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DEVELOPMENT OF A NITRIDE DISPERSION STRENGTHENED (NDS) METALLIC ALLOY FOR HIGH-TEMPERATURE RECUPERATORS

June 1985

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FINAL REPORT

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## ABSTRACT

→ The objective of this program was to demonstrate the feasibility of using nitride dispersion strengthened (NDS) stainless steel in fabricating a recuperator for advanced gas turbine engines. Test results showed an alloy--designated NDS 300--to have tensile properties comparable to those of Inconel 625 at temperatures up to 1650° F, and at higher temperatures the properties of the NDS alloy exceeded those of the Inconel 625. However, creep test results showed a three-fold improvement in strength of NDS 300 over Inconel 625 at temperatures above 1500° F. The NDS material demonstrated adequate formability and joinability by brazing with a filler metal of nominal composition Ni-19Cr-10Si (J8100). The same filler metal proved to be a good coating for high-temperature oxidation resistance. Tests on specimens prepared to a typical plate-fin recuperator configuration confirmed the strength of the brazing alloy and demonstrated the marked superiority of the NDS material over Inconel 625.

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PREFACE

This is the final technical report under Contract Number DAAG46-82-C-0081 covering the period 1 October 1982 to 30 September 1984. The program was sponsored by the Advanced Concepts Team of Department of the Army and was monitored by the Army Materials and Mechanics Research (AMMRC), Watertown, MA. Dr. P. J. Fopiano of AMMRC was the Contracting Officer's Technical Representative (COTR).

At the AiResearch Manufacturing Company (Division of Garrett Corporation), Dr. L. E. Kindlimann was the principal investigator, responsible for the initiation, carrying out, and reporting on the development of dispersion strengthened stainless steels for recuperator applications.

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## CONTENTS

<u>Section</u>		<u>Page</u>
1.	INTRODUCTION	1-1
	1.1 Overall Program Concepts	1-1
	1.2 Program Goals	1-2
2.	TECHNICAL PROGRESS	2-1
	2.1 Recuperator Material Requirements	2-1
	2.1.1 General Considerations	2-1
	2.1.2 Current Industry Status	2-2
	2.2 Nitride-Dispersion-Strengthened (NDS) Alloys	2-2
	2.3 Task 1, Material Evaluation	2-4
	2.3.1 Task 1a, Process Modification	2-8
	2.3.2 Task 1b, Alloy Modifications	2-25
	2.3.3 Cost Analysis	2-38
	2.4 Task 2, Brazing Development	2-43
	2.5 Task 3, Coating Development	2-54
	2.6 Task 4, Fabrication Development	2-60
	2.7 Task 5, Panel Testing	2-71
3.	GENERAL DISCUSSION	3-1
4.	SUMMARY	4-1
5.	CONCLUSIONS AND RECOMMENDATIONS	5-1
6.	REFERENCES	6-1
<u>Appendix</u>		
A	NITRIDING RUN DATA	A-1
B	CREEP/RUPTURE TEST DATA FOR ALL MATERIALS INDEXED BY TEST NUMBER	B-1
C	TENSILE TEST DATA FOR NDS 300 MATERIALS	C-1

## ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
2-1	Schematic of Nitride Strengthening Process	2-5
2-2	Yield Strength Improvement as a Result of Nitriding an 18Cr-12Ni-2Ti (Modified Type 321) Stainless Steel	2-5
2-3	Master Plot Comparing Projected 1-Percent Creep Behavior of NDS Alloys to Commercial Sheet Candidate Recuperator Materials	2-6
2-4	Program Flow Chart	2-7
2-5	Minimum Time to Through-Nitride 5-Mil (0.005-in.) Thick Material Versus Nickel Content (Alloys Contain 18-20 Cr and 2 Ti, Balance Fe)	2-9
2-6	Laboratory Retort Used for Nitriding and Denitriding Treatments	2-12
2-7	Yield Strength of Bare and Braze-Coated NDS 300 and IN625 as a Function of Temperature	2-19
2-8	Comparison of Creep/Rupture of NDS 300 and Inconels 617 and 625	2-20
2-9	Comparison of Microstructures of NDS 300 and Inconel 625 Creep Test Specimens (Magnification, 200X)	2-21
2-10	Comparison of Creep Curves for Braze-Coated NDS 300 and IN625	2-23
2-11	Comparison of Modified Nitride Treatment B Microstructures to the A Treatment Structure; All B Samples Were Taken from Broken Tensile Test Specimens of Table 2-8 (Magnification, 200X)	2-24
2-12	Modified Schaeffler Diagram for Austenite-Martensite-Ferrite Phase Balance in Austenitic Stainless Steel Weldments (Reference 5)	2-28
2-13	Thirty-Gram Ingot and Comparison of Nitrided Structures of Two NDS 200 Compositions Selected to NDS 300 Strip (Magnification, 250X)	2-31
2-14	Microstructures of NDS 200 (Heat RV-9307) Versus Tensile Ductility (Magnification, 250X)	2-34

## ILLUSTRATIONS (Continued)

<u>Figure</u>		<u>Page</u>
2-15	Comparison of NDS 200 to IN625 in Creep/Rupture Testing of Braze-Coated Material (IN625 Data from Table 2-5 and Figure 2-8)	2-42
2-16	Cross Sections of Braze-Coated Cyclic Oxidation Test Specimens (Magnification, 250X)	2-48
2-17	Braze Peel Test Specimen and Modes of Failure--1800°F Test (Magnification, 75X)	2-51
2-18	Sketch of Brazed Fin-Stud Pull Test Assembly (Not to Scale)	2-53
2-19	Master Plot Comparing 1-Percent Creep Behavior of NDS Alloys After Aluminizing to NDS 300 Baseline Curve	2-57
2-20	Options for Achieving Environmental Protection, Singly or in Combinations	2-62
2-21	Representative Micrographs of Cyclic Oxidation Test Specimens--See Table 2-29 (Magnification, 200X)	2-64
2-22	Example of Possible Forming Requirements Associated with Recuperator Fabrication	2-66
2-23	Ball Punch Deformation Tests	2-67
2-24	Rack Used for Nitriding 36-in.-Long Strip	2-69
2-25	Sample Microsections of NDS 300 Fin Material (Magnifications as Shown)	2-72
2-26	Results of Plate-Forming Experiments of NDS Materials (See Table 2-32)	2-74
2-27	Assembly of Heat Exchanger Panels from NDS 300 Plates and Fins	2-75
2-28	Sections of NDS 300 and IN625 Test Panels (All Were Brazed with AMI 100 Filler Metal Except (e) Which Was Brazed with MBF-50 Foil)	2-79
2-29	NDS 300 Panel Brazed with AMI 100 Filler Metal and Tested for 823.1 Hours at 1650°F with 400 psig Argon	2-80
2-30	NDS 300 Panel Brazed with AMI 100 Filler Metal and Tested for 973.1 Hours at 1725°F with 200 psig Argon (Did Not Fail--Photograph Shows Thermocouple Still in Place)	2-81

ILLUSTRATIONS (Continued)

<u>Figure</u>		<u>Page</u>
2-31	Stud-Fin Specimens: Left--Studs, Fin, and MBF-50 Foil for Assembly; Center--Unfailed, Chromized 1650°F/2 ksi Creep Test Specimens that Were Terminated after 1191 and 1195 hr; Right--Chromized 1725°F/2 ksi Creep Test Specimen that Failed at 1134 hr (See Table 2-36)	2-85
2-32	Cross Sections of Stud-Fin Specimens, Comparing IN625 to NDS 300 after Stress Rupture Testing at 1800°F--Item (f) was at 1725°F--Under a Fin Stress of 2 ksi for the Times Shown (Magnification, 25X)	2-86

## TABLES

<u>Table</u>		<u>Page</u>
2-1	Nominal Compositions of Commercial Alloys	2-3
2-2	Analyses of NDS 300 Starting Materials	2-10
2-3	Results of Nitride Processing Studies on 0.010-in.-Thick NDS 300 Alloy	2-13
2-4	Summary of Tensile Test Results on 0.010-in.-Thick NDS 300 Material	2-15
2-5	Summary of Tensile and Creep/Rupture Results on 0.010-in.-Thick IN625	2-16
2-6	Summary of Tensile and Creep/Rupture Results on 0.010-in.-Thick IN617	2-17
2-7	Summary of Creep/Rupture Test Results on 0.010-in.- Thick NDS 300 Material	2-18
2-8	Results of Final Nitride Optimization Processing Studies on 0.010-in.-Thick NDS 300 Alloy	2-26
2-9	Room Temperature Tensile Properties of Terminated Creep Test Specimens	2-27
2-10	Nominal Aim Compositions of 30-Gram Button Melts	2-29
2-11	Analyses of Initial NDS 200 Materials	2-33
2-12	Results of Nitride Processing Studies on 0.010-in.- Thick NDS 200 Alloys	2-36
2-13	Results of Cold-Rolling Experiments on NDS 200	2-37
2-14	Commercial Melt Tolerances on Elements in Stainless Steels	2-38
2-15	Analysis of Heat RV-9408 (NDS 200 Base)	2-39
2-16	Results of Final Nitride Optimization Processing Studies on 0.010-in.-Thick NDS 200 Alloy	2-40
2-17	Summary of Tensile and Creep/Rupture Results on 0.010-in.-Thick NDS 200 Material	2-41
2-18	Brazing Alloy Screening Tests on NDS 300	2-44

TABLES (Continued)

<u>Table</u>		<u>Page</u>
2-19	Brazing Alloys Selected for Further Study	2-45
2-20	Results of Cyclic Oxidation Testing of Brazing Alloys	2-46
2-21	Test Results on Braze-Coated NDS 300	2-50
2-22	Braze Peel Tensile Test Data at 1800°F	2-52
2-23	Test Results on the First Set of Chromized NDS 300	2-54
2-24	Mechanical Test Results on the Second Set of Coated Material	2-55
2-25	Cyclic Oxidation Test Results on the Second Set of Coated Materials	2-56
2-26	Test Results for Chromized Materials	2-58
2-27	Test Results for Braze-Coated Aluminized Materials	2-59
2-28	Test Results for Incoloy 800H	2-61
2-29	Cyclic Oxidation Test Results on Coated Materials	2-63
2-30	Test Results from Fabrication Ductility Studies	2-68
2-31	Test Results on Stretch-Flattened NDS 300	2-70
2-32	Recuperator Plate Formability Test Results	2-73
2-33	Test Results on Single-Sandwich Heat Exchanger Panels Brazed with MBF-50 (Metglas) Foil	2-77
2-34	Test Results on Single-Sandwich Heat Exchanger Panels Brazed with AMI 100 Tape	2-78
2-35	Tensile Test Results on Stud-Fin Specimens	2-83
2-36	Creep/Rupture Test Results on Stud-Fin Specimens	2-84

## 1. INTRODUCTION

This document describes the results of a program to develop a low-cost metallic alloy for use in advanced gas turbine engine recuperators. It is submitted in response to CDRL Sequence A004, "Final Technical Report," covering the activities performed during the period October 1, 1982 to September 30, 1984. This program was conducted by the AiResearch Manufacturing Company, a division of The Garrett Corporation, for the U.S. Army Advanced Concepts Team (ACT), Dr. Charles Church, director. It was administered by the U.S. Army Mechanics and Materials Research Center (AMMRC), Watertown, Massachusetts, under Contract No. DAAG46-82-C-0081.

The broad objective of the program was to provide for increased military vehicular gas turbine efficiency by allowing higher recuperator inlet temperatures through use of nitride-dispersion-strengthened (NDS) metal alloys with their inherently superior high-temperature capability. Specific program goals included development and optimization of the NDS alloys with regard to composition, processing, and recuperator fabrication procedures. The principal investigator for AiResearch was Dr. L. E. Kindlimann of the Materials Engineering Department. Dr. Paul Fopiano of AMMRC was technical monitor for the Army.

### 1.1 OVERALL PROGRAM CONCEPTS

There are several advantages to be gained from the use of recuperators, the most notable being higher engine thermal efficiency and the attendant fuel savings. Exceptional part-load economy, a highly desirable characteristic for vehicular propulsion, also can be obtained when a recuperator is used in an engine with variable turbine geometry. Such improvements cannot be attained by advancements in engine component performance alone, at least not on a near-term basis. To minimize recuperator size and weight, the use of high-strength metals is necessary. As turbine inlet temperatures increase, so do recuperator inlet temperatures, imposing further demands on recuperator materials.

Dispersion-strengthened metals have long been recognized for their excellent high-temperature creep strength, and offer the most promise for extending the operating temperature of a metal recuperator to temperatures as high as 1800°F. NDS metals appear especially desirable for a military application because they avoid use of critical elements such as cobalt and tungsten, and are low in chromium and nickel. In addition, NDS metals are readily producible at recuperator gages and are fabricable into a heat exchanger, offering a potential for significantly lower component costs compared to those made from the conventional nickel-based and cobalt-based superalloys.

The results of this program are directly applicable to land vehicles where emerging gas turbine engine technology requires a material with a high

operating temperature capability. For example, while recuperator metal temperatures of about 1500°F would be common with these engines when operating at high power levels, a temperature of about 1750 °F would be encountered under a low power condition (and perhaps even hotter under a free turbine stall condition wherein the recuperator would experience nearly full gas generator turbine temperature and pressure). The NDS metal alloy has the capability for service at temperatures in the 1700° to 1800°F range, easily surpassing existing sheet alloys in creep strength. This is accomplished without the extensive use of strategic elements and results in costs that are projected to be about 30 percent below conventional high-temperature superalloy sheet materials.

If NDS alloys are to be used in military vehicle recuperators, several critical steps are necessary. First, the alloy base must be optimized with respect to composition to provide: (a) an economical rate of production, and (b) minimum strategic alloy content. Second, associated manufacturing methods must be studied, particularly with regard to brazing alloy selection. This is because the filler metal must hold the heat exchanger together at high temperatures without markedly reducing parent metal strength properties. Third, because the NDS alloys are intrinsically low in chromium (a strategic alloying element) some method of protecting the unit from high-temperature oxidation and hot corrosion must be evolved.

