3 General Effects of Alloying Elements

Table 2-1 shows a generalized rating of the individual elements in the types of base compositions of interest to the Stirling engine program. It is apparent that cost can only be controlled by using a minimum content of elements from the group nickel, molybdenum, columbium, and tungsten; use of any quantity of either tantalum or hafnium is completely out of the question. (The effect of boron on cost is not completely clear because of the sole-source nature of Fe-B and Ni-B additions; this could require a separate study should a high boron alloy prove promising.) The use of manganese as a substitute for nickel has shown considerable promise with the 200-series stainless steels, (ref 3) and with more highly alloyed materials such as 21-6-9 and 22-13-5 (now called Nitronic 40 and 50, respectively) (ref 4). When combined with sufficient carbon and/or nitrogen, these materials have shown very good properties in stable austenite structures.

The structure of the base alloy is worthy of discussion. The nature of the slip systems is such that the face-centered cubic (FCC), or austenitic, structure is favored over the body-centered cubic (BCC), or ferritic, structure for high-temperature service. While it is true that certain systems exhibiting a microduplex structure of ferrite finely dispersed in austenite have shown very good strength characteristics, the presence of ferrite is generally undesirable for phase stability and hydrogen compatibility, as discussed below. Thus, it is important to balance the alloy composition to produce an essentially FCC structure that is solid-solution-strengthened and carbide-precipitation-hardened. If we are to minimize the use of nickel, then manganese, carbon, and nitrogen must be liberally used.

Relative to castability, it has long been recognized that certain elements increase metal fluidity, including carbon, silicon, and manganese. Boron is a known melting point depressant, which may also increase castability. Loading of an alloy with any of these elements will change the casting parameters, and adjustments will no doubt be necessary.

Phase stability, particularly at 775°C, is a major concern. The necessarily low nickel alloys, probably strengthened with molybdenum, will be susceptible to both sigma and chi phase formation (ref 5). This of itself may not be of direct concern, as these phases can often lead to strengthening at high temperatures. The greater concern is in low-temperature ductility (e.g., during winter in the Northern states) where mechanical or thermal shock could cause problems should a phase change occur. In particular, it will be necessary for the austenitic FCC structure to remain stable to about -50°C to prevent martensite formation, which would be embrittling.

Carbide precipitation can also be embrittling, as is apparently the case with Hastelloy X in the 650° to 870°C range. Within a few thousand hours at 760°C nearly continuous grain boundary networks are found, with tensile ductility at ambient temperature dropping to only a few percent. It may be that any system using carbide strengthening will need to be balanced so that the carbides precipitate predominantly within the grains, preferably as a result of heat treatment so the strength is available for initial service.



TABLE 2-1

EFFECTS OF ALLOYING ELEMENTS IN HIGH-TEMPERATURE, IRON-BASED MATERIALS

Elements	Approximate Cost/lb, \$*	Comments
Chromium	0.52 to 1.00	Strong ferritizer--provides oxidation/corrosion resistance
Nickel	3.45	Strong austenite stabilizer--improves strength and corrosion resistance
Molybdenum	9.90	Strong ferritizers--provide solid solution strengthening and carbide precipitates. Mo promotes pitting resistance.
Tungsten	14.66	
Aluminum	1.00	Ferritizer--provides oxidation resistance
Tantalum	130.00	Ferritizers--carbide and nitride forming elements
Columbium	6.60	
Vanadium	7.03	
Hafnium	80.00	
Manganese	0.66	Austenite stabilizer--can substitute for nickel on a basis of two Mn for one Ni
Silicon	0.50	Ferritizer--normally present as a tramp from deoxidation. Reduces oxide scaling tendency, improves fluidity.
Boron	17.88	Promotes fluidity, improves creep strength, forms borides.
Carbon	--	Austenite stabilizers. Interstitial. React to form precipitates--carbides, nitrides, and carbonitrides
Nitrogen	--	
Iron	0.20	Base (select scrap)

*As ferro-alloy melt addition. Range is for high vs low carbon.



2.1.4 Review of Candidate Systems

Alloy development of candidate systems was conducted extensively in the post-World War II years up to the mid-1950's, when alloys such as A-286 began to pave the way for the gamma-prime-strengthened, nickel-based superalloys. During this period, AF-71 was developed by Allegheny Ludlum (ref 6). It is an austenitic nickel-free Cr-Mn-C-Fe alloy, further stabilized with nitrogen and boron, and strengthened with molybdenum and vanadium. Babcock and Wilcox studied 45% Fe-based alloys for replacement of Vitallium (refs 7 and 8). These materials were nominally 20Cr-28Ni, strengthened with triple additions selected from the group Ta, Nb, Ti, Zr, Mo, W, and N, with Ta-Mo-W and Nb-Mo-W producing the highest strengths. Cornell Aeronautical Laboratory studied additions of Ti, B, V, Zr, N, and C to the 18Cr-8Ni austenitic stainless steel matrix and concluded that titanium and/or boron were the most effective elements for increasing hot strength (ref 9).

In the mid-to-late 1950's considerable work was conducted at the University of Michigan, with hundreds of experimental compositions being cast (ref 10). A promising series of 18Cr-18Ni iron-based materials based on strengthening by formation of a Mo-B eutectic from an austenitic matrix was developed (ref 11); Sponseller has since carried on this work at Climax Molybdenum's Research Laboratory, culminating in alloys XF-818 (18Cr-18Ni-8Mo-C.4Cb-0.8B) and XF-527 (27Cr-30Ni-5Mo-0.9B) (refs 12 through 15). Reference 11 is of particular importance to this current study in showing the general effects and interactions of carbon, boron, molybdenum, and tungsten in the 18Cr-18Ni-Fe austenitic matrix.

Work in the 1960's at Chrysler during development of the automotive gas turbine led to a series of four iron-based casting alloys that contained 18-24Cr, 5Ni, 5Mn, 1W and 1Mo, plus 1-2Cb and substantial additions of carbon and nitrogen (ref 16). These were designated CRM-6D, -15D, -17D, and 18D. These materials are characterized by rather low ductility, apparently due to the high carbon level (see Appendix I in ref 8) which leads to a different microstructural form of the eutectic than is found in the lower carbon (<0.3 percent) molybdenum-boron XF-818 and XF-527 (ref 10).

2.1.5 Oxidation and Hot Corrosion Resistance

There is a great deal of literature on the oxidation and hot corrosion resistance of metals, primarily generated by the gas turbine industry (see ref 1, for example). It is clear that alloys designed for high-temperature oxidation resistance invariably have high chromium (20 percent and greater), often in combination with a lesser amount of aluminum. Hastelloy X is an example of an aluminum-free alloy; some manganese is needed in this alloy to assist in CrMnO₄ spinel formation on the surface, as opposed to pure Cr₂O₃. Of the alloys containing aluminum, Inconel alloy 601 and Inconel alloy 617 (both containing nominally 1 percent Al) are examples of recent trends in alloy design.

The addition of rare earth metals has proven to be a powerful tool in enhancing environmental resistance, probably by improving resistance of the oxide film to spalling. This is demonstrated in Hastelloy S and Haynes 188,



where a few hundredths of a percent of lanthanum are very effective. Work on systems involving the use of yttrium has led to the development of a series of MCrAlY coatings, where M can be iron, nickel, or cobalt, each in combination with fairly high chromium, moderate aluminum, and a small addition of yttrium. The MCrAlY alloys by themselves are not particularly strong; hence they have application only as coatings (except for oxide-dispersion-strengthened materials, e.g., MA-956, which are very expensive).

There have been a number of studies on silicon additions in combination with aluminum and/or rare earths. High silicon levels (generally over 1 percent) have long been recognized for reducing the tendency for oxide scaling in austenitic alloys, e.g., Type 302B stainless steel and RA330. Some recent work in Japan on nominally 300-Type stainless steels (19 percent Cr - 13 percent Ni) with 3 percent Si showed promise of improved resistance to oxide spalling when rare earths and/or calcium were added to the base metal (ref 17). It is now generally recognized that a combination of internal oxides and a defect structure is created beneath the normal protective oxide when the rare earth metals are added, markedly influencing the behavior of the external scale.

Relative to hot corrosion resistance, the 775°C temperature is within the range of formation of molten salts (primarily Na₂SO₄-NaCl mixtures) which will flux the normally protective oxide from the metal surface. Accordingly, the full understanding of fuel composition is important. For example, in the presence of sulfur, nickel alloys are particularly susceptible to sulfidation damage, as the nickel-sulfide eutectic can form, causing liquid damage below 650°C (ref 18). Austenitic stainless steels such as 304, 347, etc., are susceptible to grain boundary attack in the presence of sodium chloride, even as a vapor, above about 575°C (ref 19).

For resistance to these types of attack, the general alloy philosophy has been to increase chromium as high as possible, although, as noted earlier, higher chromium tends to reduce creep-rupture strength, especially in nickel-based alloy. Alternately, coatings have been used, such as diffused aluminides and the MCrAlY types; more recently, combinations of alumide coatings with thin noble metal barriers have been used. Gaseous aluminizing and/or chromizing may also offer a way to provide protection to otherwise poor oxidation/hot corrosion resistant alloys. Gaseous treatment can be applied to the finished assembly (or subassembly) and will reach all surfaces readily. An inexpensive coating may be necessary to meet the cost restraints imposed on the material.

No problems are foreseen with ambient temperature or cold corrosion. Materials which have been engineered for adequate oxidation/hot corrosion resistance would not be expected to corrode under mild aqueous conditions. There are two notable exceptions: sulfuric acid condensation and chloride salt deposits. Short of burning very high sulfur fuel, sulfuric acid condensation would not be expected at 200°C and above (ref 20), and the small amount formed at shutdown should be no worse than in similar equipment now in operation (e.g., diesel engines and exhaust systems). Protection against hot sulfuric acid is very difficult, such that designers generally keep operating temperatures above the condensation point.



Salt deposits containing chloride ions can lead to pitting if condensed moisture creates an electrolyte. Again, most high-temperature alloys would contain sufficient chromium and molybdenum to retard pitting during down periods, but conditions conducive to pitting should be avoided as much as possible. As noted above, the presence of chlorides in the engine is also undesirable relative to hot corrosion problems.