## 1.2 PROGRAM GOALS

The objectives of this program were to:

- (a) Extend development of the nitride strengthening concept to heat exchanger applications, specifically gas turbine recuperators that encounter high operating temperatures
- (b) Evolve an economical method of material manufacture
- (c) Evaluate associated heat exchanger manufacturing methods
- (d) Provide design data for the NDS material

The overall goal was to provide for increased military vehicle gas turbine engine efficiency by allowing higher recuperator inlet temperatures. To meet program requirements, the following tasks were established:

Task 1a, material evaluation, process modifications

Task 1b, material evaluation, alloy modifications

Task 2, brazing development

Task 3, coating development

Task 4, fabrication development

Task 5, panel testing

In accordance with the work statement, the basic requirement of Task 1, material evaluation, was the determination of process controls necessary to consistently obtain a strong NDS material in a cost-effective manner. In Task 2, brazing development, commercially available filler metals were to be evaluated for use with the NDS alloy in fabricating a plate-fin recuperator. Task 3, coating development, was directed toward evaluating state-of-the-art diffusion coatings for high-temperature protection of the recuperator. Task 4, fabrication development, and Task 5, panel testing, were the final steps in evaluating the NDS material for fabrication into a recuperator. Data obtained from tests of heat exchanger panels in Task 5, combined with basic property data from Task 1, provide a base for future recuperator design. The results of each of these tasks are described in Section 2. Overall, work on the program proved to be largely successful.

## 2. TECHNICAL PROGRESS

For coherency, before beginning with the detailed discussion on the technical accomplishments of each task, background information on recuperator material requirements will be summarized. A brief introduction to the basic technical concepts of the NDS alloy system also is presented.

### 2.1 RECUPERATOR MATERIAL REQUIREMENTS

#### 2.1.1 General Considerations

In selecting materials for a high-temperature recuperator, a number of factors must be considered. These include mechanical properties (ductility plus tensile and creep strength), environmental resistance (oxidation and hot corrosion), fabricability (formability, brazeability, or possibly weldability), and cost. Physical properties such as thermal expansion, conductivity, and modulus of elasticity also must be considered where thermal stresses and performance are concerned. However, the impact on thermally related behavior is generally slight within a given class of materials, as differences in physical properties do not vary significantly between individual alloys.

For engine service, mechanical strength (for both pressure and thermal stresses) and oxidation/hot corrosion resistance are very important. Resistance to low-cycle fatigue (LCF) caused by thermal transient stresses will be influenced by ductility if the material is subjected to stresses above the elastic limit. Because recuperated engines generally run at pressure ratios of less than 12:1, material strength in the manifold sections is usually the governing criterion for pressure containment. Thus, fins are sized primarily for thermal conductivity and are only lightly stressed. Thermal stresses, especially during transient conditions at startup, can exceed pressure stresses and be especially damaging, even though their duration is short. Hence, high yield and creep strength are very desirable. Most candidate recuperator materials have sufficient strength to resist damage by ingested material. For example, foreign object damage has not been found to be a problem in recuperators; this is because gas velocities (by design) are always below Mach 0.2.

Even though military vehicle design lives typically are short (i.e., on the order of several thousand hours), maximum metal losses due to oxidation and hot corrosion damage must be considered, particularly where operating temperatures exceed 1500°F. Recuperators are generally built of light-gage materials where a corrosive attack of only a few mils\* can result in loss of structural integrity, or, at the least, can cause significant degradation of

\*A mil, as used in this report, is equal to 0.001 in.

heat transfer capability. Sulfur-containing exhaust gases are to be expected, since most fuels contain sulfur. Salts also may be present, primarily from ingestion of contaminated air such as might occur in close proximity to ocean water. Therefore, exposed surfaces must either be inherently resistant to high temperature environmental attack, or must have a protective coating applied after fabrication.

For fabrication, the material must be sufficiently ductile to allow any necessary forming. The material also must be brazeable, i.e., the filler metal must wet the surface. Where the alloy being brazed contains more than about 1 percent uncombined aluminum plus titanium (i.e., where these metals are not present as a carbide or nitride) nickel plating becomes necessary to achieve acceptable furnace brazing. The plating process is expensive and requires very close control to ensure strong brazed joints.

### 2.1.2 Current Industry Status

Current recuperator technology extends to about 1550°F by using Inconel alloy 625 above 1300°F--the point where stainless steels begin to lose usable strength.\* Of the conventional superalloys, Haynes Stellite 188 and Inconel alloy 617 have been reported to have the highest creep-rupture strength for materials that can easily be reduced to the required form and thickness for lightweight heat exchangers. While some typical wrought gas turbine alloys such as Rene 41 and Udimet 700 are reducible to sheet, the cost is prohibitive when compared to the strength increment achieved. Thus, the best of the conventional heat exchanger materials are significantly weaker than a typical cast turbine alloy such as IN100. Expressed in terms of temperature at equivalent stresses, the heat exchanger alloys suffer a 200° to 300°F temperature disadvantage over cast material. Even if one were to attempt to use the higher-strength, precipitation-hardenable alloys such as Rene 41, it is doubtful that reported property values would be achieved. This is because complex, high-performance heat exchangers are not readily amenable to the complicated heat treatments required to develop strength.

In addition to processing costs incurred in reducing high-temperature alloys to appropriate gages for recuperator applications, the scarcity of strategic elements must be considered. In particular, molybdenum, cobalt, and tungsten are very expensive, and in times of shortages often must be bought on the free market where the asking price can be several times that of the producer's price. This problem also exists with nickel and chromium, but to a lesser extent; it is still desirable to minimize use of these elements, however, because both are imported, with chromium being an extremely important political issue. (The United States is self-sufficient in only molybdenum and copper; all other metals are imported to at least some degree, including iron and aluminum.)

## 2.2 NITRIDE-DISPERSION-STRENGTHENED (NDS) ALLOYS

Typically, dispersion-strengthened metallic alloys exhibit excellent creep and rupture characteristics in high-temperature service. These

\*Compositions of alloys referenced are given in Table 2-1.

TABLE 2-1  
 NOMINAL COMPOSITIONS OF COMMERCIAL ALLOYS\*

Alloy	Weight Percent														
	C	Mn	Si	Cr	Ni	Co	Mo	W	Cb	Ti	Al	B	Zr	Fe	Other
Inconel 625	0.05	0.25	0.25	21.5	Bal	--	9	--	3.65	0.2	0.2	--	--	2.5	--
Haynes 188	0.10	0.75	0.4	22	22	Bal	--	14	--	--	--	--	--	1.5	0.08 La
Inconel 617	0.07	--	--	22	Bal	12.5	9	--	--	--	1	--	--	--	--
Rene' 41	0.09	--	--	19	Bal	11	10	--	--	3.1	1.5	0.005	--	--	--
Udimet 700	0.08	--	--	15	Bal	18.5	5.2	--	--	3.5	4.3	0.03	--	--	--
IN 100	0.18	--	--	10	Bal	15	3	--	--	4.7	5.5	0.014	0.06	--	1 V
Type 321	0.06	0.75	0.5	18	10.5	--	--	--	--	5XC	--	--	--	Bal	--
Type 201	0.10	6.5	0.5	17	4.5	--	--	--	--	--	--	--	--	Bal	--
Type 409	0.05	0.5	0.5	11	--	--	--	--	--	0.5	--	--	--	Bal	--
Type 310	0.10	0.75	0.5	25	20	--	--	--	--	--	--	--	--	Bal	--
Incoloy 800H	0.08	0.8	0.5	21	32	--	--	--	--	0.4	0.4	--	--	Bal	--
14 Cr-4Mo	0.03	0.5	0.3	14	--	--	4	--	0.6	--	--	--	--	Bal	--

\*Listed in order mentioned in text.

materials offer the most promise for extending the operating temperature of a metal recuperator to 1800°F and above. The high strength and stability of dispersion-strengthened alloys are conferred by the incorporation of a uniformly spaced array of a very fine and relatively insoluble (inert) dispersoid, such as an oxide, nitride, or carbide. Although the dispersoid will tend to grow and become less finely spaced during prolonged service at elevated temperatures, growth is generally quite slow. This is in contrast to conventional precipitation-hardened superalloys that lose strength at elevated temperatures because of dissolution of the precipitate.

Historically, dispersion-strengthened alloys have been expensive to produce because of the complex procedures needed to ensure uniform particle distribution. A technique has been identified as having potential for significantly lowering the cost of production (see Reference 1). This method utilizes a gaseous ammonia treatment to create a titanium nitride dispersoid within a stainless steel base. By nitriding a titanium-containing austenitic stainless steel at a high temperature, a stable TiN dispersoid is created in situ, leading to the pronounced strengthening inherent to dispersion-strengthened metals (see Figure 2-1). The nitriding process is analogous in almost all respects to the well-documented internal oxidation process (Reference 2), but is several orders of magnitude faster.

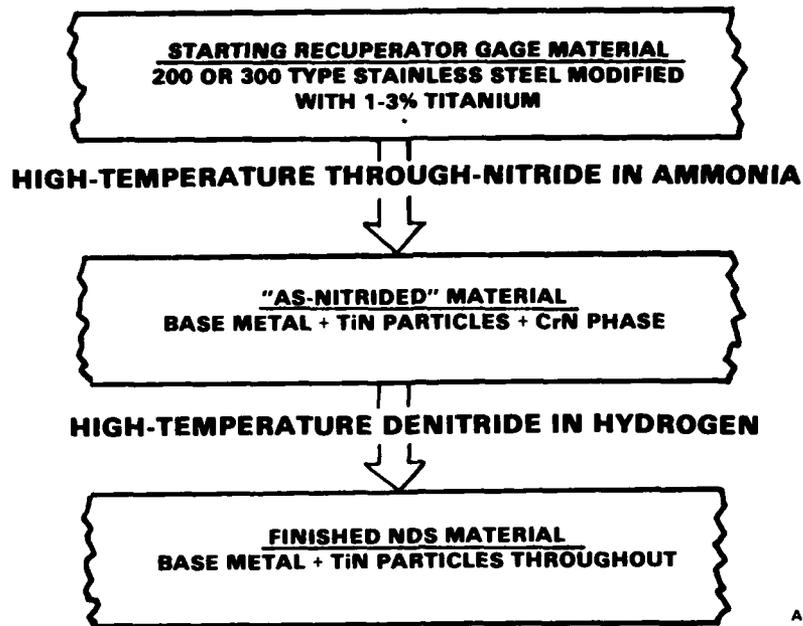
Applying the high-temperature nitriding treatment to a stainless steel of composition that is nominally 18Cr-12Ni-2Ti-Balance Fe (a low-carbon, high-titanium modification of Type 321 stainless steel) results in improved mechanical strength, as shown in Figure 2-2. Creep properties are shown in Figure 2-3, where the Larson-Miller master curve technique is used to compare commercial sheet alloys with projections for the NDS alloy taken from References 3 and 4 and from preliminary testing prior to the inception of this program. Curves for other materials were plotted from vendor and literature data. For prolonged service above 1500°F, the NDS alloys have significantly higher projected creep strength, with the margin becoming even greater as the time-temperature parameter increases.

Data used to generate the curves given for NDS alloys in Figures 2-2 and 2-3 were obtained primarily from laboratory-sized samples that were produced individually. Creep data above 1500°F were from fairly short time tests (typically, several hundred hours) or were estimated from rupture test results. There are a number of major prerequisites to the successful implementation of an NDS alloy to the production of a new high-operating-temperature recuperator. These prerequisites form the basis for the work on the individual tasks shown in Figure 2-4 and described below.

### 2.3 TASK 1, MATERIAL EVALUATION

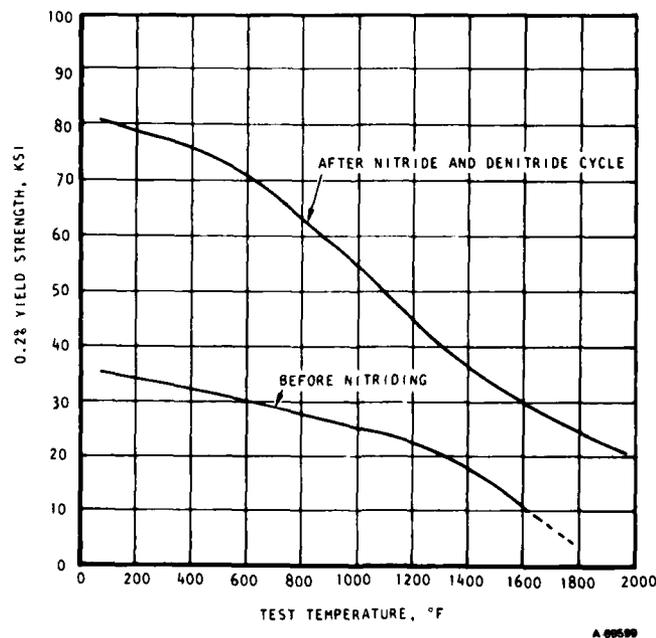
For successful commercialization of NDS alloys, these materials must be capable of being produced in quantity,\* either in large batches on a

\*Recuperators can weigh hundreds of pounds per engine and require a large amount of surface area for high effectiveness.



A-70482

Figure 2-1. Schematic of Nitride Strengthening Process



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Figure 2-2. Yield Strength Improvement as a Result of Nitriding an 18Cr-12Ni-2Ti (Modified Type 321) Stainless Steel (Reference 2)

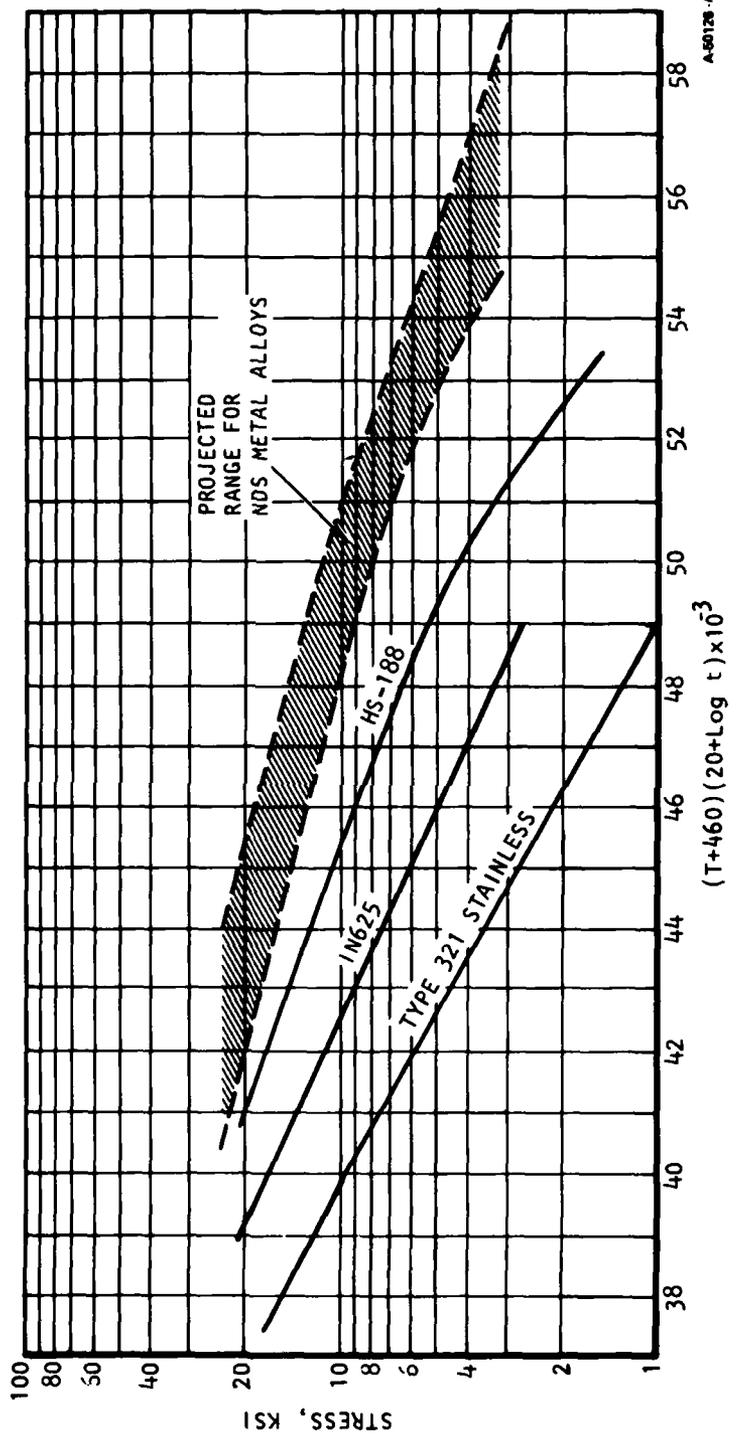
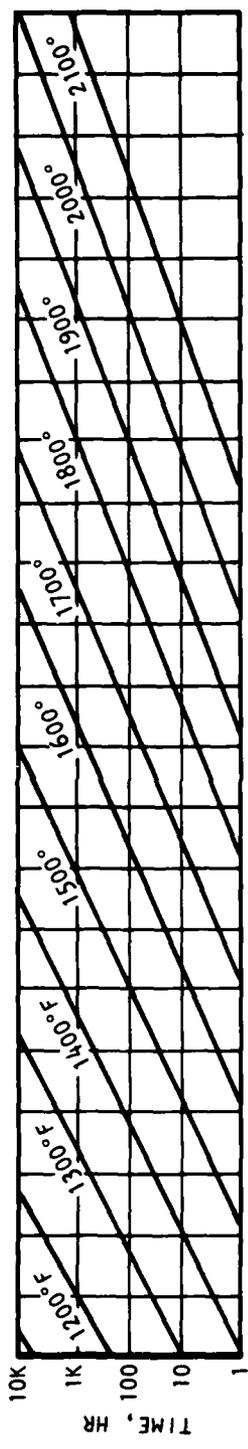
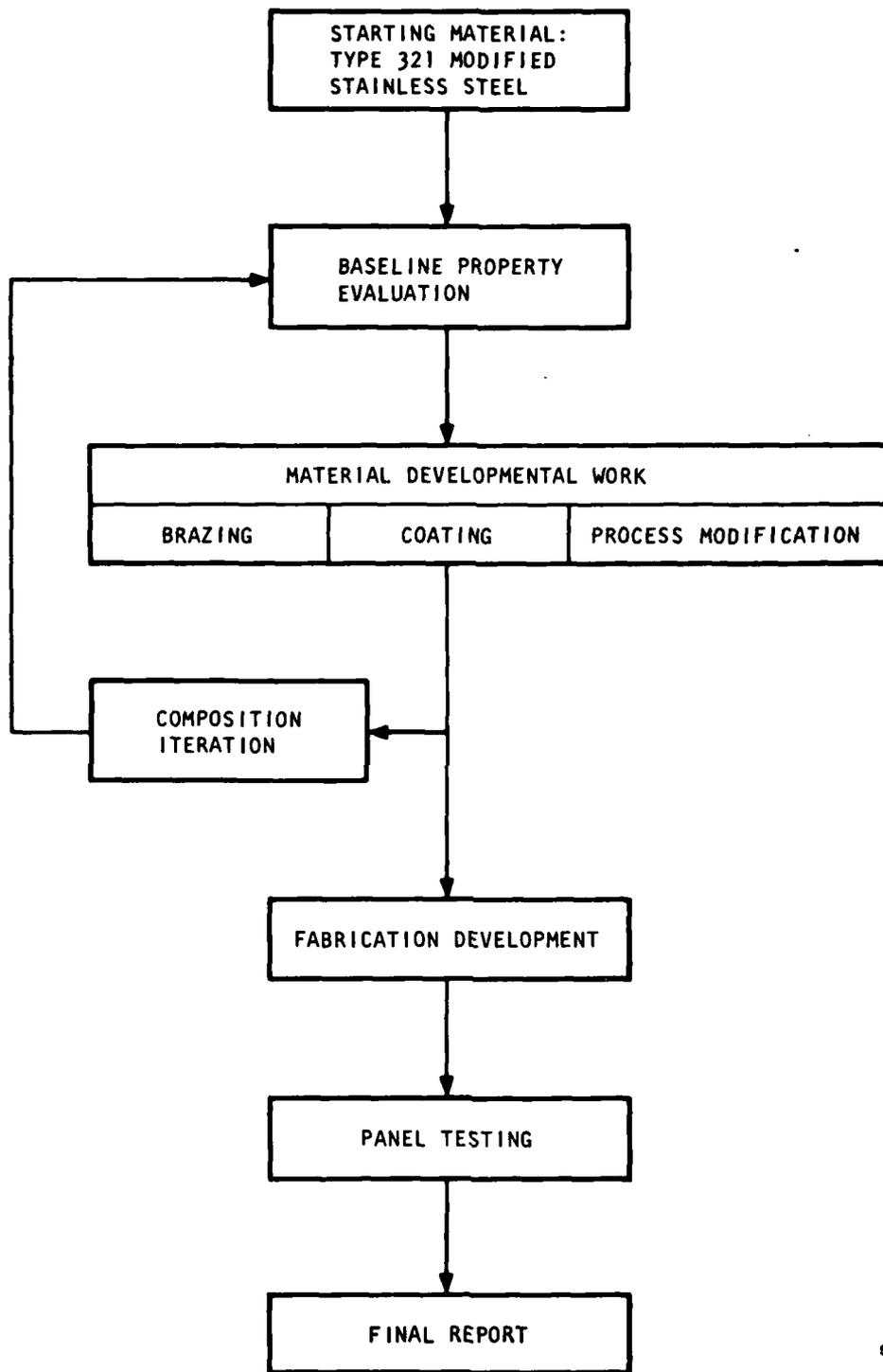


Figure 2-3. Master Plot Comparing Projected 1-Percent Creep Behavior of NDS Alloys to Commercial Sheet Candidate Recuperator Materials



8-42337

Figure 2-4. Program Flow Chart

continuous line, or by a combination wherein initial nitriding is done on a continuous basis and followed by batch denitriding. As shown in Figure 2-1, the denitriding process is used to eliminate chromium nitride particles that form during the initial operation and lead to a very brittle material. Earlier studies showed that up to 3 hr in hydrogen may be required to effect denitriding, making line operation very slow and uneconomical. Conversely, as shown in Figure 2-5 for iron-nickel alloys containing nominally 20Cr and 2Ti, 0.005-in.-thick alloys can be through-nitrided in a few minutes if the nickel content is held low.

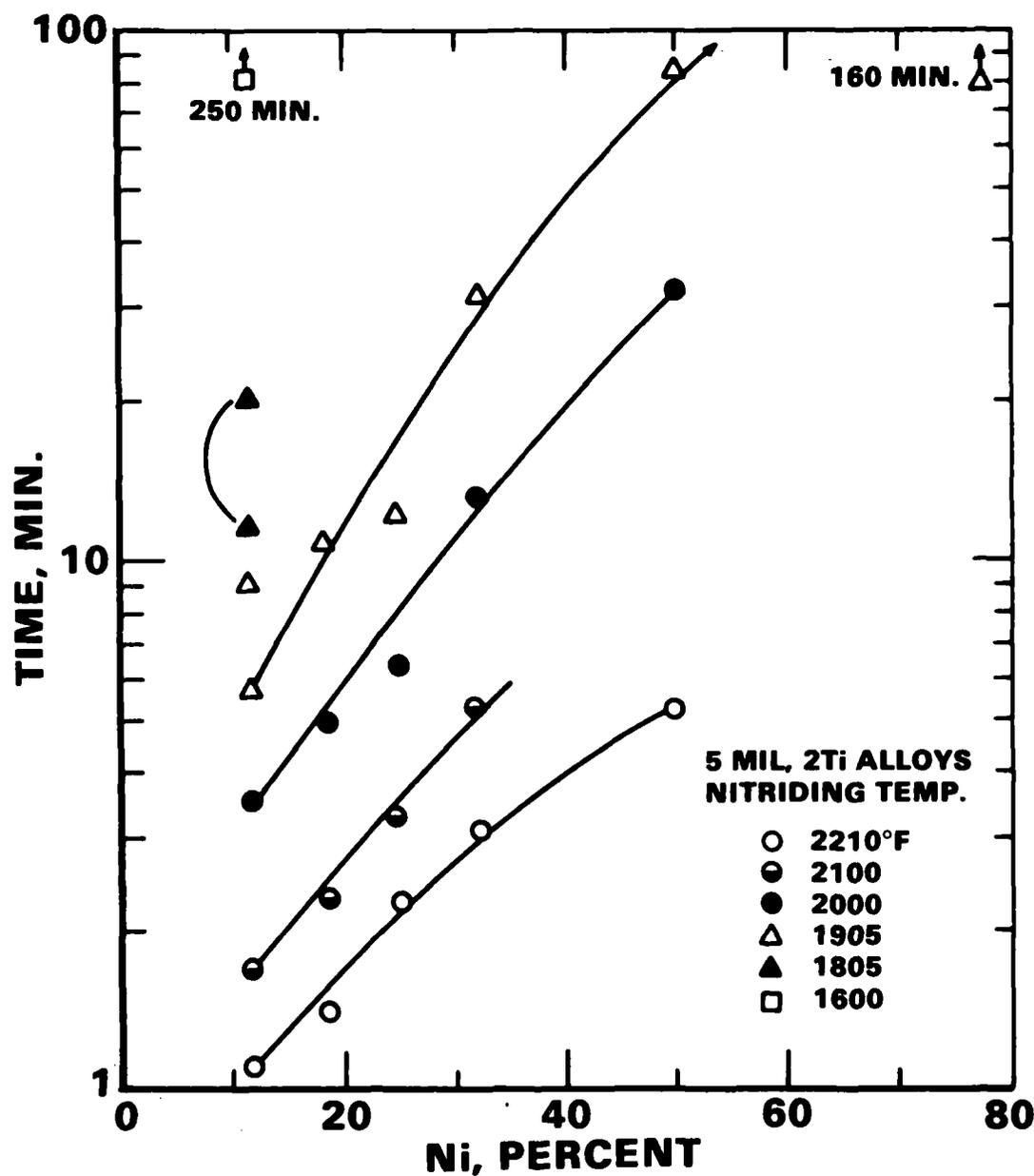
Times shown in Figure 2-5 would be typical for recuperator fin material. For plate material (sized 0.010-in.-thick for strength and fabricability), the times in Figure 2-5 must be increased. For example, it had been found previously that to ensure complete nitriding of the titanium-modified 18Cr-12Ni stainless steel described earlier, 30 minutes is required for treatment of 0.010-in. material at 1900°F. Unfortunately, this is a fairly long time for a continuous line process; however, the excellent properties noted in Figure 2-3 were derived from material nitrided at about 1900°F.

With this background, the work in Task 1 was directed at two potential methods for reducing processing time: (1) nitride only part of the way through and allow residual nitrogen in the surface of the material to complete the reaction while heating to denitride later, and (2) change the alloy composition, such as by reducing nickel content, to enhance the nitriding rate (see Figure 2-5). Consistent with the first approach was an attempt to reduce denitriding time significantly below the currently used 3 hr at 2000° to 2050°F. (Insufficient denitriding results in decreased ductility.) Evaluation was by tensile and creep testing. This approach was applied primarily to the aforementioned NDS 18Cr-12Ni-2Ti modified type 321 stainless steel; this material will often be referred to in this report as NDS 300.

The second approach, in addition to decreasing nitriding time, had a potential extra benefit in reduced levels of both nickel and chromium. (These two elements are tied together in stainless steels, where it is desirable to maintain the face-centered cubic, or austenitic, crystal structure for better high-temperature strength.) The basic modification was the substitution of manganese for nickel, as has been done commercially with development of the 200-series stainless steels and the Nitronic alloys (Armco Steel designation). Manganese also is known to increase the solubility of nitrogen in the base metal, which would directly increase the nitriding rate by establishing a higher diffusion gradient at the surface. The manganese-modified material will generally be referred to as NDS 200 in this report. Evaluation also was primarily by tensile and creep testing.

### 2.3.1 Task 1a, Process Modification

For this work on the modified type 321 alloy, nominally 0.010-in.-thick material was used from the earlier preliminary study conducted by AiResearch to determine the basic feasibility of applying this alloy to a high-temperature recuperator; a repeat heat later in the program was nearly an exact duplicate, as shown in Table 2-2. As noted above, a through-nitriding time of 30 min at 1900°F was used earlier. For the current program, initial



A-3548-A

Figure 2-5. Minimum Time to Through-Nitride 5-Mil (0.005-in.) Thick Material Versus Nickel Content (Alloys Contain 18-20Cr and 2Ti, Balance Fe)

TABLE 2-2  
ANALYSES OF NDS 300 STARTING MATERIALS

Element	Weight Percent in Heat	
	RV-7757	RV-9481
C	0.009	0.0089
Mn	0.49	0.50
P	0.007	0.023
S	0.004	0.0045
Si	0.77	0.68
Cr	18.05	18.17
Ni	12.02	12.08
Ti	1.90	1.90
N	not measured	0.0022
B	0.0004	0.0002
Pb	<0.0001	<0.0001
Sn	0.0019	0.0020
Bi	<0.00002	<0.00002

emphasis was on partial nitriding in the range 1900° to 2000°F, followed by batch denitriding, nominally at 2025°F for 3 hr, also as established earlier.

The laboratory nitriding/denitriding retort is shown in Figure 2-6. Two 150-lb bottles of liquid ammonia (NH<sub>3</sub>) are manifolded together to provide a sufficient flow of NH<sub>3</sub> gas. Prepurified hydrogen was supplied from a bottle, while high-purity argon (for flushing) was supplied from a liquid source. Flow rates were related to numerous factors, such as temperature and load size, and must necessarily be determined empirically for any given set-up.