2.1.6 Hydrogen Embrittlement

Hydrogen damage can take two forms: hydrogen can either enter the metal lattice (charging), or react instantaneously with an advancing crack tip (creating notch sensitivity). In carbon steels, for example, it is believed that there is internal reaction between carbon and hydrogen to form methane, which collects to form blisters or pores with resulting cracks in the material. By adding sufficient chromium, vanadium, or other elements to form stable carbides, methane formation can be minimized or eliminated (ref 21).

In the austenitic alloys there is good correlation between stability (freedom from deformation martensite) and resistance to high-pressure hydrogen. Similarly, the high-strength martensitic (and PH) steels are highly susceptible to hydrogen embrittlement. The effect of the presence of soft ferrite is not completely clear, as tests on duplex (austenite-ferrite) steels have been limited, but results to date have not shown a deleterious effect due to the presence of a small amount of ferrite in austenite (ref 22).

Literature on hydrogen embrittlement is also abundant. Most of it is directed toward discussions of mechanisms, however, as opposed to alloying effects on resistance. It is generally known that nickel-based alloys are fairly susceptible, whereas austenitic iron-based alloys are the least susceptible to hydrogen damage. Several papers have been written on the effects of alloying elements on hydrogen embrittlement of A-286 (refs 23 and 24).

Smugeresky (ref 23) showed that a high Ti + Al content enhanced resistance to hydrogen damage. Conversely, Thompson and Brooks (ref 24) showed that removal of manganese was beneficial, especially in the presence of increased (5%) nickel, with adverse effects from titanium and aluminum additions. At this point, it is not clear what the alloying effects really are; testing will be necessary.

2.2 PRELIMINARY SCREENING STUDY

In this section, the rationale for selecting the starting alloy compositions is discussed, as well as the procedures used in casting and testing and results of the first tests.

2.2.1 Selection of Candidate Systems

As described in the literature review, work on iron-based alloy systems for high-temperature service has been very limited in recent years, with primary emphasis on oxidation-resistant ferritic alloys that simply lack the required strength to meet the targets of this program. The review has identified several candidate austenitic systems for further exploration. These include CRV-6D, XF-818, and AF-71 as the most promising; 19-9DL appears to have little



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potential, and already has been heavily investigated (DX, WMo, WX). Initially, the two items of concern are stress rupture strength and cost, which are covered in Figure 2-1 and Table 2-2, respectively.

Data in Figure 2-1 are from the DMIC compilation (ref 25); costs were calculated from the alloy element costs shown in Table 2-1.

The basic approach is to work with an iron-based austenitic matrix, i.e., Cr-Ni, Cr-Ni-Mn, or Cr-Mn, and strengthen by one or more of the following techniques: (1) M_3B_2 eutectic with molybdenum or tungsten; (2) carbides of columbium, vanadium, chromium, molybdenum, and tungsten; and (3) solid solution with molybdenum or tungsten. In cases (1) and (2), applicable heat treatments include high-temperature solutioning to spheroidize, plus aging to form precipitates, especially carbides. Starting compositions for the first half of the screening study are given in Table 2-3, and are referenced to the starting alloys. Group 4, based on 18Cr-18Ni-5Mo-1.25B-2W, was derived from some of the early Sponseller work (ref 11).

Group 1 of Table 2-3 is based on CRM-6D. As indicated in Figure 1, creep strength is near the target level. Projected cost is below that of 19-9DL, giving room for alloy additions from the group Mo, W, Cb, and V. This will provide both solid solution and carbide strengthening. It should be possible to solution treat and age to enhance strength through precipitation of carbides. Chromium level has also been reduced to enhance creep strength.

The basis for group 2 is XF-818. Because it has a cost above the target level, the alloying approach is to reduce molybdenum level while adding boron and carbon to increase strength. This material should also be heat-treatable by the spheroidizing high temperature anneal reported by Sponseller et al. (ref 11).

The group 3 base is AF-71, which, by virtue of being nickel-free, has the lowest base cost, as shown in Table 2-2. Because it was developed as a wrought alloy, there is room for substantial increase in alloy content, particularly boron, which has been reported to lead to significantly improved strength (ref 26). It has also been reported that vanadium is very effective in promoting precipitates when aged at 1300°F (refs 6 and 26). A molybdenum increase may also prove beneficial, e.g., to form the M_3B_2 eutectic in a nickel-free base.

The fourth group, based on a tungsten-containing version of XF-818 which has shown attractive properties in earlier work (ref 11), will combine boron- and carbide-strengthening. Varying levels of molybdenum, columbium, and vanadium are utilized for both cost control and strength. Manganese has been partly substituted for nickel in some versions, again to reduce the inherently high cost of this base. These materials should also be heat-treatable by solution plus age, though initially they will be evaluated without heat treatment.

In order to allow the early test results to influence the choice of compositions to be investigated, the initial alloy composition matrix has been limited to 21 alloys, as agreed by ACC and NASA-Lewis Research Center during a review meeting held September 26, 1980 at NASA-Lewis.

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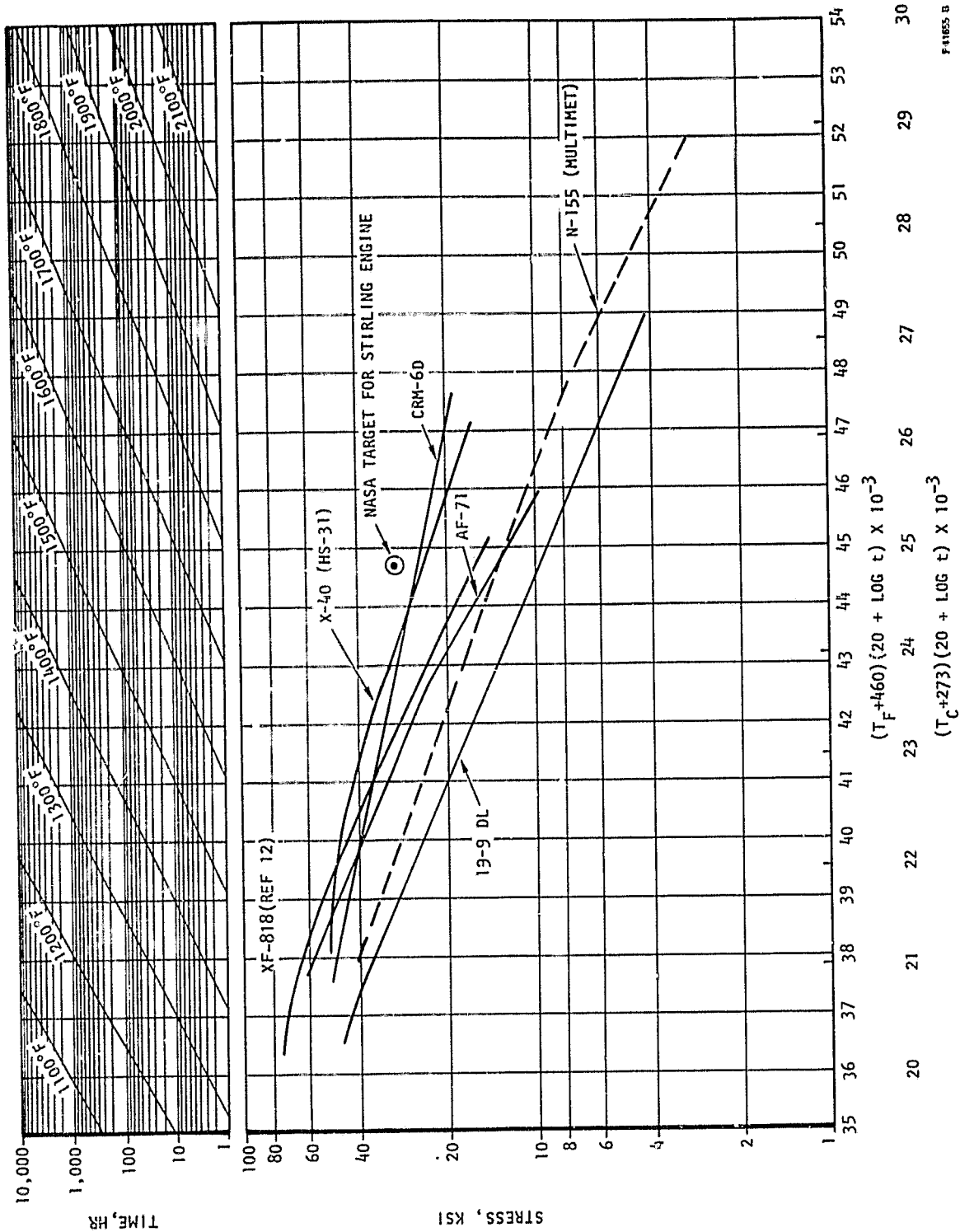


Figure 2-1. Stress-Rupture Strength of Candidate Starting Alloys, Reference Alloys, and Target (ref 25)

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TABLE 2-2
COMPOSITIONS OF CANDIDATE STARTING ALLOYS
AND REFERENCE MATERIALS

Designation	C	N	Mn	Si	Cr	Ni	Co	Mo	W	Co	Fe	Ti	B	V	Approximate Raw Material Cost, \$/lb
AF-71	0.3	0.2	18.0	0.3	12.0	-	-	3.0	-	-	BAL	-	0.2	0.90	0.72
CPM-65	0.5	-	5.0	0.5	22.0	5.0	-	1.0	1.0	1.0	BAL	-	0.033	-	2.77
XF-818	0.2	-	0.1	0.3	18.0	18.0	-	7.5	-	0.4	BAL	-	0.6	-	1.79
19-9DL	0.3	-	1.1	0.6	19.0	9.0	-	1.25	1.2	0.4	BAL	0.3	-	-	1.09
N-155	0.15	0.15	1.5	0.5	21.0	25.0	20	3.0	2.5	1.0	BAL	-	-	-	6.50*
X-40 (HS-31)	0.5	-	1.0	0.5	22.5	10.5	BAL (52.5)	-	7.5	-	2.0	-	0.01	-	14.29*

*Cobalt included at \$24/lb.