### 2.3.1.1 Nitriding Trials

Test results from nitriding and denitriding studies on the NDS 300 alloy are summarized in Table 2-3.\* Except where noted, heatup was in ammonia. Where judged favorable by room-temperature tensile testing, i.e., yield strength of at least 80 psi with a minimum of 15-to 20-percent elongation to failure, creep testing was conducted at 1500°F under a stress of 20 ksi, and/or at 1650°F under a 15-ksi stress. These creep test results should be compared to a baseline, which is the lower NDS curve in Figure 2-3. Hence, life to 1-percent creep at 1500°F and 20 ksi should be at least 26 hr; at 1650°F and 15 ksi it should be at least 12 hr. (Early test conditions at 1500°F with 17 ksi and 1650°F with 14 ksi tended to result in excessively long times to 1-percent creep extension.) For simplicity in making this comparison and because recuperator design is typically based on 1-percent creep strength, rupture data have not been given here; full details of all creep testing are given, however, in Appendix B. Because of the wide variation in processing shown in Table 2-3, the rupture lives vary from only slightly longer than minimum 1-percent creep to more than 10 times longer. Likewise, elongation to failure in rupture varied from about 1.5 to 3.5 percent. Dispersion-strengthened alloys as a class have not been noted for high rupture elongation because the matrices are so strong that failure is primarily by grain boundary separation. This is compounded, of course, in thin-gage sheet. Many creep tests were terminated after 1- to 2-percent creep so that they could be tensile tested at room temperature in order to determine if there were long-term embrittling effects of stress and temperature. Results are given in paragraph 2.3.1.3.

The results of these tests (Table 2-3) showed that a variety of nitriding conditions could be used to produce material meeting the minimum, or baseline, creep strength requirements at 1500° and 1650°F. Processing at 1900°F for 15 to 25 minutes, or at higher temperatures to 2000°F for shorter times, appeared realistic and represented as much as a 50 percent decrease in time over that previously established at 1900°F. Of even greater significance, however, was a reduction in denitriding time from nominal 3 hr at 2025°F to 20 minutes at 2100°F as demonstrated by runs 243 and 247 (see the bottom of Table 2-3). In addition to property measurements, nitrogen analyses confirmed that denitriding was complete, i.e., a maximum of 1200 ppm of nitrogen was left in solution, which is well within commercial limits for 300-series stainless steels. This suggests possible continuous line processing for both nitriding and denitriding.

\*For a complete list of individual nitriding runs see Appendix A.

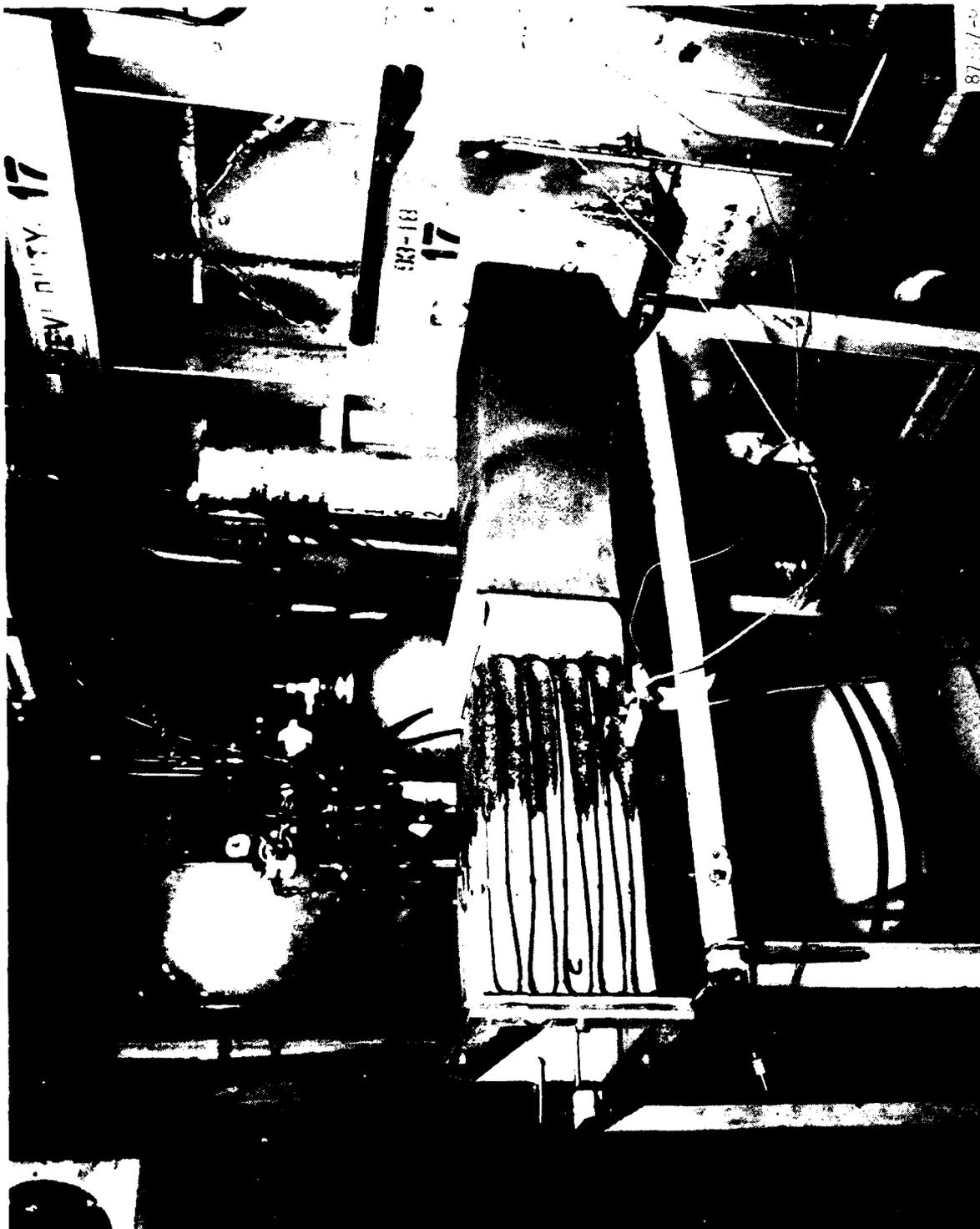


Figure 2-6. Laboratory Retort Used for Nitrifying and Denitrifying Treatments

TABLE 2-3  
RESULTS OF NITRIDE PROCESSING STUDIES ON 0.010-IN.-THICK NDS 300 ALLOY

Nitride Test Code No.*	Nitridding		Denitridding		Room-Temperature Properties			Creep Test Data		
	Temperature, of	Time, min	Temperature, of	Time, min	0.2% Yield, ksi	Ultimate, ksi	% Elongation over 1 in.	Hours to 1% Extension***	15000F/20 ksi	16500F/15 ksi
105R(H)	1900	15	2025	180	86, 88	138, 139	17, 18	603 (566)	>160(14 ksi) (569)	
106R(H)	2000	10	2025	180	77, 78	133, 136	22, 20	189 (568)		
107R(H)	1950	25	2025	180	75	146	16			
108R(H)	1950	15	2025	180	86, 87	122, 137	23, 24			
110R(H)	1900	30	2025	180	72	143	17			
118R(H)	1900	25	2025	180	67, 75	137, 141	26, 22			
120R	1900	30	2025	180	77, 86	140, 143	20, 20			
124R	1900	30	2025	180	90, 93	150, 150	18, 18	1.5(16500F) (572)	228 (573)	
127T	1900	12.5	2025	180	82	147	22			
132T	2000	8.5	2025	180	70	122	11			
134R	2000	15	2025	180	89, 94	144, 142	20, 14	171 (574)	22 (575)	
135R	1900	30	2025	180	83	141	15	304 (578)		
144T(H)	2000	10	2025	180	71	139	25			
148T(H)	2050	10	2025	180	50	123	34			
149T(H)	1950	15	2025	180	72	136	24			
175T(H)	1900	15**	2025	180	55, 59	119, 121	28, 25			
179T	1900	15	2025	180	76, 74	139, 138	25, 27			
180T(H)	1900	15**	2025	180	76, 77	138, 138	26, 25			
187T(H)	1975	10	2025	180	60, 66	120, 126	22, 22			
232R	1900	15	2025	180	73	137	21			102 (704)
233R	1900	15	2025	60	89	134	14			120 (714)
234R	1900	15	2100	60	74	131	24			92 (716)
236R	1900	15	2025	120	83	129	16			94 (715)
237R	1900	15	2040	120	79, 84	137, 137	22, 17			19 (722)
243R	1900	15	2040	120	81, 88	140, 143	21, 17			120 (744)
247R	1990	10	2100	20	90, 94	149, 156	19	>603 (721)		51 (748)

\*R = Furnace retort T = Tubular retort (H) = Heated to temperature in hydrogen or dissociated ammonia (slow flow)

\*\*Ammonia flow was increased for run 180T(H)

\*\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B

Early in the program it was recognized that heatup rate of the heavy retort (Figure 2-6) was considerably slower than might be experienced with a continuous line process. These heatup rates were subsequently reduced considerably by oversetting the furnace temperature and using  $\text{NH}_3$  gas flow rate to control retort internal temperature. In the meantime, a long tubular retort was used to allow duplicate specimens to be plunged into the furnace hot zone, which resulted in heatup rates of about 1 to 1.5 min. Data so obtained are flagged in the footnote to Table 2-3; results, determined by tensile testing and metallography, did not appear to vary significantly between the retorts. Because of limited capacity in the tube, it was necessary to use the large retort to produce most of the material for the other portions of the test program.

### 2.3.1.2 Data Generation

Having established that shortened nitriding and denitriding cycles could be used to make NDS 300, the following three treatment conditions were selected for use in making large batches of samples for testing:

- A - Heat in ammonia to 1900°F, hold 16.5 min, denitride
- B - Heat in hydrogen to 1900°F, change to ammonia and hold 15 min, denitride
- C - Heat in partially dissociated ammonia to 1975°F, change to ammonia and hold for 10 min, denitride

These treatments encompass the possible variations on a continuous line (operator controllable). Treatments A and B would be most likely to give the high creep strength needed for recuperator plates. Treatment C would apply to fin material or to lower stressed plate material. To simulate a continuous line where the material is forward nitrided and reverse denitrided, all material was reheated in hydrogen after nitriding to the original nitriding temperature, stabilized, then heated quickly to 2100°F and held for 20 min.

Samples were tested for properties either bare or as-coated with AMI 100 filler metal to represent recuperator plate material as described in Sections 2.4 and 2.7. Similar samples of Inconel 625 and Inconel 617, two of the strongest commercially available sheet alloys, were prepared for direct comparison from 10-mil stock. Braze-coated samples were fused in a vacuum at 2100-2120°F for 10 min; bare samples of Inconels 625 and 617 were run through the same cycle, which is less severe than the aforementioned 20-min denitriding treatment for NDS 300.

Tensile and creep test results for this test series are summarized in Tables 2-4 through 2-7 and in Figures 2-7 through 2-9. Additional data are given in Appendixes B and C. Mid-range tensile values are plotted; note from Table 2-4 that the data for braze-coated material plotted in Figure 2-7 are based on the original parent metal thickness before coating. Generally, the A material is strongest, followed by the B and C materials, the last being the weakest. Also shown in Figure 2-7 is the original yield strength curve from Figure 2-2 for fully treated material. It is interesting to note that higher

TABLE 2-4

SUMMARY OF TENSILE TEST RESULTS ON 0.010-IN.-THICK ADS 300 MATERIAL\*

NDS Process Code	Test Temp., OF	Uncoated			Brazed Coated with 100 Filler Metall**		
		0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.
A	RT	89-113	145-160	18-23	89-94 (122-136)	111-131 (156-171)	6-19
	700	72-73	110-111	13-14	75-90 (108-124)	86-99 (122-135)	1-2.5
	1100	60-61	84-85	15-15	63-68 (93-108)	70-83 (110-122)	1-9
	1300	54-57	64-67	9-9	41-54 (58-71)	48-62 (68-82)	4-12
	1500	41-42	45-45	4-4.5	28-29 (38-42)	30-31 (40-45)	5-9
	1650	34-35	36-37	3-3	22-28 (30-37)	24-29 (32-39)	5-9
B	1800	29	31	2	18 (28)	20 (30)	3
	RT	75-94	135-150	19-25	82-84 (101-117)	117-137 (156-171)	8-12
	700	65	104	11	69-80 (88-115)	85-92 (118-122)	1.8-6
	1100	77	78	11	58-76 (74-100)	80-80 (102-104)	1.4-11
	1300	46-51	53-63	5-11	55-58 (72-74)	63-68 (82-87)	5-6.4
	1500	36-38	40-40	2.5-4	32-33 (43-44)	35-36 (46-49)	6-6.5
C	1650	27-32	30-34	2-5	21-27 (31-35)	22-28 (34-36)	4-9
	RT	71-85	133-146	23-29	87-92 (106-122)	124-125 (152-164)	6-9
	700	53	94	17	-	-	-
	1100	49-52	81-82	14-18	61-66 (82-83)	74-76 (100-102)	3-5.8
	1300	36-44	46-54	12-15	-	-	-
	1500	32-33	36-37	5-6	26-28 (31-36)	31-34 (41-43)	14-16
1650	27-30	30-32	4-4.2	-	-	-	

\*Minimum duplicate test results except single tests where no range is shown. See text for process codes.

\*\*Numbers in parentheses have been calculated on original section thickness before braze coating.

TABLE 2-5

## SUMMARY OF TENSILE AND CREEP/RUPTURE RESULTS ON 0.010-IN.-THICK IN625

Test Temp., of	Braze-Cycled (uncoated)*			Braze Coated with 100 Filler*		
	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.
<u>Braze-cycled or coated</u>						
RT	49-64	120-124	42-50	64-69 (102-108)	72-82 (120-121)	14-16
1000	31-33	92-97	44-48	59-62 (90-95)	63-63 (95-97)	10-12
1500	33-37	55-56	33-37	30-31 (47-48)	35-36 (56-56)	27-29
1650	-	-	-	22-23 (33-34)	23-23 (24-24)	44-52
1800	15.5	15.5	20	12.2 (19.3)	12.2 (19.3)	32
<u>As received</u>						
RT	71-72	134-139	42-47			

Test Temp., of	Stress,** ksi	Creep/Rupture *** Life in Hours for				Rupture
		Braze-Cycled 1% Creep	(Uncoated) Rupture	Braze Coated		
				1% Creep	5% Creep	
1500	10	14	472 (735)	12	229	1159 (764)
1575	14	-	-	1	5.5	42 (782)
1650	5	2.5	254 (733)	12	118	>1000 (763)
1650	2.5	22	NM (736)	-	-	-
1725	8	-	-	0.5	2.6	12 (781)
1800	5	-	-	NM	-	21 (788)
1800	2	6.5	312 (734)	-	-	-
<u>As received</u>						
1650	5	17	46 (737)	-	-	-

\*Duplicate tensile tests. Numbers in parentheses are calculated on original thickness before coating with 100 filler metal.

\*\*Stresses based on original thickness before braze coating.

\*\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

NM = Not measured

TABLE 2-6

## SUMMARY OF TENSILE AND CREEP/RUPTURE RESULTS ON 0.010-IN.-THICK IN617

Test Temp., OF	Braze-Cycled (Uncoated)			Braze Coated with 100B Filler		
	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	0.2% YS,* ksi	UTS,* ksi	% Elong. over 1 in.
RT	49	127	32	55 (80)	74 (107)	20

Test Temp., OF	Stress,*** ksi	Creep/Rupture** Life in Hours for			
		Braze-Cycled (Uncoated)		Braze Coated with 100B Filler	
		1% Creep	Rupture	1% Creep	Rupture
1650	7.5	7.2	476+ (823)	-	-
1725	4	16.5	476+ (824)	-	-
1725	8	<1	24 (836)	<1	13 (837)****
1800	5	NM	272 (821)	NM	89 (822)

\*Numbers in parentheses are calculated on original thickness before coating with 100B filler metal.

\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

\*\*\*Stresses based on original thickness before braze coating.

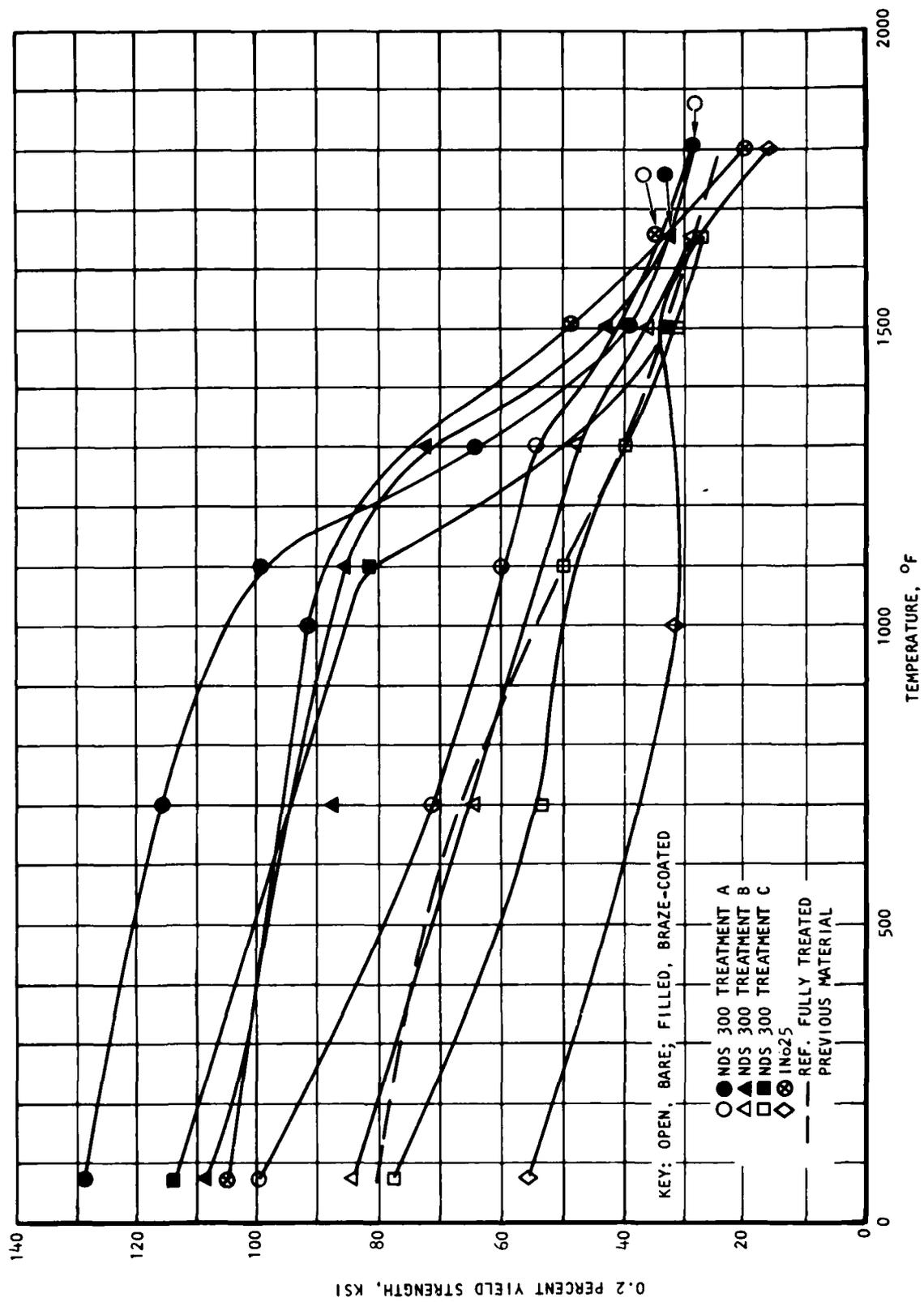
\*\*\*\*Coated with 100 filler metal.

TABLE 2-7

SUMMARY OF CREEP/RUPTURE TEST RESULTS ON 0.010-IN.-THICK NDS 300 MATERIAL\*

NDS Process Code	Condition and Test Type	Time in Hours to Reach Creep or Rupture at										
		15000F 20 ksi	15750F 16 ksi	15750F 14 ksi	15750F 12.5 ksi	16500F 15 ksi	16500F 12.5 ksi	17250F 12 ksi	17250F 10 ksi	17250F 8 ksi	18000F 7.5 ksi	18000F 5 ksi
A	Uncoated 1% Creep Rupture	-	261 1226+	-	-	44-77 >50-163+	-	16 NM	208 248+	-	NM 90-162	NM 314
	Braze coated 1% Creep Rupture	-	-	168 1097+	1330 2012+	-	109 593	39 NM	-	60 529	NM 92+	NM 1070+
B	Uncoated 1% Creep Rupture	48-600+ 111-600+	-	-	-	19-98 79-177	-	-	239+ 239+	-	NM >90-173	NM 238+
	Braze coated 1% Creep Rupture	-	340+ 912+	-	-	45-120 >115-192	324-352 >777-867+	-	28-55 >55-88+	122 770+	NM 245	NM-58 >191-743
C	Uncoated 1% Creep Rupture	-	-	-	-	-	77 544	-	148 156	184 379	-	NM 400
	Braze coated 1% Creep Rupture	-	-	365 932+	-	-	106 499	-	-	27 648	-	NM 649

\*Ranges or single points as indicated. > or + signs are terminated tests. NM = Not Measured. All stresses are on original area before braze coating.



A-69987

Figure 2-7. Yield Strength of Bare and Braze-Coated NDS 300 and IN625 as a Function of Temperature

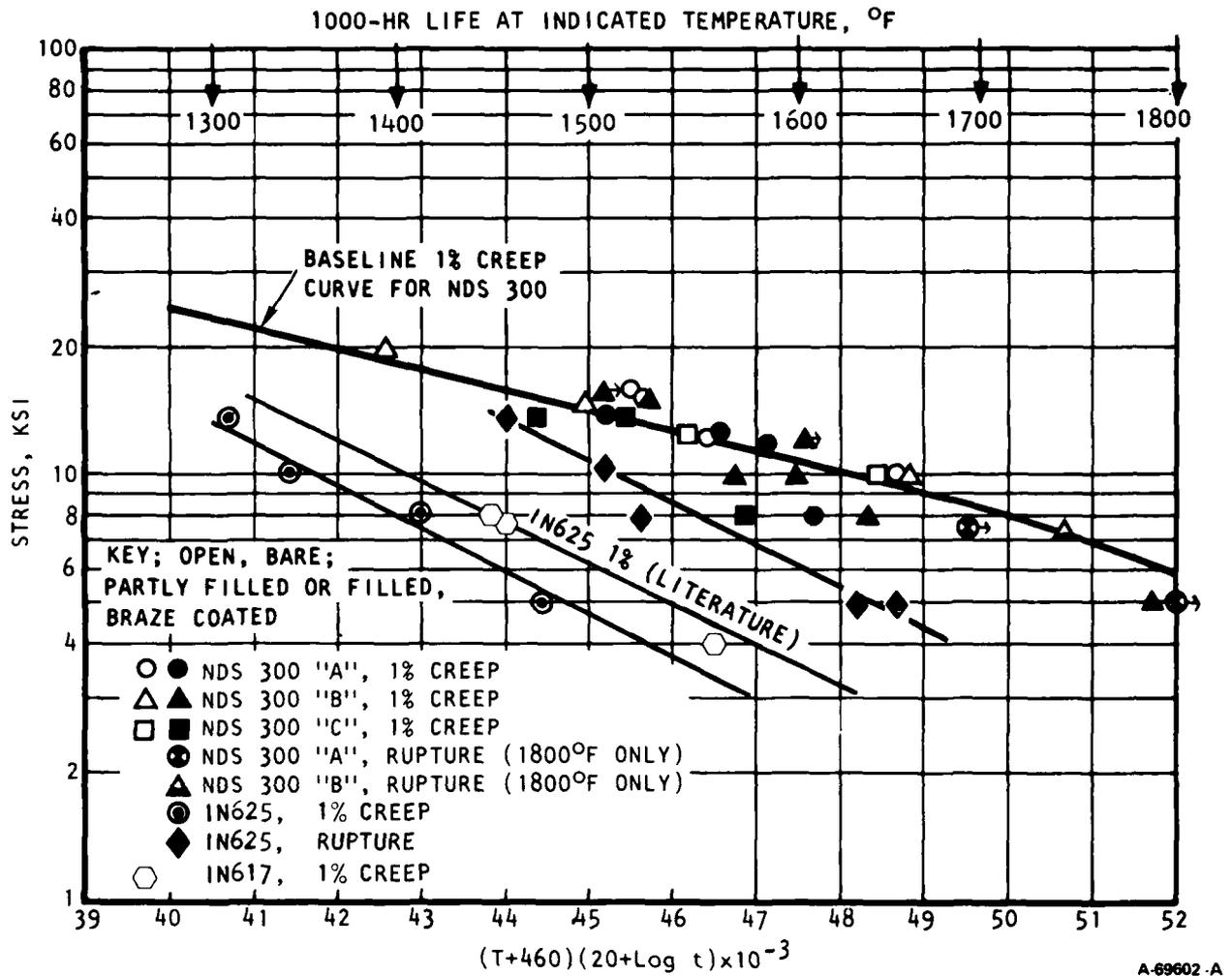
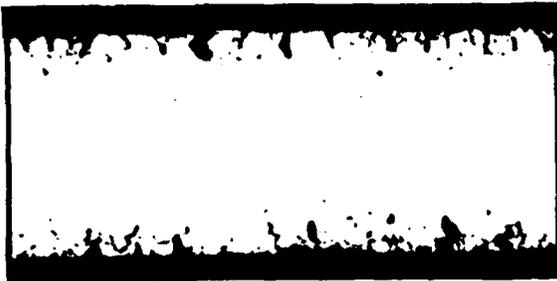


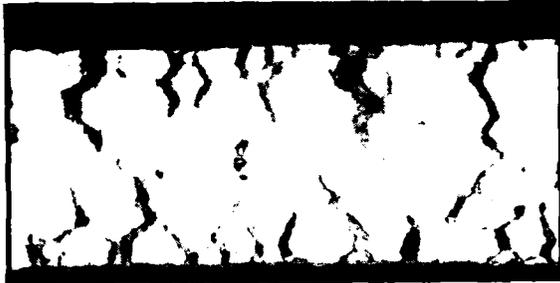
Figure 2-8. Comparison of Creep/Rupture of NDS 300 and Inconels 617 and 625



a. NDS 300, BARE, CREEP  
TEST CODE 643. FAILED  
IN 173 HR AT 1800°F, 7.5 KSI



b. NDS 300, AMI 100, CREEP  
TEST CODE 703. FAILED  
IN 317 HR AT 1800°F, 5 KSI



c. IN625, BARE, CREEP  
TEST CODE 733. FAILED  
IN 254 HR AT 1650°F, 5 KSI



d. IN625, BARE CREEP  
TEST CODE 734. FAILED  
IN 312 HR AT 1800°F, 2 KSI

F-46029

Figure 2-9. Comparison of Microstructures of NDS 300 and Inconel 625 Creep Test Specimens (Magnification, 200X)

yield strength curves are now being achieved with the partial processing of A and B techniques. Similar data for Inconel 625, a currently used production recuperator material, are given in Table 2-5 and are also plotted in Figure 2-7. The braze-coated Inconel 625 is very similar in yield strength to NDS 300 up to about 1650°F, but continues to decrease significantly in strength at higher temperatures, especially by comparison to NDS 300 at 1800°F. Also, although braze-coated IN625 is similar in yield strength to braze-coated NDS 300 below 1650°F, bare IN625 clearly is inferior in strength in the intermediate temperature range where high transient stresses are often encountered. Similarly, the few tests run on IN617 (Table 2-6) also show that NDS 300 is markedly superior in tensile strength properties.

Creep test results on NDS 300 for the three treatments are given in Table 2-7. Minimum values for each test condition, bare and braze-coated, are plotted in Figure 2-8 for 1-percent creep extension (designated 1800°F points are rupture only for braze-coated material). Inconel 625 and 617 data from Tables 2-5 and 2-6 are also plotted in Figure 2-8. Again the A and B treated materials are strongest. The 1 percent creep points for braze-coated IN625 (plotted in Figure 2-8) are close to the literature curve, and are significantly higher than the braze-cycled bare material (Table 2-6); the braze-coated material is also stronger in rupture. The NDS 300 material is about three times as strong as IN625 in creep strength in the range of interest for recuperators, i.e., in the Larson-Miller parameter range of 45 to 47.5 (1000 hr at 1500° to 1600°F) where stresses are highest. This strength advantage for NDS 300 is apparently operative for even the weaker C material.

IN617 is stronger than IN625 in creep strength (as can be seen by comparison of Tables 2-5 and 2-6, and as shown in Figure 2-8), but it too has markedly lower strength than NDS 300. The Inconel alloys have been observed to stretch readily at these temperatures, even under low stresses. Creep strength is very low, and, while rupture strength is better, metallographic analysis has indicated that substantial intergranular oxidation is occurring (see Figure 2-9), with attendant loss in ductility at intermediate temperatures. Under the high stresses being used to test NDS 300, the lives of these Inconels have been short (compare Tables 2-5, 2-6, and 2-7). As a further illustration, creep curves for NDS 300 and IN625 are compared in Figure 2-10. While the Inconels show very high rupture ductility, extensive creep is generally unacceptable, i.e., design stress levels will have to be held very low by comparison to those allowable with NDS 300.