TABLE 2-3
ALLOY APPROACH

Alloy No.	Composition - Wt. percent (bal. Fe)										
	C	Mn	Si	Cr	Ni	Mo	B	N	W	Cb	V
Group 1 - Base CRM-6D											
1-1	1.0	5	0.3	22	5	1.0	-	-	1.0	1.0	-
1-2	1.0	5	0.3	17	5	3.0	-	-	1.0	1.0	-
1-3	1.0	5	0.3	17	5	2.0	-	-	2.0	1.0	-
1-4	1.0	5	0.3	17	5	1.0	-	-	1.0	1.5	0.5
1-5	1.0	5	0.3	17	5	1.0	-	-	1.0	1.0	1.0
Group 2 - Base, XF-818											
2-1	0.2	0.2	0.3	18	18	7.5	0.7	0.1	-	0.4	-
2-2	0.35	0.2	0.3	18	18	5.0	1.0	0.1	-	0.4	-
2-3	0.5	0.2	0.3	18	18	5.0	1.25	0.1	-	0.4	-
2-4	0.5	0.2	0.3	18	18	7.5	1.25	0.1	-	0.4	-
2-5	0.08	0.2	0.3	18	18	6.0	1.5	0.1	-	-	-
2-6	0.5	0.2	0.3	18	18	5.0	1.25	0.2	-	0.4	-
Group 3 - Base, AF-71											
3-1	0.3	18	0.3	12	-	3.0	0.2	0.2	-	-	0.9
3-2	0.3	18	0.3	12	-	3.0	0.75	0.2	-	-	0.9
3-3	0.3	18	0.3	12	-	5.0	1.25	0.2	-	-	0.9
Group 4 - Base, Fe - 18Ni - 18Cr - 5Mo - 1.25B - 2W - 0.25C											
4-1	0.25	0.2	0.3	18	18	5.0	1.25	-	2.0	-	-
4-2	0.5	0.2	0.3	18	18	5.0	1.25	-	2.0	-	-
4-3	0.5	0.2	0.3	18	18	4.0	1.25	-	2.0	2.0	-
4-4	0.5	0.2	0.3	18	18	4.0	1.25	-	2.0	1.0	1.0
4-5	0.25	3.0	0.3	18	10	5.0	1.25	-	2.0	-	-
4-6	0.5	3.0	0.3	18	10	4.0	1.25	-	2.0	1.0	1.0
4-7	0.65	0.2	0.3	18	18	5.0	2.0	-	-	-	-



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2.2.2 Experimental Procedure

2.2.2.1 Preparation of Molds

Wax injection tools for the stress-rupture specimen shown in Figure 2-2 and the castability test specimen shown in Figure 2-3 were procured. Tooling for the round tensile test specimen shown in Figure 2-4 was already on hand at ACC. Wax patterns were assembled as shown in Figure 2-5, with each assembly being made up of four stress-rupture specimen patterns, three tensile test specimen patterns, and one castability test specimen pattern. Patterns were dipped in refractory slurries and stuccos to build up shell molds, following standard precision casting foundry practice. A mold system with a zircon face coat bonded with colloidal silica was used. After steam autoclave dewaxing, molds were loaded into a preheat furnace and held at least 3 hr for firing.

2.2.2.2 Melting and Casting

Raw materials used for casting the experimental alloys listed in Table 2-3 are shown in Table 2-4. Most were obtained from Certified Alloy Products, Inc. and are standard melting stock.

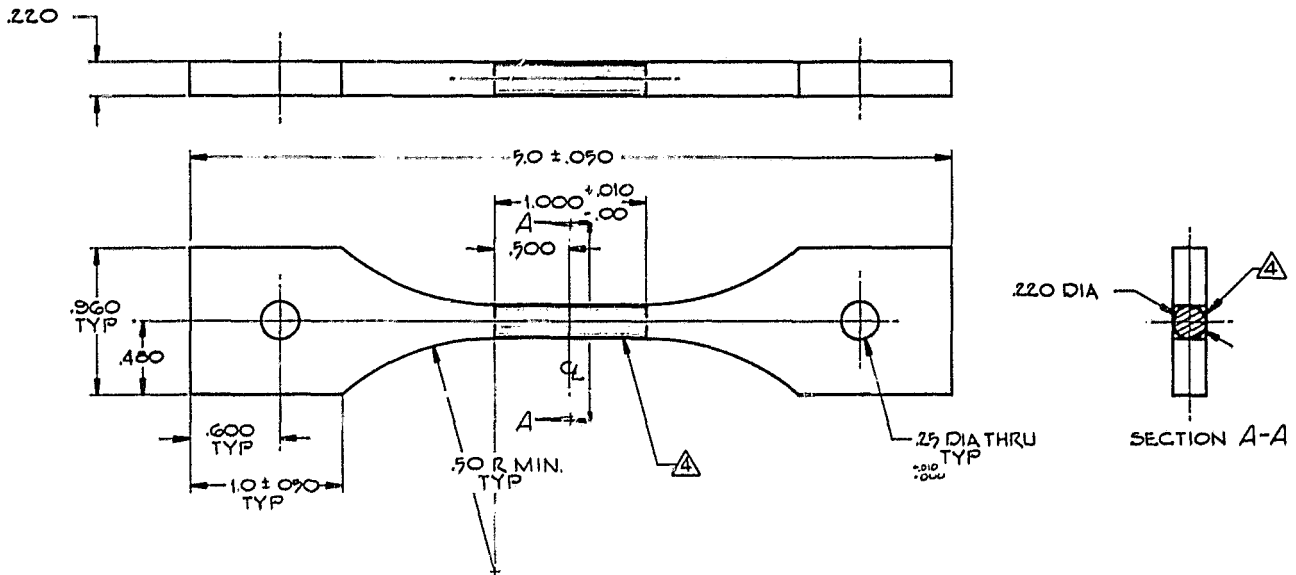
Melting procedures for the four groups of alloys are given in Table 2-5. Alloys were induction-melted in air as 10-lb heats using an MgO crucible. Heats N10001, N10002, and N10003 were melted in a silica crucible. An argon cover was used in melting the alloys of Group 1 and Group 3 because of their higher manganese content. Pouring temperatures were 2800°F for the alloys of Group 1 and 2700°F for the remainder of the alloys. Melts were poured into molds which had been heated to 1900°F and allowed to cool for 3 to 3-1/2 minutes before pouring (shown by thermocouple measurement to result in a mold temperature of 1500° to 1600°F). An exothermic hot-topping compound (Supertherm No. 3, Exomet Inc., Conneaut, Ohio) was used to improve metal feeding.

Representative castings were photographed after mold removal and again after sandblasting. Specimens were then cut off and inspected by X-ray radiography. Specimens other than the above were cut from the gating, sandblasted, and inspected by X-ray radiography. In a number of cases, radiography was erroneously conducted before sandblasting. Careful inspection of the specimens and X-ray film, however, confirmed that x-ray density of refractory material which adhered to surfaces of the specimens was sufficiently low that no radiographic density variation due to this cause could be detected. Stress-rupture specimens were ranked according to radiographic quality, with the best two from each group of four being selected for testing.

Specimens for chemical analysis and preliminary metallographic examination were cut from the bottom ring gate of each casting. Chemical analysis was determined by spark emission spectrography, except for two groups of elements: (1) chromium, nickel, and molybdenum, which were analyzed by X-ray fluorescence, and (2) carbon, oxygen, and nitrogen, which were analyzed by Leco combustion methods.

Rockwell "C" hardness was determined on a sample from the bottom gating ring of each casting, utilizing the average of at least three measurements.

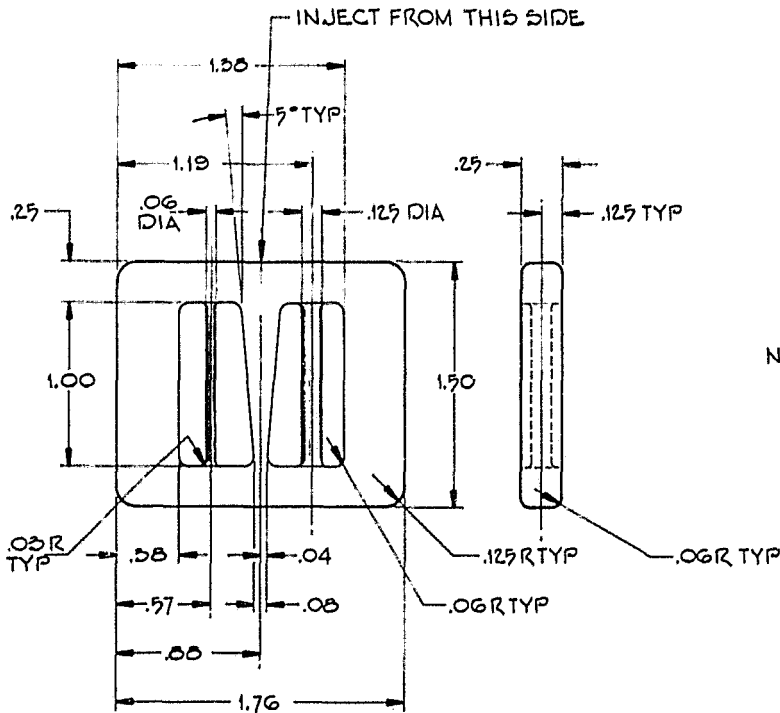




NOTES:

1. STAMP TOOLING USING .09 HIGH LETTERING, THE FOLLOWING:
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CONTRACT N° DEN D-234
TOOL N° T-62252
2. DIM'S DO NOT INCLUDE SHRINK. SHRINK TO BE 1.025IN./IN.
3. DIM'S - ±.010, EXCEPT AS NOTED.
- △ TRANSITION FROM SQUARE TO ROUND MUST BE SMOOTH.