Although markedly superior to the Inconels in creep strength, NDS 300 failed to meet the projected minimum curve of Figure 2-3 in longer-time, lower-stress tests above 1700°F, especially when braze coated. From microstructural analysis, this appeared to be caused by insufficient nitriding with the partial treatment cycle. Accordingly, several tests were conducted near the end of the program using modifications of the previously described B and C treatments. High elongation of A material rupture tested at 1800°F suggested a much higher creep rate, possibly due to the fine grain size. Microstructures of the material produced with a modified B treatments are compared to A-treated material in Figure 2-11. The structures shown for runs 286, 287, and 288 correspond to material given a B treatment and show a coarser grain size near the surface than occurs with A material heated in ammonia. The darker areas near the center are indicative of a coarser TiN dispersoid which will be less effective in strengthening. Note that this characteristic essentially

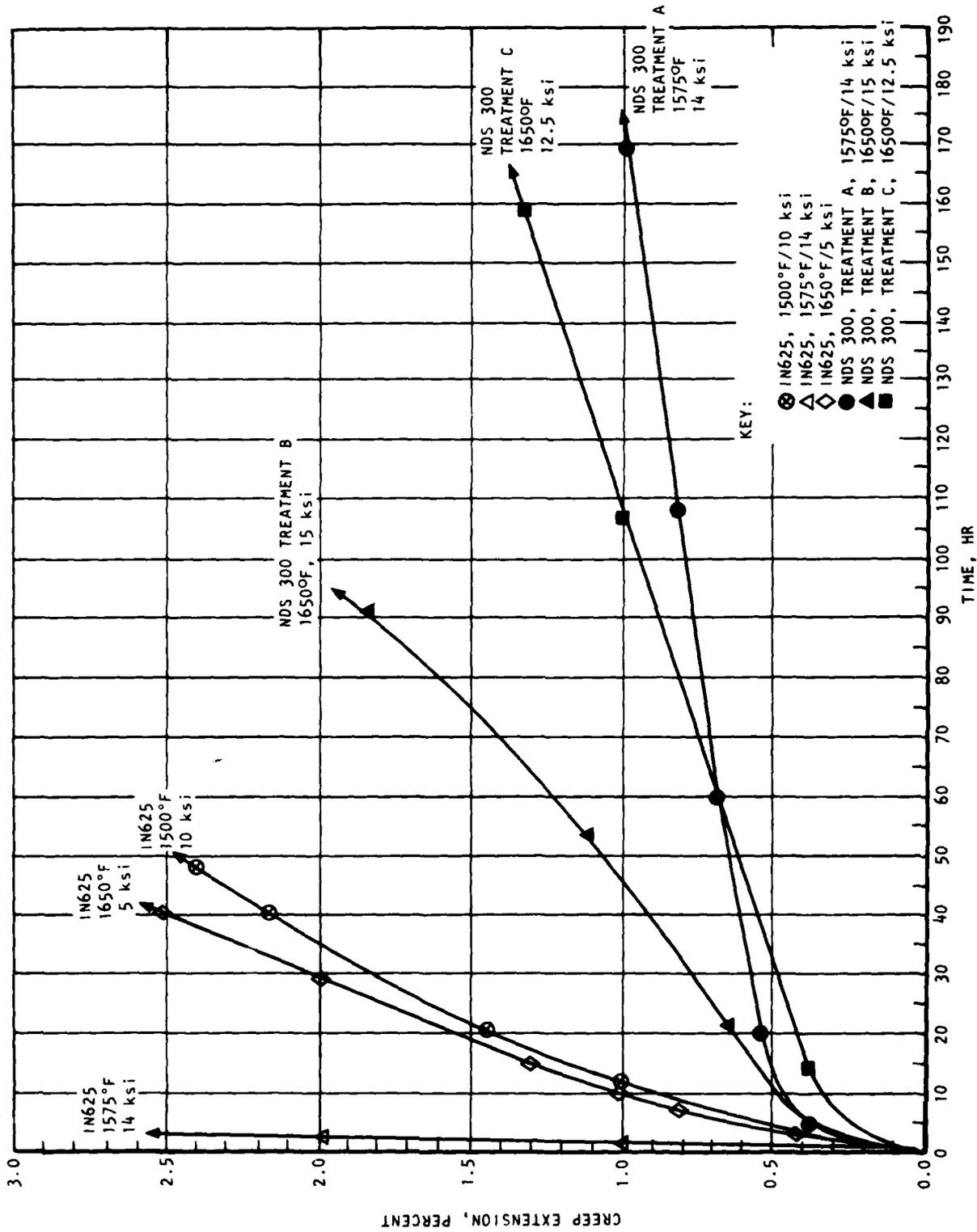
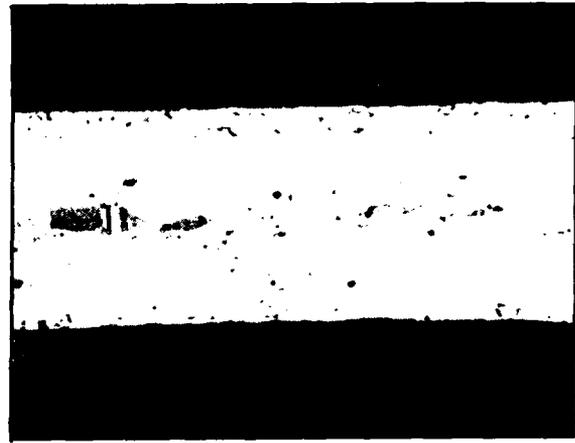


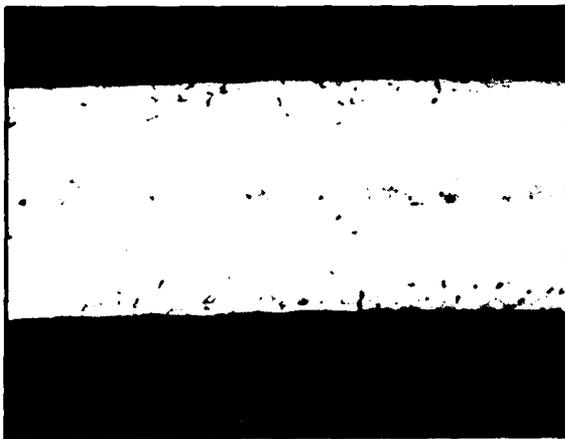
Figure 2-10. Comparison of Creep Curves for Braze-Coated NDS 300 and IN625



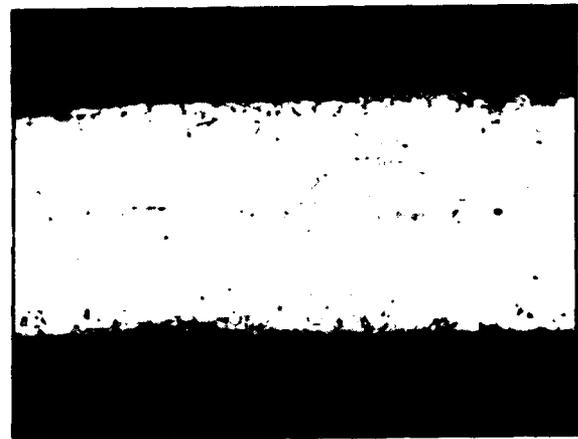
a. CONDITION A MATERIAL



b. RUN 286



c. RUN 287



d. RUN 288

F-46028

Figure 2-11. Comparison of Modified Nitride Treatment B Microstructures to the A Treatment Structure; All B Samples Were Taken from Broken Tensile Test Specimens of Table 2-8 (Magnification, 200X)

disappears in material from runs 287 and 288. Note also the slightly coarser grain size near the surface of run 288 which was given a short anneal prior to nitriding.

Test results are given in Table 2-8. Some improvement in creep strength was achieved. However, tensile properties at ambient were below those of A material, so compromise may be necessary depending on the specific property characteristics required for application to a recuperator.

### 2.3.1.3 Post-Service Ductility

The basic concept of internal nitriding has led many to believe that in-service ductility loss will be a problem. However, as shown in Table 2-9 (which presents room temperature tensile test data for terminated creep test specimens) embrittlement does not appear to be a problem, except possibly for braze-coated NDS 300 material subjected to long-term tests at 1800°F. Since braze-coated NDS 300 did show good rupture ductility in 1800°F tests, however, an in-service problem would not be expected. The Inconels, when stressed to similar or even lower levels where considerable creep occurred, also showed very poor ambient ductility for all creep test temperatures above 1500°F. In fact, most of the Inconel 625 creep test samples that were terminated prior to failure were so brittle they could not be removed from the test fixtures without breaking in numerous places. This behavior in Inconel 625 will be discussed in more detail in Section 2.7.

### 2.3.2 Task 1b, Alloy Modifications

In modifying the base chemistry for increased nitriding rate, the austenitic structure must be maintained if high-temperature strength is to be retained. There have been many treatises on the effects of composition on phase balance in stainless steels. One of the most recent is by INCO (Reference 5) which deals with replacement of chromium in stainless steels. Simply put, the three possible room-temperature phases--austenite, ferrite, and martensite--can be mapped by compositional balance, as shown in Figure 2-12. The important points to recognize are (1) the need to increase nickel equivalent (by manganese addition) as chromium level is decreased in order to stay within the austenitic field, and (2) the need to stay along the left-hand side of the austenite field so that the nominal 2Ti addition does not push the starting raw material into the two-phase austenite plus ferrite region to the right side of Figure 2-12. (Once the TiN dispersoid is formed, the titanium content of the alloy is ignored in calculating the chromium equivalent per Figure 2-12.) The objective was to achieve an increase in nitriding rate by adding manganese for increased nitrogen solubility (Reference 1) without upsetting the austenite phase balance. Conversely, elements other than titanium which also form nitrides should be decreased to help achieve a faster nitriding rate. For example, additions of aluminum, vanadium, and columbium would be undesirable, because these elements form nitride particles, which slow the overall nitriding rate without providing the degree of strength improvement obtainable with titanium nitride particles alone. Silicon and chromium have a similar effect, but to a lesser degree.

#### 2.3.2.1 Button Melts

For this portion of the study, an electric arc button melter was used to make 30-gram ingots to the nominal compositions shown in Table 2-10. Starting

TABLE 2-8

RESULTS OF FINAL NITRIDE OPTIMIZATION PROCESSING STUDIES ON 0.010-IN.-THICK NDS 300 ALLOY\*

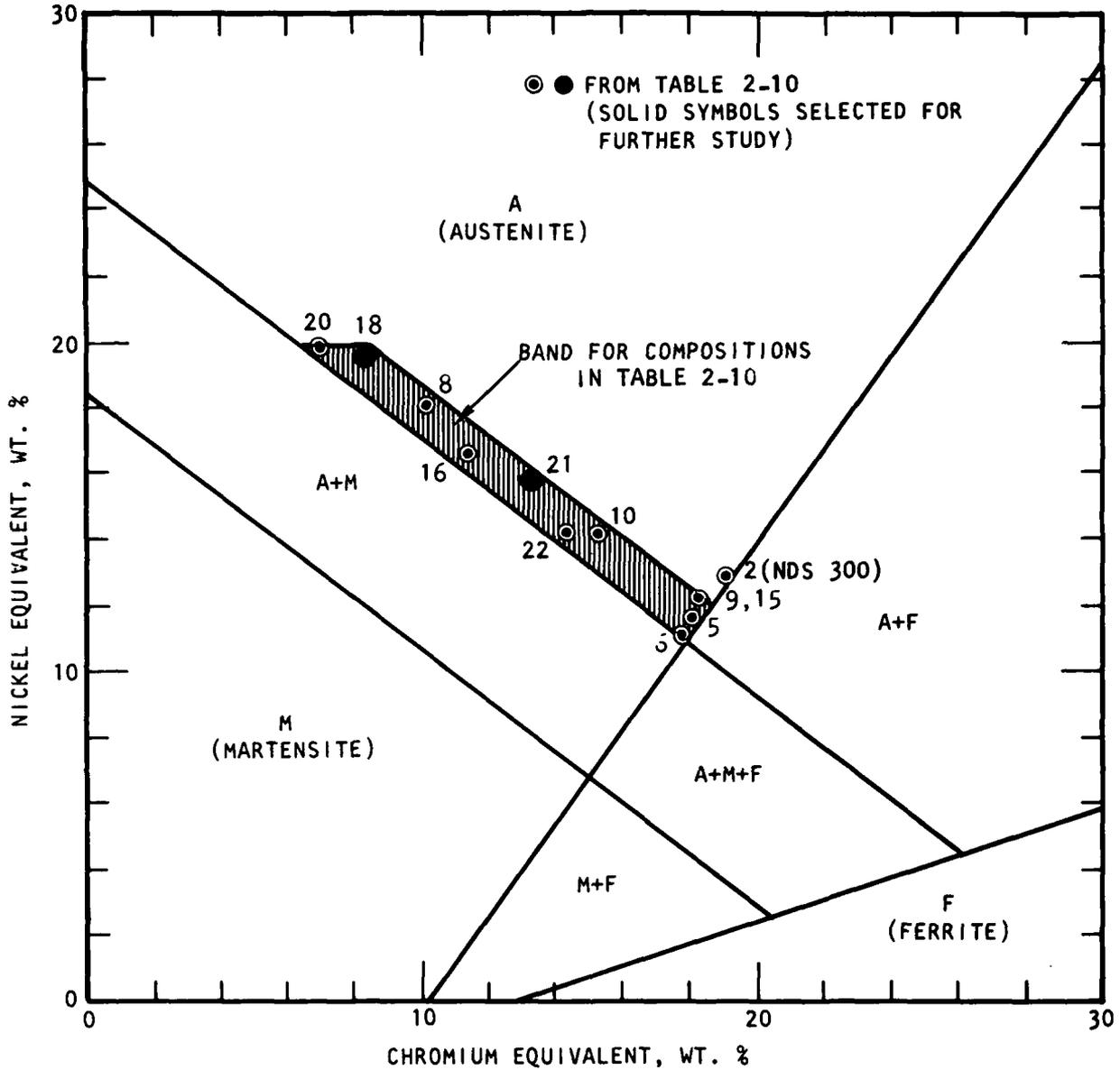
Nitride Test Code No.	Nitriding Temperature, OF	Nitriding Time, min	Tensile Properties				Creep Test Data: Hours at					
			Temp., OF	0.2% Yield, ksi	Ultimate, ksi	% Elongation over 1 in.	17250F/8 ksi for		18000F/5 ksi for			
							% Creep	Rupture***	% Creep	Rupture***		
284	1950	10	RT	89,86	148,144	23,22	142	-	688(804)***	-	-	-
285	1900	17.5	RT	90,98	147,152	23,19	-	-	-	-	-	-
286	1900	17.5	RT	93	151	20	114	-	(827)***	100	>662	875(834)***
286	1900	17.5	RT	-	-	-	-	-	-	100	560	>731(835)**
287	1900	20	RT	94,98	150,152	19,19	-	-	-	245	606	>732(830)***
287	1900	20	1800	21,8(27,2)**	25,6(32,0)**	10	-	-	-	507	620	>663(831)**
288****	1900	20	RT	89	148	20	178	-	>840(828)**	100	214	>732(832)***
288****	1900	20	1800	19,9(26,9)***	23,8(32,1)***	36	194	-	>812(826)***	245	826	916(833)**

\*All samples were denitrided by the 21000F/20 min treatment established earlier.  
 \*\*Test specimens were coated with MBF-50. Numbers in parentheses are based on original cross section before coating. Creep test stress levels are similarly based on original cross section.  
 \*\*\*Test specimens were coated with AMI 100. Stresses calculated the same as for MBF-50 above.  
 \*\*\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.  
 \*\*\*\*\*Held for 3 min at 19000F in hydrogen prior to nitriding.

TABLE 2-9  
ROOM TEMPERATURE TENSILE PROPERTIES OF TERMINATED CREEP TEST SPECIMENS

Creep Test No.	Material	Original Condition*	Creep Test Conditions			Mechanical Properties**			
			Temp., of	Stress,** ksi	Time, hr	Creep Extension, %	0.2% YS, ksi	UTS, ksi	% Elong.*** over 1 in.
702	NDS 300	Braze coated/Fin	1800	2	1607	1.8	71	76	1.0
721	NDS 300	Early B	1500	20	603	0.29	84	128	11.0
755	NDS 300	A	1725	10	248	1.3	94	123	5.0
757	NDS 300	B	1725	10	239	<1.0	76	107	3.0
762	NDS 300	A	1575	16	1226	1.8	92	134	8.0
767	NDS 300	A/MBF-50	1800	7.5	92	1.6	104	148	5.0
775	NDS 300	A/braze coated	1575	14	1097	3.5	100	151	7.3
784	NDS 300	B/braze coated	1575	16	912	2.0	90	158	6.4
785	NDS 300	B/braze coated	1650	12.5	867	2.5	88	94	2.2
786	NDS 300	B/braze coated	1725	8	770	2.5	95	116	3.2
787	NDS 300	A/braze coated	1575	12.5	2012	1.6	112	150	5.0
796	NDS 300	C/braze coated	1575	14	932	3.1	96	162	12.5
808	NDS 300	B/braze coated	1650	12.5	777	3.0	96	101	2.3
823	IN617	Braze cycled	1650	7.5	476	35	-	2.9	0
824	IN617	Braze cycled	1725	4	476	12.3	-	7.6	0
826	NDS 300	Run 288/AMI 100	1725	8	812	3.0	88	119	2.7
828	NDS 300	Run 288/MBF-50	1725	8	840	2.3	96	131	2.7
831	NDS 300	Run 287/AMI 100	1800	5	663	2.3	95	127	1.5
832	NDS 300	Run 288/MBF-50	1800	5	732	3.2	75	75	1.0
835	NDS 300	Run 286/MBF-50	1800	5	731	2.7	96	110	1.5

\* Braze alloy coating was 100 filler metal unless noted otherwise as MBF-50.  
 \*\* Stresses were calculated on original thickness prior to any brazing alloy coating.  
 \*\*\* Tensile elongation is in addition to any prior creep deformation.



$$\text{Ni EQUIVALENT} = \%Ni + \%Co + 30(\%C) + 25(\%N) + 0.3(\%Cu) + 0.5(\%Mn)$$

$$\text{Cr EQUIVALENT} = \%Cr + 2(\%Si) + 1.5(\%Mo) + 5.5(\%Al) + 1.5(\%Ti)$$

A-50122 -A

Figure 2-12. Modified Schaeffler Diagram for Austenite-Martensite-Ferrite Phase Balance in Austenitic Stainless Steel Weldments (Reference 5)

TABLE 2-10

## NOMINAL AIM COMPOSITIONS OF 30-GRAM BUTTON MELTS

No.	Cr	Ni	Mn	Mo	Ti	Fe	Schaeffler Equivalent*		Depth of Penetration,** Mils		Hardness Readings (DPH200 converted)	
							Cr	Ni	CrN	TiN	TiN Layer	Base Metal
2	18	12	-	-	2	Bal.	19	13	1.0	3.6	HRC 39	HRB 71
5	17	8.5	4	-	2	Bal.	18	11.5	0.5	4.0	NT	NT
6	16	6	7	-	2	Bal.	17	10.5	<0.2	2.0	HRC 31	HRC 24
7	14	8	5	-	2	Bal.	15	11.5	0.4	2.6	HRC 43	HRC 20
8	9	12	10	-	2	Bal.	10	18	0.8	3.6	HRC 34	HRB 90
9	11	11	-	4	2	Bal.	18	12	0.4	2.6	HRC 36	HRB 86
10	12	11	4	1	2	Bal.	15	14	0.5	2.6	HRC 36	HRB 86
11	12	11	8	-	2	Bal.	13	16	0.4	3.1	HRC 39	HRB 80
12	12	10	10.5	-	2	Bal.	13	16	0.9	3.4	HRC 37	HRB 84
13	14	10	6	-	2	Bal.	15	14	0.7	3.1	HRC 39	HRB 80
14	14	8	10	3	2	Bal.	18	14	NT	3.3	HRC 39	HRB 97
15	15	10	2	1.5	2	Bal.	18	12	0.6	3.0	HRC 39	HRB 86
16	9	8	16	1	1.5	Bal.	11	17	1.4	5.0	HRC 42	HRB 89
17	9	8	18	-	2	Bal.	10	18	2.0	5.0	HRC 41	HRB 84
18	7.5	10	17	-	2	Bal.	8	19.5	3.0	5.6	HRC 42	HRB 86
19	7.5	12	12	0.75	2	Bal.	9	19	1.5	4.4	HRC 42	HRB 84
20	5	12	14	1	2	Bal.	7	20	1.4	4.4	HRC 38	HRB 86
21	12	8	14	-	2	Bal.	13	16	1.2	3.9	HRC 46	HRB 90
22	13	8	10	-	2	Bal.	14	14	0.6	3.4	HRC 46	HRB 90
23	13	6	14	-	2	Bal.	14	14	1.0	4.1	HRC 44	HRB 94
24	11	6	14	1.5	1.5	Bal.	14.5	14	0.6	4.6	HRC 44	HRB 93

\*Includes corrections for residuals such as N and Si, but does not include Ti since it will be combined as TiN.

\*\*Internal boundary of indicated phase after nitriding for 15 min at 1900°F.

materials were either the NDS 300 18Cr-12Ni-2Ti base or a known lot of type 201 stainless steel. Additions of pure nickel, manganese, molybdenum, and titanium were made. Low-carbon ferrochromium was also used. Where dilution was required a known lot of type 409 stainless steel was utilized. The analyses shown in Table 2-10 were selected based on maintaining a stable austenitic structure, determined by Figure 2-12. (Note Figure 2-12 was derived for weld metal, i.e., a cast structure very similar to the button heats. Some shifting of the phase boundary lines apparently occurs in wrought material, hence Figure 2-12 should be considered a first round approximation only.) Several compositions from Table 2-10 are plotted in Figure 2-12; except for nos. 2 (NDS 300), 7, and 14, all aim (target) analyses from Table 2-10 fall within the band shown in Figure 2-12. Button melts were sectioned and checked for chemistry by X-ray fluorescence (see Figure 2-13a). A center slice was then cut for nitriding at a constant condition of 1900°F for 15 min. Nitride penetration depths, nitride layer hardness, and basic ingot hardness were all determined; test results are given on the right side of Table 2-10. The basic material structure also was examined using metallography. Figures 2-13 b and c show typical nitrided structures of the two NDS 200 button melts chosen for further analysis. Figure 2-13d shows NDS 300 strip used for control.\* The dark bands near the surfaces contain chromium nitride which is eliminated during denitriding.

The results of these tests strongly showed the benefits of high manganese, low nickel, and low chromium content, although austenite phase stability is somewhat suspect with very high manganese, low chromium, or low nickel. Based on alloy content, nitriding rate, hardness, and structure (with emphasis on alloy content and nitriding rate), two compositions, numbers 18 and 21 in Table 2-10, were selected for further development. Both are low in chromium and nickel content, nitride faster than the NDS 300 base, and produce a higher TiN layer hardness, meaning potentially higher strength. These two materials also represented a reasonable range of chromium (7.5 to 12 percent), nickel (8 to 10 percent), and manganese (14 to 17 percent) for this class of alloys, and were free of molybdenum which is a more costly addition. Alloy 23, a possible candidate but outside this chemistry range, was one of the harder base metals of the group and appeared to have some martensite present, which would make processing to strip difficult. Alloy 17 was essentially in the range selected.

Compositions 18 and 21 were melted as 17-lb gross heats (Table 2-11), and partly processed to 0.010-in.-thick foil by Allegheny Ludlum Research Center. (Allegheny Ludlum recommended the larger heats instead of buttons, based on achieving better chemistry control.) To hold costs in line, only a portion of each was processed to final gage, with the balance of each held in reserve. Approximately 9 ft of material, 4-in. wide by 0.010-in. thick, was supplied for each composition. The balance of each was supplied as hot-rolled band 0.100-in. thick, annealed and descaled, for a total length of about 4 ft per alloy.

### 2.3.2.2 Early Nitriding Trials

Initial nitriding tests on these two NDS 200 materials showed that nitriding rates were markedly improved, e.g., the 12Cr-14Mn-8Ni-2Ti alloy (Heat RV-9307, Table 2-11) would through-nitride at about 1900°F in just 5 min, which is

\*Nitride testing of a button melt from NDS 300 basic alloy (cut-up strip) showed the viability of using this technique with a cast structure.



a. "INGOT" SLICED FOR NITRIDING AND CHEMISTRY

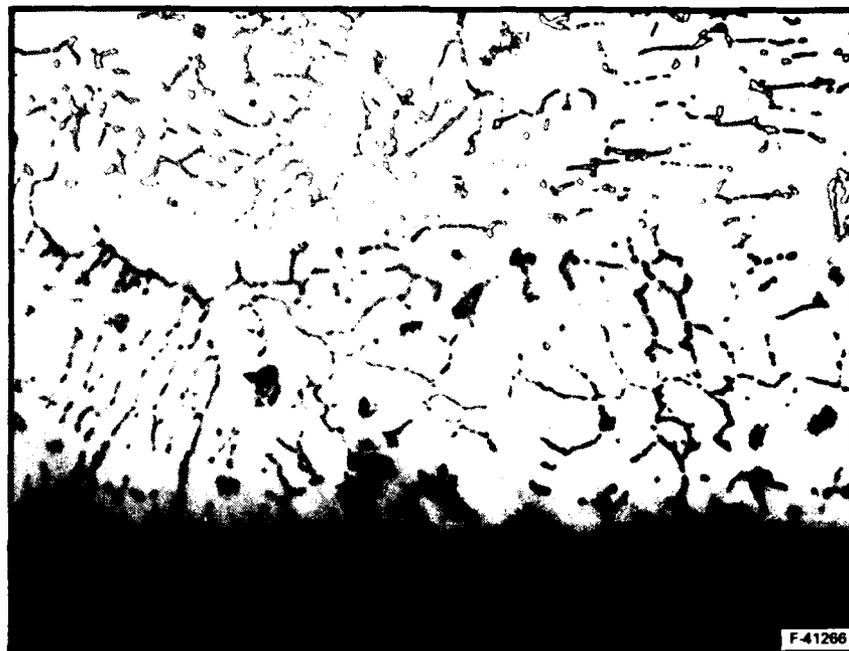


TiN DEPTH  
(DARK LAYER AT  
BOTTOM INCLUDES  
OTHER NITRIDES)

b. COMPOSITION NO. 18 FROM TABLE 2 10

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Figure 2-13. Thirty-Gram Ingot and Comparison of Nitrided Structures of Two NDS 200 Compositions Selected to NDS 300 Strip (Magnification, 250X)



c. COMPOSITION NO. 21 FROM TABLE 2-10



d. NDS 300 STRIP (CONTROL)

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Figure 2-13. (Continued)

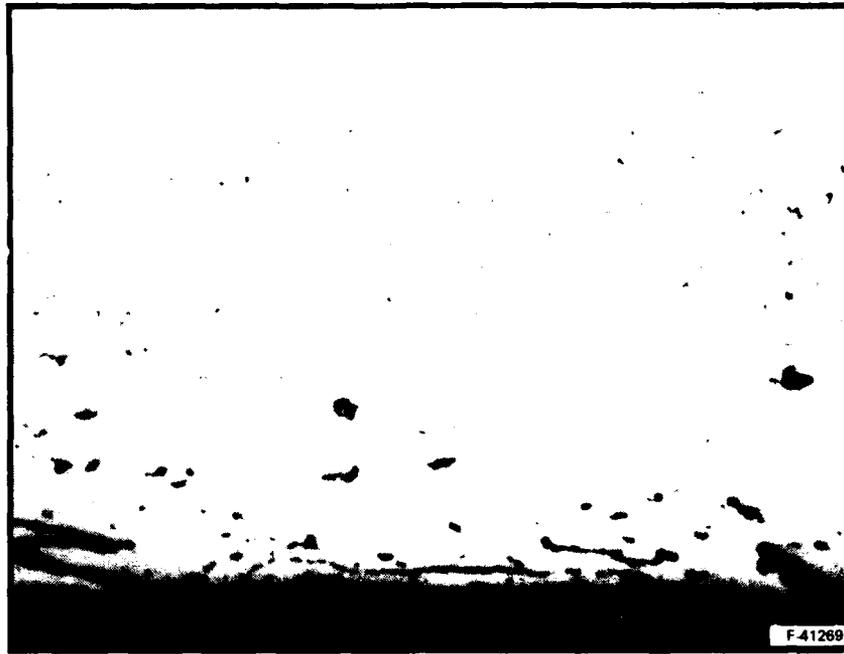
TABLE 2-11

## ANALYSES OF INITIAL NDS 200 MATERIALS

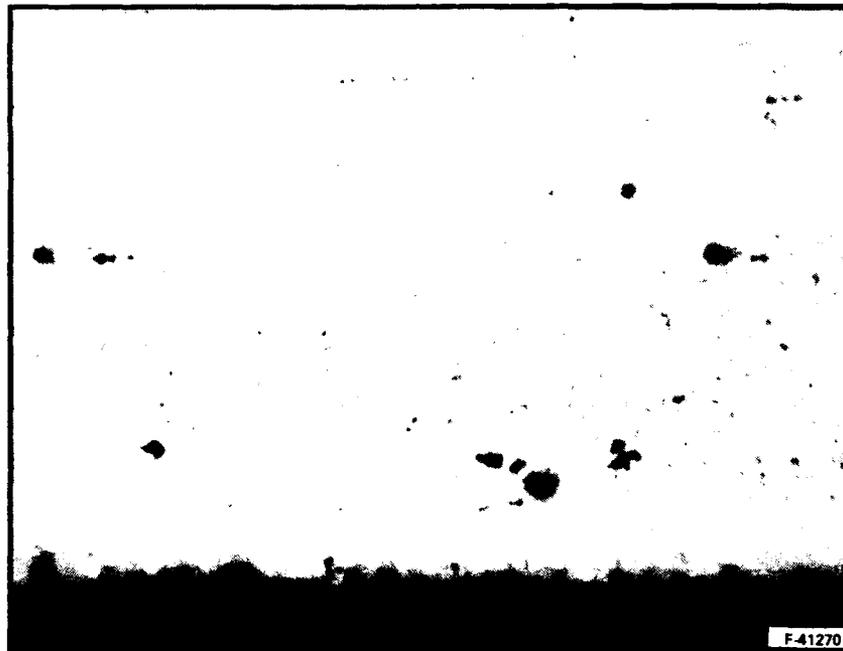
Element	Weight Percent in Heat	
	RV-9307	RV-9308
C	0.0014	0.0016
Mn	13.85	16.30
P	0.008	0.009
S	0.007	0.007
Si	0.39	0.34
Cr	11.70	7.10
Ni	8.02	10.26
Al	0.0057	0.0070
Mo	0.006	0.011
Cu	0.012	0.003
Nb	0.002	0.002
V	0.083	0.077
Ti	2.29	2.32
N	0.012	0.002
Co	0.095	0.10
W	0.024	0.026
Sn	0.007	0.007
Pb	0.0002	0.0002

only one-third of the minimum partial cycle found for the NDS 300 baseline material, and a definite advantage for continuous line production. (The time required for heat RV-9308 was about 25 percent longer.) However, the new materials also became fairly brittle as a result of nitriding (6 to 10 percent ductility compared with 18 to 25 percent for the NDS 300 alloy) and further adjustments from the processing technology for the baseline material appeared necessary.