Figure 2-2. Stress-Rupture Specimen



NOTES:

1. STAMP TOOLING USING .09 HIGH LETTERING, THE FOLLOWING:
NASA LEWIS
CONTRACT N° DEN D-234
TOOL N° T-62254

Figure 2-3. Castability Test Specimen



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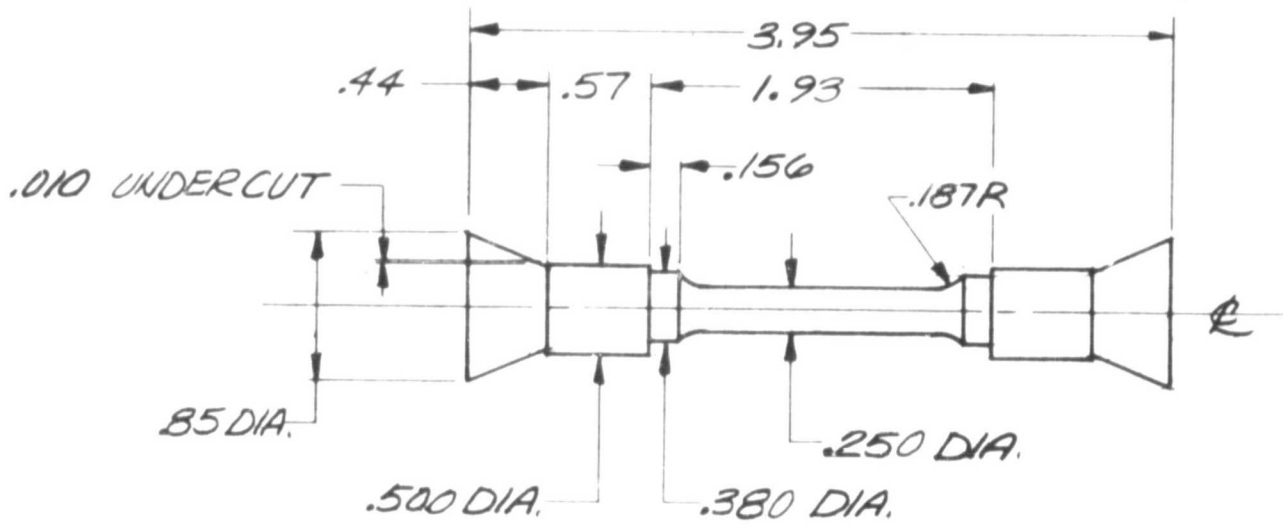


Figure 2-4. Tensile Test Specimen

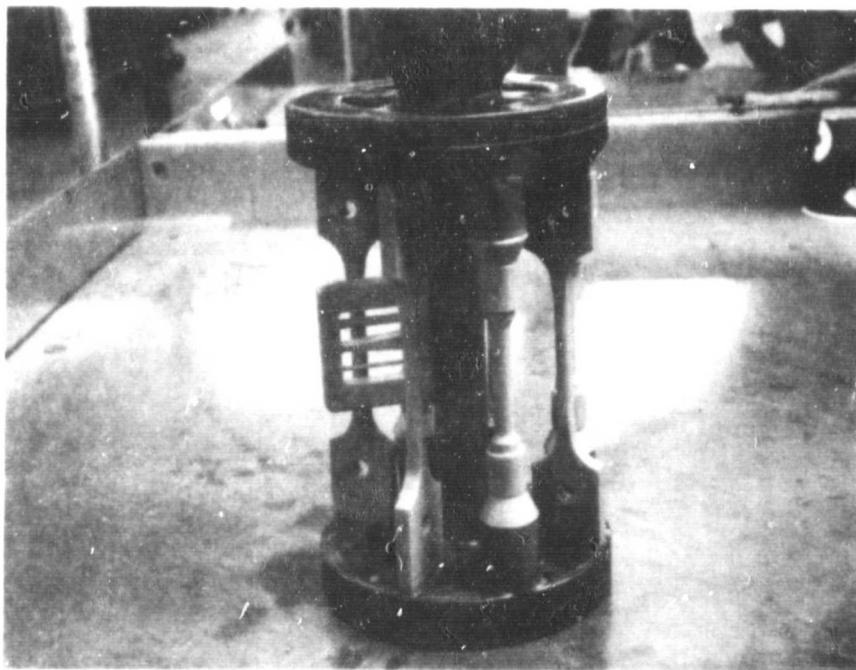


Figure 2-5. Wax Pattern Assembly

F-33235



TABLE 2-4
RAW MATERIALS FOR MELTING

Electrolytic iron
Carbonyl nickel
Vacuum grade chromium
Pure molybdenum, melting grade
Pure tungsten
Electrolytic manganese
Ferroboron (17.5% B)
Ferrovanadium (78% V)
Ferrochromium (60% Cr)
Ferrosilicon (75% Si)
Asbury 607 carbon
High nitrogen ferrochromium (5.5% N, 65% Cr)



TABLE 2-5
MELT PROCEDURES

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Group 1 Alloys (Argon blanket)

Charge Fe, Ni, W, C, 1/3 Cr, 1/3 FeSi

Melt down

Add Mo

Heat to 2900°F

Add balance of FeSi, balance of Cr

Adjust to 2900°F

Add FeCb, FeV, Mn

Adjust to 2800°F

Slag

Pour

Group 2 Alloys

Charge Fe, Ni, High N FeCr, C, 1/3 Cr, 1/3 FeSi

Melt down

Add Mo

Heat to 2900°F

Add balance of FeSi, Mn, balance of Cr

Reheat to 2900°F

Cool to 2800°F, slag

Add FeCb, FeB

Adjust to 2700°F

Pour



TABLE 2-5 (Continued)

Group 3 Alloys (Argon Blanket)

Charge Fe, Mn, High N FeCr, C, Mo, 1/3 Cr, 1/3 FeSi

Melt down

Heat to 2800°F

Add balance of FeSi, balance of Cr

Adjust to 2800°F

Cool to 2700°F, slag

Add FeV, FeB

Adjust to 2700°F

Pour

Group 4 Alloys

Charge Fe, Ni, C, V, 1/3 Cr, 1/3 FeSi

Melt down

Add Mo

Heat to 2900°F

Add balance of FeSi, Mn, balance of Cr

Reheat to 2900°F

Cool to 2800°F, slag

Add FeCb, FeV, FeB

Adjust to 2700°F, slag

Pour



No heat treatments were performed at this stage on specimens from Groups 2 and 4. Group 1 specimens are currently being heat-treated for 100 hr at 1200°F, as recommended for CRM-6D (ref 25). Solution treatment experiments on the Group 3 modifications of AF-71 indicated that partial melting occurred, even at 1950°F, though solution treatment at temperatures as high as 2050°F is recommended for AF-71 (ref 6). As stress-rupture testing proceeds, therefore, Alloys 3-2 and 3-3 will be given aging treatments only, while Alloy 3-1 (AF-71) will be solution-treated and aged. A protective foil pouch will be used for solution treatment.

Stress-rupture testing is in progress, at 830°C (1526°F) and 200 MPa (29 ksi), using calibrated test facilities at the AIResearch Manufacturing Company of California. Stress-rupture life of approximately 330 hr at this temperature is equivalent to the target life of 5000 hr at 775°C. Limited results of completed tests are shown in a later section of this report.

2.2.3 Results and Discussion

2.2.3.1 Melting and Casting

No major difficulties were encountered in melting or casting any of the experimental alloys. The high manganese alloys of Group 3 showed evidence of oxide evolution during melting, even with use of an argon blanket. Surface quality of all castings, as shown in the examples of Figures 2-6 through 2-8, was good to excellent. Group 3 alloy castings, while fully acceptable, had the poorest surface quality, as can be seen in Figure 2-7. Examination of castability test tabs revealed no problems with hot tearing or lack of fluidity.

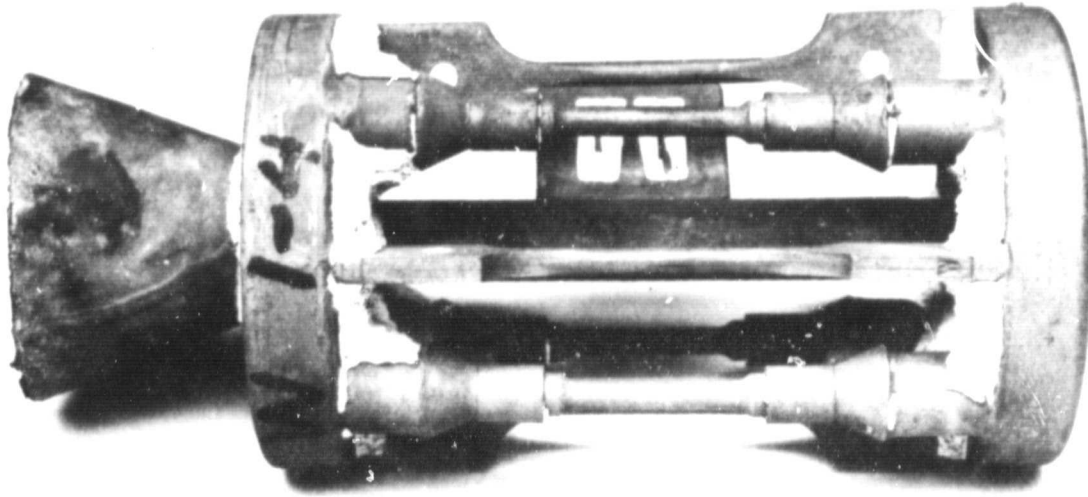
X-ray radiography revealed internal porosity in some of the specimens. All stress-rupture specimens were rated, and those with no porosity or with the smallest amounts of porosity were selected for stress-rupture testing.

2.2.3.2 Chemical Analysis

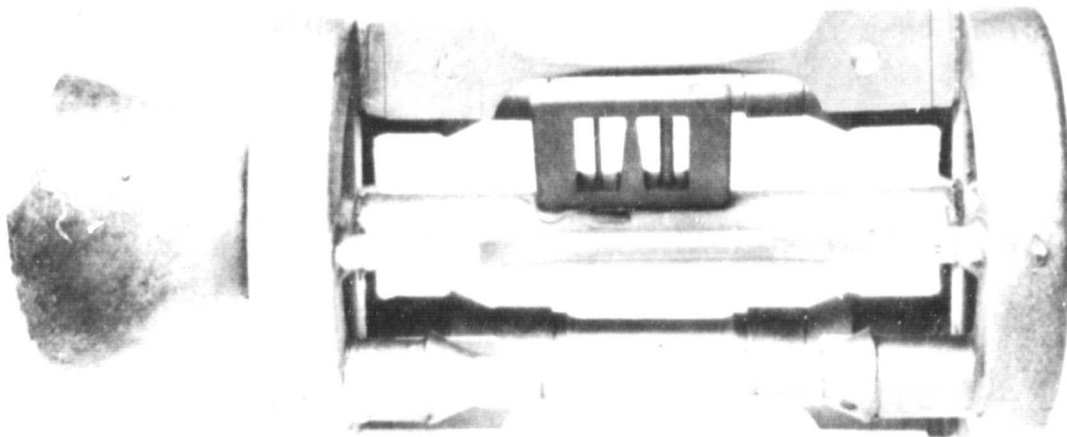
Compositions of experimental castings are shown in Table 2-6. With the exception of apparent inadvertent omission of tungsten in five alloys and lower nitrogen than expected in several alloys (most notably the three alloys of Group 3), compositions were judged acceptable for purposes of this screening study. The eight alloys indicated with an asterisk in Table 2-6 are being remelted. A decision on possible remelting of the Group 2 alloys on the basis of low nitrogen contents will be made after initial stress-rupture testing. Stress-rupture data will be determined for all compositions, even those obtained through melting errors.



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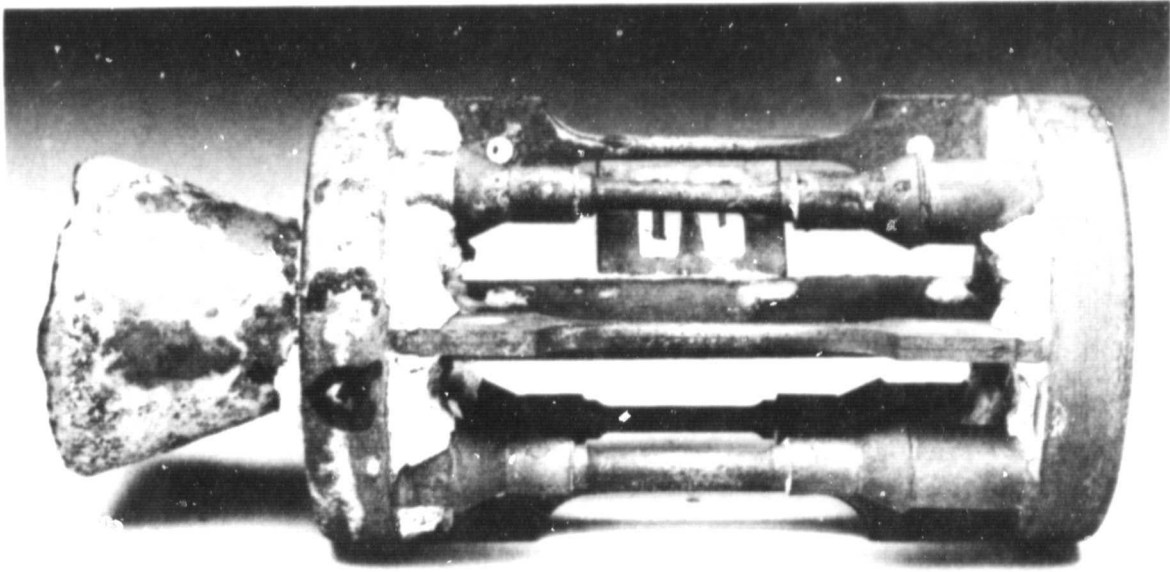
N 10004
ALLOY 1-4, MOLD "W"

83913-3

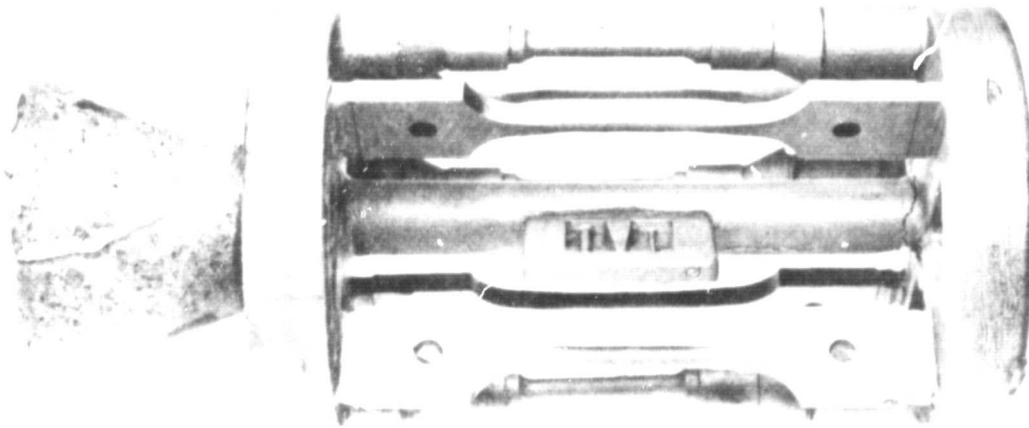
F-33237

Figure 2-6. Alloy 1-4 Casting (Heat N10004) Top, As Cast; Bottom, Sandblasted





83913-5



N10014
ALLOY 3-3, MOLD "Q"

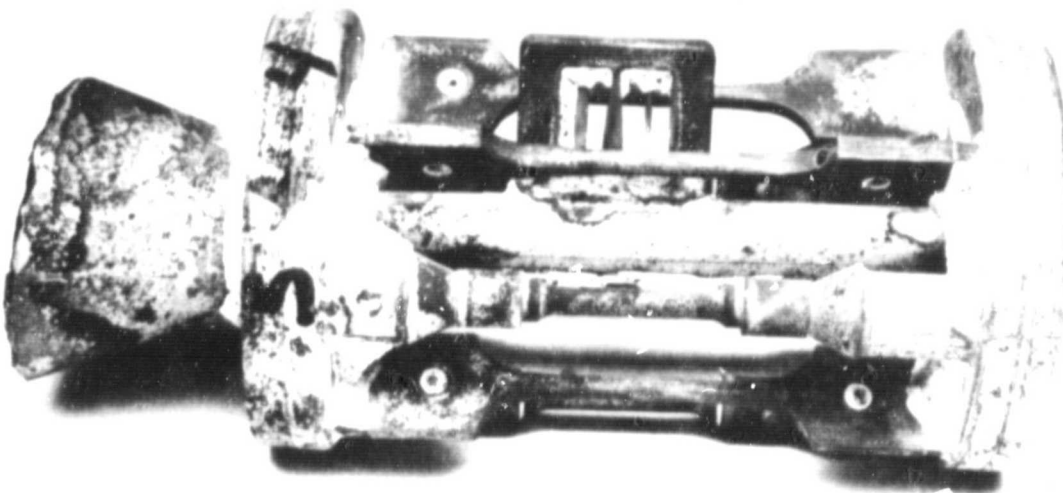
83913-2

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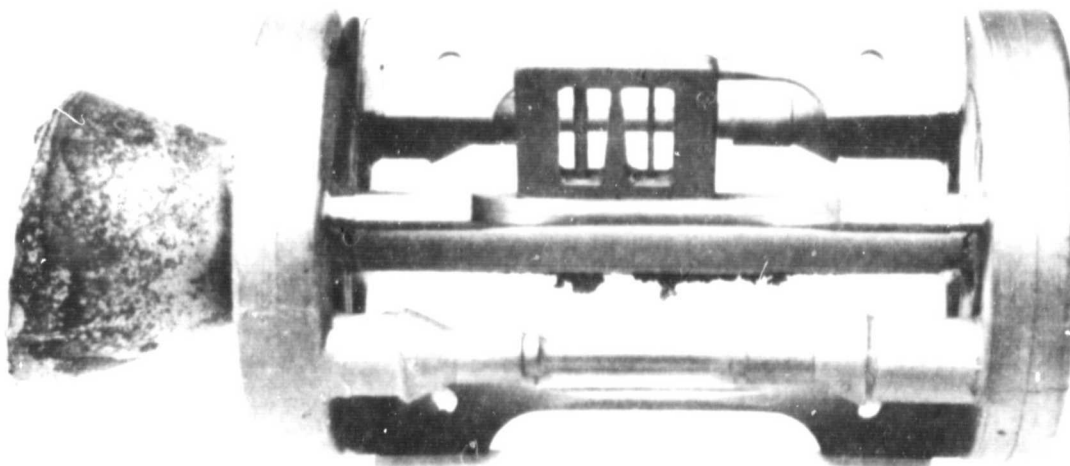
Figure 2-7. Alloy 3-3 Casting (Heat N10014) Top, As Cast; Bottom, Sandblasted



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N 10018
ALLOY 4-4, MOLD "S"

83913-1

F-33238

Figure 2-8. Alloy 4-4 Casting (Heat N10018) Top, As Cast; Bottom, Sandblasted



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Torrance, California

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Page 2-21

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TABLE 2-6
ANALYSES OF EXPERIMENTAL ALLOYS

Alloy No.	Heat No.	Weight percent (bal. Fe)											
		C	Mn	Si	Cr	Ni	Mo	B	N	W	Cb	V	O
Group 1 - Base CRM-6D													
1-1	NI0001	0.84	4.09	0.57	21.64	4.83	1.01	<.001	0.040	1.04	1.12	<.01	0.061
	Aim	1.0	5.0	0.3	22.0	5.0	1.0	-	-	1.0	1.0	-	-
1-2	NI0002	0.90	4.19	0.51	16.41	4.82	2.86	<.001	0.050	1.10	1.10	<.01	0.025
	Aim	1.0	5.0	0.3	17.0	5.0	3.0	-	-	1.0	1.0	-	-
1-3	NI0003*	0.97	4.65	0.46	17.51	4.96	1.96	<.001	0.039	<.05	1.13	<.01	0.019
	Aim	1.0	5.0	0.3	17.0	5.0	2.0	-	-	2.0	1.0	-	-
1-4	NI0004	0.89	5.05	0.33	16.89	4.73	1.16	0.028	0.072	1.04	1.57	0.28	0.015
	Aim	1.0	5.0	0.3	17.0	5.0	1.0	-	-	1.0	1.5	0.5	-
1-5	NI0005	0.93	4.80	0.32	17.17	4.72	1.17	0.009	0.073	1.10	1.13	1.16	0.014
	Aim	1.0	5.0	0.3	17.0	5.0	1.0	-	-	1.0	1.0	1.0	-
Group 2 - Base, XF-818													
2-1	NI0006	0.18	0.20	0.29	18.49	18.12	7.98	0.72	0.038	<.05	0.51	<.01	0.0048
	Aim	0.2	0.2	0.3	18.0	18.0	7.5	0.7	0.1	-	0.4	-	-
2-2	NI0007	0.32	0.25	0.35	20.18	17.72	5.61	1.15	0.041	<.05	0.54	<.01	0.0074
	Aim	0.35	0.2	0.3	18.0	18.0	5.0	1.0	0.1	-	0.4	-	-
2-3	NI0008	0.46	0.26	0.36	20.59	17.36	5.57	1.27	0.038	<.05	0.50	<.01	0.0088
	Aim	0.5	0.2	0.3	18.0	18.0	5.0	1.25	0.1	-	0.4	-	-
2-4	NI0009	0.48	0.27	0.39	19.07	18.37	8.11	1.26	0.052	<.05	0.52	<.01	0.012
	Aim	0.5	0.2	0.3	18.0	18.0	7.5	1.25	0.1	-	0.4	-	-
2-5	NI0010	0.11	0.26	0.36	19.74	17.91	6.54	1.44	0.072	<.05	0.034	<.01	0.0042
	Aim	0.08	0.2	0.3	18.0	18.0	6.0	1.5	0.1	-	-	-	-
2-6	NI0011	0.45	0.28	0.42	20.35	17.53	5.61	1.35	0.050	<.05	0.52	<.01	0.0048
	Aim	0.5	0.2	0.3	18.0	18.0	5.0	1.25	0.2	-	0.4	-	-
Group 3 - Base, AF-71													
3-1	NI0012*	0.38	11.58	0.34	12.70	<.10	4.00	0.25	0.043	<.05	0.041	1.21	0.0078
	Aim	0.3	18.0	0.3	12.0	-	3.0	0.2	0.2	-	-	0.9	-
3-2	NI0013*	0.34	17.80	0.31	11.82	<.10	3.44	0.42	0.067	<.05	0.025	0.76	0.0064
	Aim	0.3	18.0	0.3	12.0	-	3.0	0.75	0.2	-	-	0.9	-
3-3	NI0014*	0.34	17.36	0.37	11.47	<.10	5.87	0.88	0.097	<.05	0.036	1.07	0.0028
	Aim	0.3	18.0	0.3	12.0	-	5.0	1.25	0.2	-	-	0.9	-
Group 4 - Base, Fe - 18Ni - 18Cr - 5Mo - 1.25B - 2W - 0.25C													
4-1	NI0015*	0.25	0.39	0.37	19.57	17.73	5.37	1.35	0.047	<.01	0.031	<.01	0.0084
	Aim	0.25	0.2	0.3	18.0	18.0	5.0	1.25	-	2.0	-	-	-
4-2	NI0016*	0.46	0.28	0.37	19.60	17.90	5.38	1.44	0.049	<.01	0.031	<.01	0.0082
	Aim	0.5	0.2	0.3	18.0	18.0	5.0	1.25	-	2.0	-	-	-
4-3	NI0017*	0.44	0.27	0.37	19.26	17.93	4.40	1.25	0.064	<.01	2.01	<.01	0.011
	Aim	0.5	0.2	0.3	18.0	18.0	4.0	1.25	-	2.0	-	-	-
4-4	NI0018	0.49	0.25	0.37	19.26	18.01	4.35	1.32	0.054	2.31	1.07	0.98	0.088
	Aim	0.5	0.2	0.3	18.0	18.0	4.0	1.25	-	2.0	1.0	1.0	-
4-5	NI0019*	0.28	2.53	0.33	20.49	7.90	5.58	1.31	0.075	<.01	0.032	<.01	0.0088
	Aim	0.25	3.0	0.3	18.0	10.0	5.0	1.25	-	2.0	-	-	-
4-6	NI0020	0.46	2.39	0.36	19.80	10.13	4.57	1.10	0.069	2.37	0.97	1.12	0.0070
	Aim	0.5	3.0	0.3	18.0	10.0	4.0	1.25	-	2.0	1.0	1.0	-
4-7	NI0021	0.52	0.31	0.38	20.74	17.63	5.46	1.81	0.044	<.01	0.032	<.01	0.0044
	Aim	0.65	0.2	0.3	18.0	18.0	5.0	2.0	-	-	-	-	-

*Alloys to be remelted.



2.2.3.3 Structure, Hardness, and Stress-Rupture Data

Metallographic structures of all 21 heats currently being tested are shown in Figures 2-9 through 2-12. Hardness data and initial stress-rupture results are summarized in Table 2-7. Because stress-rupture data are not complete, a detailed evaluation of the relationships between composition, microstructure, and properties cannot yet be made. The following are preliminary observations.

2.2.3.3.1 Group 1 Alloys, Base CRM-6D

All alloys have structures containing the expected austenite matrix with interdendritic carbides. Two distinct types of carbide can be seen. The first is a massive carbide that is semicontinuous in Alloys 1-1 (CRM-6D) and 1-2. The second is a lamellar form, found as an eutectic with austenite. More than one lamellar form may exist. The alloy with lowest total content of the carbide formers Cr, Mo, W, Cb, and V (Alloy 1-4) appears to have the smallest amount of total carbide (Alloy 1-4) and has the lowest as-cast hardness. Aging improves hardness of all alloys in the series by substantial amounts.

2.2.3.3.2 Group 2 Alloys, Base XF-818

The Group 2 alloys have the expected austenite matrix with carbide/boride interdendritic phases, which are either continuous or semicontinuous. Hardness of the baseline alloy (Alloy 1-1) is lower than the Rockwell "C" 20 expected for XF-818, possibly because of the low nitrogen content and because hardness measurements were made on a 3/4-in.-thick gating section rather than the more rapidly cooled tensile specimen cross section on which Sponseller's hardness determinations were made (ref 10).

Note that the highest hardness values were obtained in alloys which appear to have the smaller amounts of total carbide plus boride phases (Alloys 2-3 and 2-6). The longest stress-rupture life in a Group 2 alloy to date has been obtained in Alloy 2-4, with the highest carbon content and the greatest amount of interdendritic eutectic. Stress-rupture data show that an attempt to obtain strengthening predominantly with borides (Alloy 2-5) was not successful.

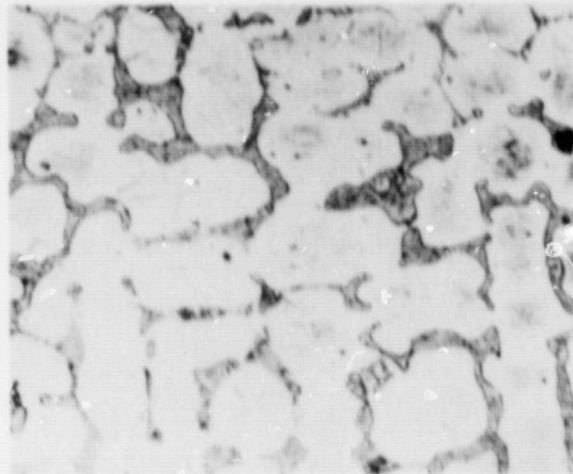
2.2.3.3.3 Group 3 Alloys, Base AF-71

Alloys of Group 3 also contain a combination of dendritic matrix and continuous or semicontinuous interdendritic eutectic. There is a substantial difference between the three alloys in terms of both the quantity and nature of the interdendritic eutectic. Alloy 3-3, with the highest boron content, has the highest hardness and the greatest quantity of interdendritic phase.

2.2.3.3.4 Group 4 Alloys, Base Fe-18Ni-18Cr-5Mo

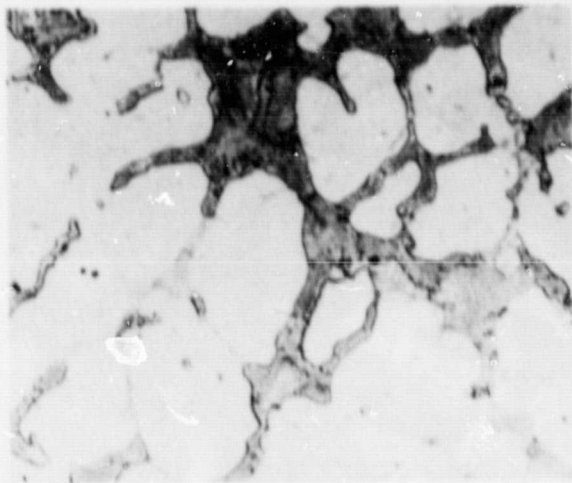
A comparison of Group 2 alloys and Group 4 alloys shows that the latter generally have higher as-cast hardness and better stress-rupture behavior. Microstructures of the two groups are basically similar, with varying amounts of interdendritic eutectic. The nature of the interdendritic phase is different in Alloy 4-6, with large amounts of the carbide formers W, Cb, and V than in other Group 4 alloys. Alloy 4-7, with high B and only Cr and Mo as





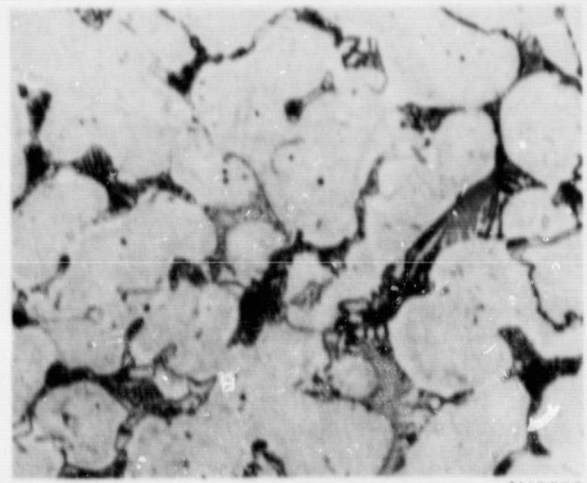
Alloy 1-1, CRM-6D

N10001



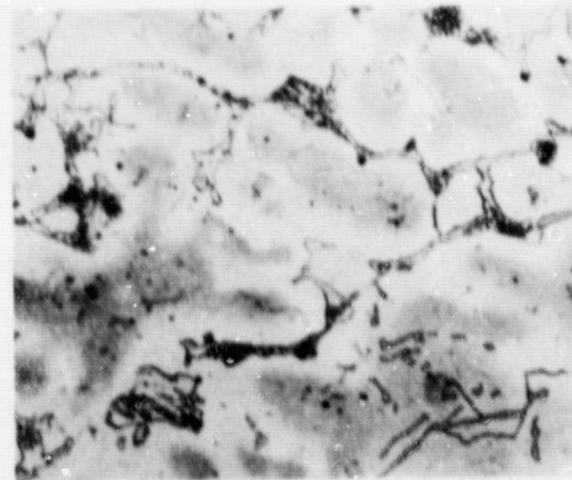
Alloy 1-2

N10002



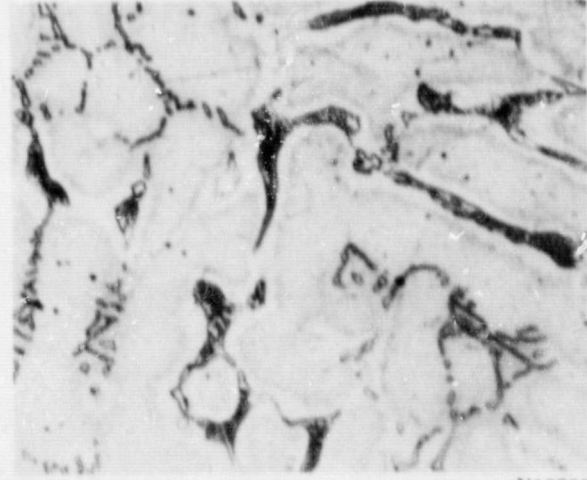
Alloy 1-3

N10003



Alloy 1-4

N10004



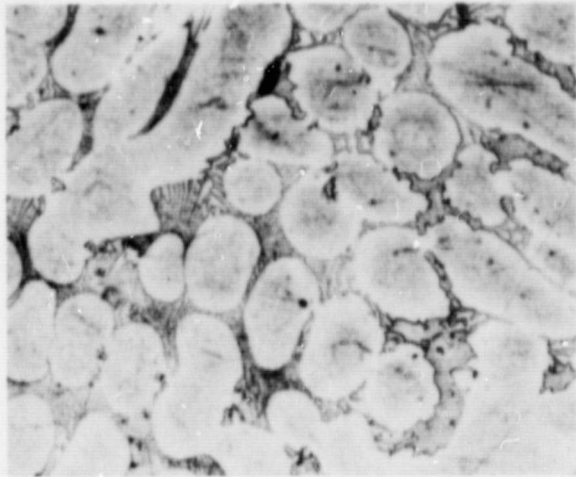
Alloy 1-5

N10005

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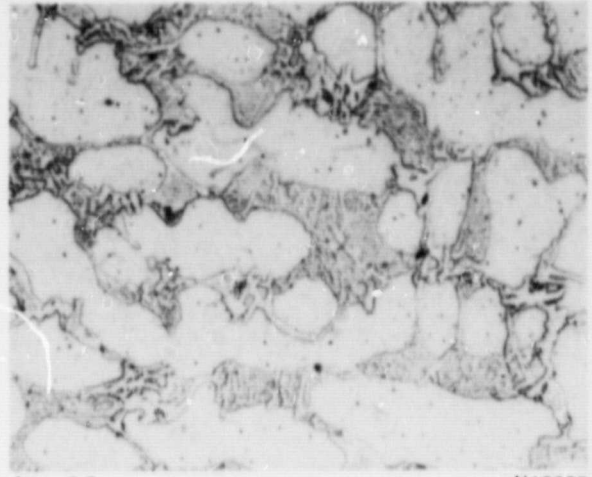
Figure 2-9. Optical Micrographs of Group 1 Alloys,
Etchant: Fry's Reagent, Magnification: 200X





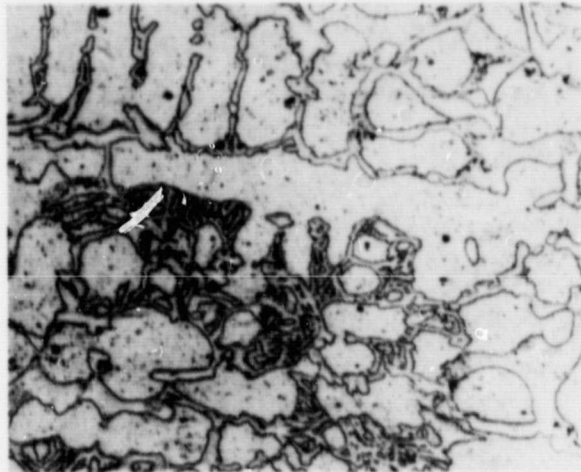
Alloy 2-1, XF-81B

N10006



Alloy 2-2

N10007



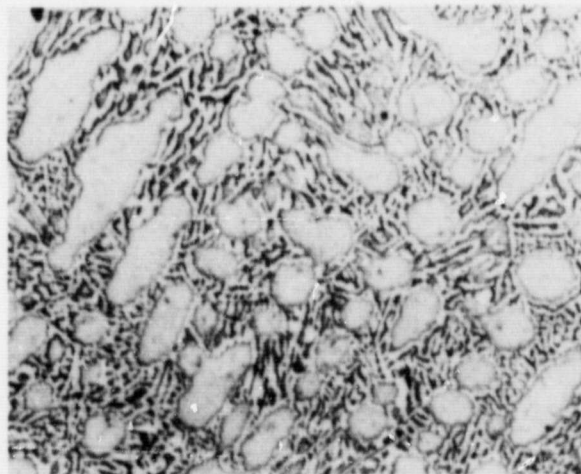
Alloy 2-3

N10008



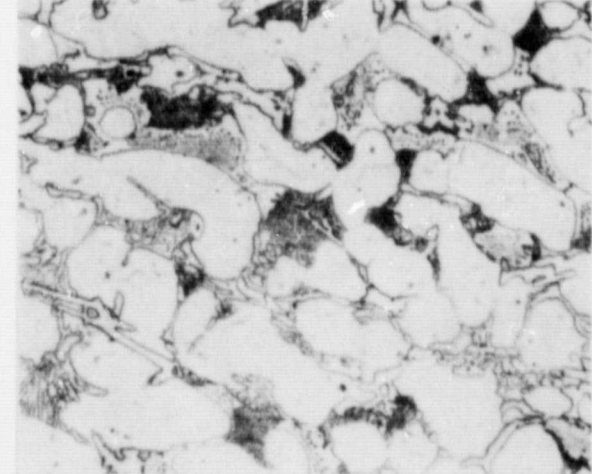
Alloy 2-4

N10009



Alloy 2-5

N10010



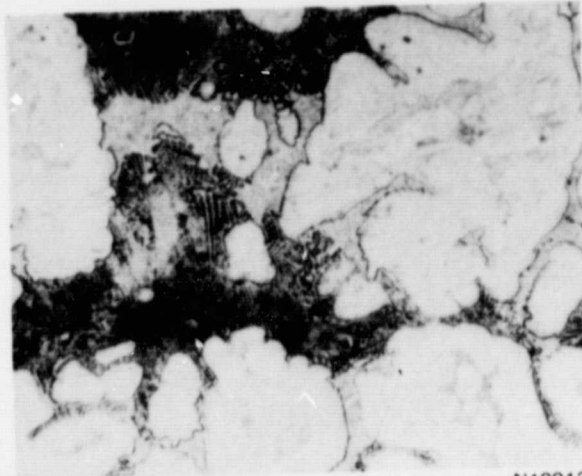
Alloy 2-6

N10011

F-33231

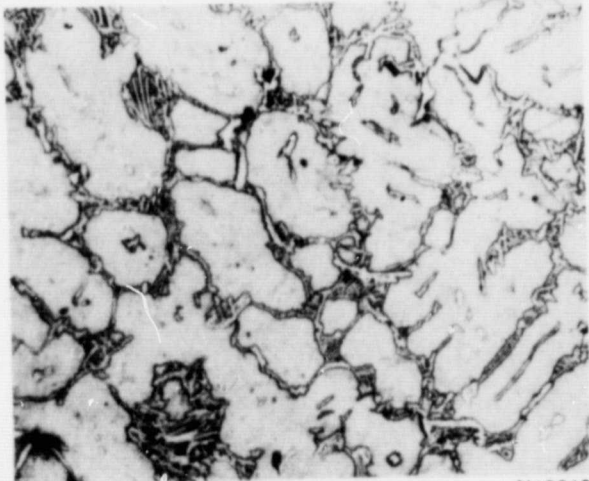
Figure 2-10. Optical Micrographs of Group 2 Alloys,
Etchant: Fry's Reagent, Magnification: 200X





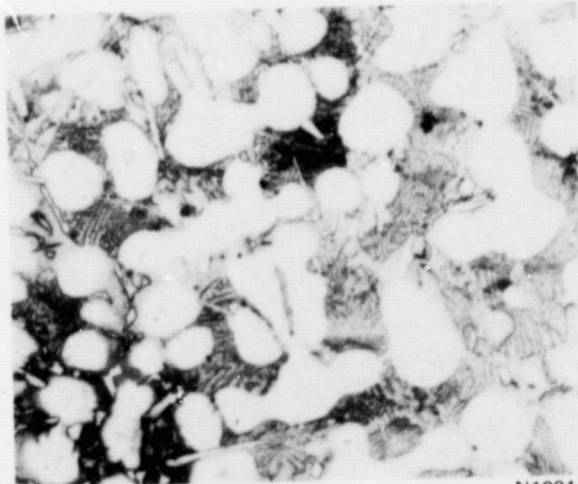
Alloy 3-1, AF-71

N10012



Alloy 3-2

N10013



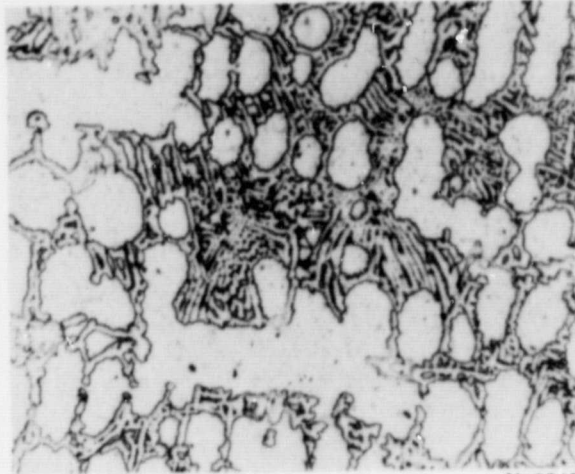
Alloy 3-2

N10014

F-33234

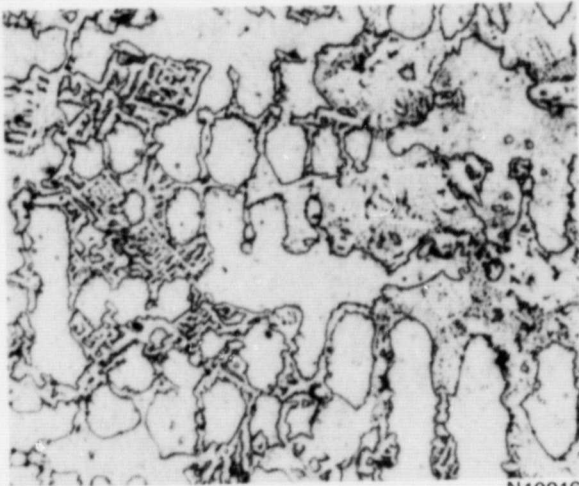
Figure 2-11. Optical Micrographs of Group 3 Alloys,
Etchant: Fry's Reagent, Magnification: 200X





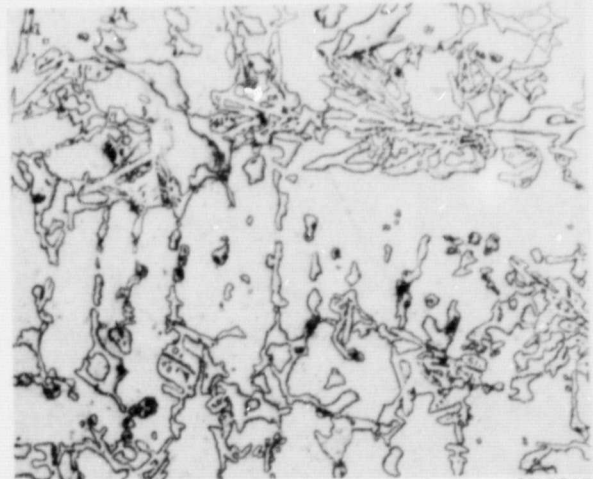
Alloy 4-1

N10015



Alloy 4-2

N10016



Alloy 4-3

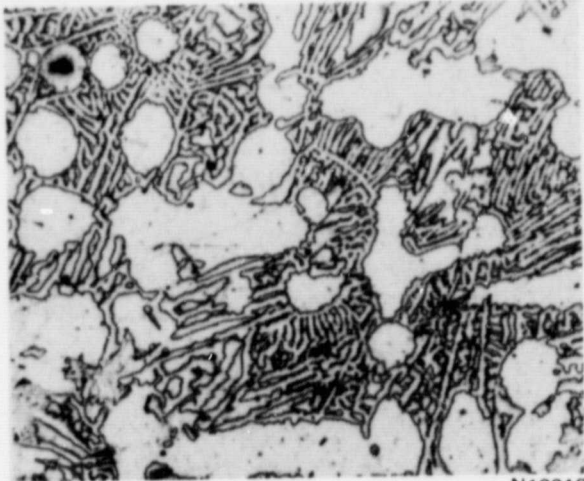
N10017

F-33233

Figure 2-12. Optical Micrographs of Group 4 Alloys,
Etchant: Fry's Reagent, Magnification: 200X

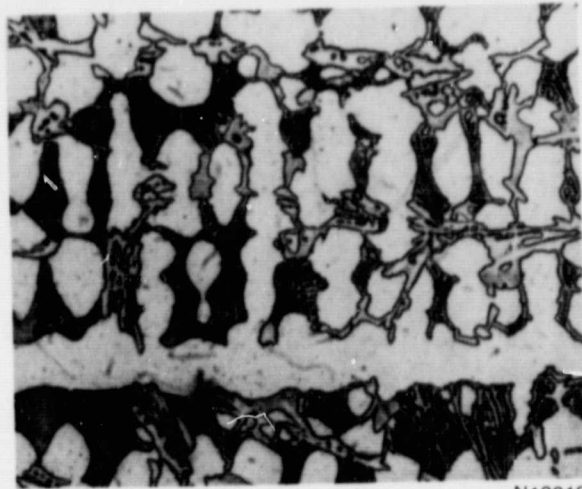


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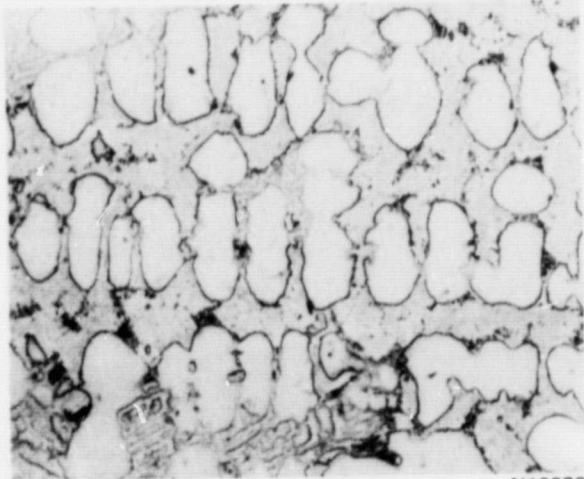
Alloy 4-4

N10018



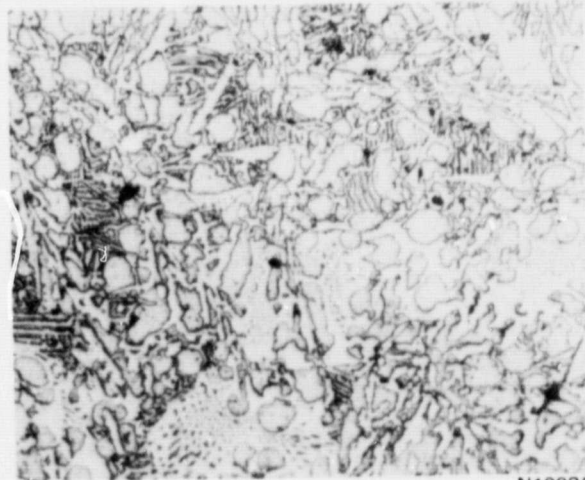
Alloy 4-5

N10019



Alloy 4-6

N10020



Alloy 4-7

N10021

F-33232

Figure 2-12. (Continued)



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TABLE 2-7

HARDNESS AND STRESS-RUPTURE DATA

Alloy No.	Heat No.	Condition	Hardness, Rockwell "C"	Stress-Rupture Life, Hours at 830°C, 200 MPa (E)
1-1	N10001	As-cast 100 h/1200°F	22.5 32.5	X
1-2	N10002	As-cast 100 h/1200°F	23.5 36.5	X 20+
1-3	N10003	As-cast 100 h/1200°F	21 33	X
1-4	N10004	As-cast 100 h/1200°F	(R _B 100) 33	X
1-5	N10005	As-cast 100 h/1200°F	25 40	X 21+
2-1	N10006	As-cast	(R _B 96.5)	3.9, 3.6
2-2	N10007	As-cast	25.5	22.5*, 38.4
2-3	N10008	As-cast	31	49.4*, 67+
2-4	N10009	As-cast	29	43.9+
2-5	N10010	As-cast	27	12.4, <1
2-6	N10011	As-cast	31	42.9
3-1	N10012	As-cast 1 h/1950°F/WQ +16 h/1300°F 16 h/1300°F	28 30.5	X
3-2	N10013	As-cast 16 h/1300°F	26.5	X
3-3	N10014	As-cast 16 h/1300°F	35.5	X
4-1	N10015	As-cast	25.5	136, 21.4*
4-2	N10016	As-cast	31.5	167+, 22.1*
4-3	N10017	As-cast	26.5	167+
4-4	N10018	As-cast	23.5	62.8, 24+
4-5	N10019	As-cast	31.5	63.1, 14.0
4-6	N10020	As-cast	30.5	67.3
4-7	N10021	As-cast	36.5	15.3, 32.7

LEGEND:

E 329 hr at 830°C is equivalent in Larson-Miller parameter to 5000 hr at 775°C

X No test to be conducted in this condition

* Test terminated due to equipment malfunction. Specimen not broken.

+ Test continuing



as carbide formers, is the only exception to the obvious dendritic solidification pattern of all those melted to date.

Stress-rupture life of Alloys 4-2 and 4-3 is the best obtained to date in this program. Specimens have run over 167 hr and are still running. Life of 329 hr at 830°C is equivalent in Larson-Miller parameter to the target life of 5000 hr at 775°C.

2.3 TASK IV, REPORTING

The oral presentation of the work Plan was made at NASA-Lewis Research Center by the ACC project manager on September 26, 1980. The selection of alloy approach was discussed in detail and was agreed to, with minor modifications by the NASA project manager.



3. CURRENT PROBLEMS

The program is currently behind schedule by approximately three weeks, due to difficulties encountered in conducting stress-rupture testing. The problems with equipment have been addressed and testing is proceeding with eight stress-rupture stands. Consideration is being given to conducting a portion of the stress-rupture testing at an outside facility, if necessary.

No difficulties have been encountered with other phases of the work.



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4. WCRK PLANNED

During the next month, stress-rupture testing will continue. The remelt heats will be evaluated and new composition variations which result from current testing will be melted and evaluated. This work will culminate in the selection of ten candidate alloy compositions for more detailed evaluation in the next portion of Task I of this program.



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