As a result, for a while the major emphasis for the entire program was on finding a solution to the brittleness problem for the modified alloys. In reviewing the microstructures of the modified materials, it was found that the least ductile materials did not fully recrystallize during the early part of the high-temperature nitride and showed pronounced retention of the cold-worked structure; this characteristic led to splitting when tensile-tested (Figure 2-14a). However, the more ductile material was recrystallized in the center of the strip and showed a very fine structure near each outer surface, indicating that recrystallization had occurred without substantial grain growth (Figure 2-14b). From these observations a two-phase approach evolved. The first was to modify the nitriding procedure to allow nearly complete recrystallization to occur. The second was to reduce the amount of rolling reduction to final gage, i.e., after the last intermediate anneal, since recrystallization kinetics are strongly influenced by the energy stored in the material as a result of cold work. It was expected that this second approach would result in an overall coarser grain size, with correspondingly greater creep strength.



a. 12-PERCENT ELONGATION



b. 22-PERCENT ELONGATION

F-41271

Figure 2-14. Microstructures of NDS 200 (Heat RV-9307) Versus Tensile Ductility (Magnification, 250X)

From data given in Table 2-12, ambient tensile testing and limited creep-rupture testing at 1500° and 1650° F did indicate that the NDS 300 nitriding process can in fact be modified to accommodate the NDS 200 material to produce similar high strength and ductility. (See Appendix B for full details on creep test results.) Hence, the first approach from the previous paragraph was successful. However, this success was achieved only with the 12Cr-14Mn-8Ni-2Ti (heat RV-9307) composition; application of the technique to the second modified alloy (heat RV-9308) did not show any improvement, and splitting was still a problem. Furthermore, even though judged successful, the grain size of the recrystallized center portion of the strip was fairly small, and it was believed that creep strength could be even further enhanced if this grain size could be coarsened somewhat. (Grain sizes of ASTM 9 to 10 were observed, while NDS 300 showed about a size 6.)

### 2.3.2.3 Raw Material Processing Effects

Accordingly, contract modification No. P0002 was negotiated with AMMRC for a study of the effect of the final rolling reduction on the 12Cr-14Mn-8Ni-2Ti (heat RV-9307) material response during nitriding. The original trial lot of this material produced by Allegheny Ludlum had been reduced 75 percent to the final thickness of 0.010 in. For this experiment, reductions of 42 percent, 50 percent, and 57 percent were made, and the materials were subjected to nitriding trials. Results are shown in Table 2-13. Although not rigorously clear from the test data, a final rolling reduction of 60 percent was selected. This reduction was based on a combination of properties and a knowledge of steel mill processing, which dictates that the final reduction to gage be as large as possible because of handling problems during annealing and descaling lighter-gage materials. A center grain size of about ASTM 8 was achieved with this material.

It is possible that further processing modifications would have proved successful with the RV-9308 material. Thus, in choosing the higher-chromium RV-9307 modified material (now designated NDS 200) for continuation, several factors other than nitride embrittlement were considered. The first consideration was the uncertainty of success and the inevitable time delay to the program. The second was the concern voiced by Allegheny Ludlum personnel with regard to commercial producibility (at reasonable cost) of the lower-chromium/higher-manganese material. Finally, the corrosion and oxidation resistance of a 7-percent-Cr steel is below that of materials classified as stainless steels, thereby increasing both handling problems by the equipment fabricator and the potential for in-service burn-through in the event of a coating failure. (It was even necessary to coat the 12-percent-Cr material with a brazing alloy such as AMI 100 or 300; see Appendix B.) However, the program goal of reducing the content of strategic metals was met; further reductions, although possible, were beyond the technical scope and financial constraints of the program.

The new 17-lb heat essentially duplicated heat RV-9307. Although it was melted to the same nominal composition as the original (Table 2-11), residual element content was aimed at commercial steel mill practice rather than that level achievable by starting with all high-purity materials. (Table 2-14 shows commercial limits proposed by Allegheny Ludlum Steel Corp.) While past experience suggests no problems in translating this composition to production, it is important to ensure the higher residual elements do not interfere with nitriding or lead to a significant reduction in strength or ductility. Composition

TABLE 2-12

## RESULTS OF NITRIDE PROCESSING STUDIES ON 0.010-IN.-THICK NDS 200 ALLOYS\*

Test Code No.**	Heat No. (from Table 2-11)	Nitriding Temperature, OF	Time of Nitriding, min	Room Temperature Properties			Creep Test Data:*** Hours to 1% Extension	
				0.2% Yield, ksi	Ultimate, ksi	% Elong. over 1 in.	1500/20 ksi	1650/15 ksi
127T(H)	RV-9307	1900	12.5	78, 80	128, 114	15, 6		
127T(H)	RV-9308	1900	12.5	79, 84	136, 133	11, 10		
128T(H)	RV-9307	2000	8.5	88, 91	126, 136	8, 12		
128T(H)	RV-9308	2000	8.5	87, 87	126, 138	8, 12		
131T(H)	RV-9307	1900	12.5	80, 80	112, 122	6, 10		
131T(H)	RV-9308	1900	12.5	80, 82	144, 147	11, 13		
132T(H)	RV-9307	2000	8.5	70, 82	135, 125	11, 10		
132T(H)	RV-9308	2000	8.5	83, 102	145, 139	13, 11		
133R	RV-9307	1900	20	98, 98	125, 143	5, 11		
133R	RV-9308	1900	20	92, 103	144, 148	12, 10		
136R	RV-9307	1900	20	94	136	11		
136R	RV-9308	1900	20	82	125	10		
140R	RV-9307	2000	7	88	139	11		
140R	RV-9308	2000	7	79	124	11		
141R	RV-9307	1900	10	94	141	17		
141R	RV-9308	1900	10	81	128	13		
142R(H)	RV-9307	2000	7	83	143	21		1(580)
142R(H)	RV-9308	2000	7	80	136	21		5(581)
145T(H)	RV-9307	2000	5	90	138	18		26(589)
146T(H)	RV-9308	2000	5	74	122	10		25(593)
147T(H)	RV-9307	2000	4	73	142	24		4(592)
150T(H)	RV-9307	2000	5	68	134	24		
151R(H)	RV-9307	2000	5	57	69	3		
151R(H)	RV-9308	2000	5	68	84	4		
152T(H)	RV-9308	2000	6	68	125	15		
153T(H)	RV-9308	2000	5	64, 69	132, 137	28, 25		
154T(H)	RV-9307	1900	10	69, 71	101, 126	6, 11		
155T(H)	RV-9307	1900	8	65, 67	85, 87	4, 5		
156T(H)	RV-9307	1900	6	84, 84	146, 149	19, 21		
157T(H)	RV-9307	1900	4	82, 92	142, 152	18, 20		
158T(H)	RV-9308	1900	4	73, 82	126, 134	19, 20		
159T(H)	RV-9308	1900	6	89, 90	130, 132	9, 10		
160R	RV-9307	1885	6	92, 94	133, 131	11, 8		
161R	RV-9307	1875	5	97, 99	138, 142	12, 12		15(594)
161R	RV-9308	1875	5	95, 96	134, 130	11, 11		<1.5(595)
163R	RV-9307	1950	5	84, 86	138, 137	18, 14		
164R	RV-9307	1930/1950	5	79	143	29		
165R	RV-9307	1910/1950	6.5	86, 90	146, 150	25, 24		
166R	RV-9307	1910/1950	6.5	83, 88	142, 149	22, 22		98(609) 42(610)
167R	RV-9308	1895/1955	7.5	81, 82	121, 111	10, 6		

\* Denitriding was for 180 min at 2025°F

\*\* R = Furnace retort T = Tubular retort (H) = Heated to temperature in hydrogen or dissociated ammonia (slow flow)

\*\*\* Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

TABLE 2-13

RESULTS OF COLD-ROLLING EXPERIMENTS ON NDS 200\*

Test Code No.	Percent Reduction to Gage,	Room-Temperature Properties			Creep Property Data:**	
		0.2% Yield, ksi	Ultimate, ksi	% Elong. over 1 in.	Hours to 1% Elongation	16500F/15 ksi
171	42	81	142	20	--	16.5(20 ksi) (616)
171	50	--	--	--	19.5 (25 ksi)	9 (20 ksi) (619)
171	57	80	140	15	128 (620)	7.5 (20 ksi) (621)
172	42	103	146	11	--	63 (623)
172	50	--	--	--	--	31 (20 ksi) (624)
172	57	--	--	--	--	76.5 (20 ksi) (626)
173	42	80	136	20	94 (629)	85 (630)
173	57	72	135	22	136 (633)	24 (634)
173	75 (Control)	74	132	26	111 (631)	30 (632)

\*For 0.010-in.-thick material of RV-9307 composition as shown in Table 2-11. All nitriding was for 6.5 min in the range 19000 to 19500F (H<sub>2</sub> heatup). Denitriding was for 180 min at 20250F.

\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B. All creep tests were coated with AMI 300 filler metal.

TABLE 2-14

## COMMERCIAL MELT TOLERANCES ON ELEMENTS IN STAINLESS STEELS

Major Additions		Residuals	
Element	Range*	Element	Maximum Limits*
Cr	+0.25	C	0.02
Ni	+0.25	P	0.035
Mn	+0.50	S	0.035
Ti	+0.25	Mo	0.25
Si	+0.25	Cu	0.50

\*In percent by weight.

of the new heat RV-9408 is shown in Table 2-15. Nitriding tests did not show any significant differences between heats.

#### 2.3.2.4 Final Optimization of NDS 200

Up to this point, the most consistent creep results with NDS 200 were achieved by preheating to nitriding temperature in hydrogen, then switching to  $\text{NH}_3$  to nitride. Variations on this technique (see Table 2-16) were performed to produce samples for creep testing up to 1800°F. As indicated, several tests were run using various preanneal treatments in an attempt to coarsen the grain size and improve creep strength. The best results were achieved with runs 271 and 272 which featured a preanneal in hydrogen for 3 min at 1900°F, followed by nitriding around 1900°F for 6 min. The samples were then denitrided for one hour at 2050°F (which, based on NDS 300 tests, can probably be shortened considerably by increasing the temperature to 2100°F). Results are summarized in Table 2-17 and shown plotted in Figure 2-15. NDS 200 material strength is still significantly above that of IN625.

The original program plan was to run nitriding cycle variations on NDS 200 to develop representative tensile and creep data as shown in para. 2.3.1.2 for NDS 300. However, in meetings with AMMRC and the Tank Automotive Command (TACOM) at the end of the first year's work, it was urged that NDS 300, since it is the more fully developed and understood material, be prepared for use in first generation recuperators. Work on NDS 200 was accordingly deemphasized.

#### 2.3.3 Cost Analysis

At the end of the program, a comparison was made of the basic starting 18-12-2 material for NDS 300 to Inconel 625, both in nominal 10-mil thickness. The price was in the \$5 to \$6 per pound range for the 18-12-2 material compared to Inconel 625 at \$9.50 to \$13 per pound. Inconel 625 prices are currently depressed because of the market price weakness in metals such as nickel and molybdenum. This will not significantly affect the price of the 18-12-2 base material. However, as melting techniques for high-content titanium alloys

TABLE 2-15  
ANALYSIS OF HEAT RV-9408 (NDS 200 BASE)\*

Element	Weight Percent in Heat	
	Aim	Actual
C	0.03 max.	0.0039
Mn	14.0	13.94
P	0.035 max.	0.028
S	0.035 max.	0.007
Si	0.40	0.33
Cr	12.0	11.70
Ni	8.0	8.02
Al	0.05 max.	0.004
Mo	0.25 max.	0.26
Cu	0.50 max.	0.38
Nb	0.05 max.	0.002
V	0.05 max.	0.078
Ti	2.0	2.17
N	0.03 max.	0.007
Co	0.10 max.	0.091
W	0.05 max.	0.037
Sn	0.01 max.	0.005
Pb	0.001 max.	0.0015

\*Remake of Heat RV-9307 (Table 2-11) to commercial tolerances on elements (also see Table 2-14).

TABLE 2-16

RESULTS OF FINAL NITRIDE OPTIMIZATION PROCESSING STUDIES  
ON 0.010-IN.-THICK NDS 200 ALLOY\*\*

Nitride Test Code No.	Heat No. from Tables 2-11 and 2-15	Final Rolling Percent Reduction to 0.010 in.	Preanneal Treatment, OF/min	Nitriding Temperature, OF	Nitriding Time, min	Room-Temperature Properties			Creep Test Data***		
						0.2% Yield, ksi	UTS, ksi	% Elong. over 1 in.	1520/20 ksi	1650/15 ksi	1800/7.5 ksi
189	RV-9307	57	None	1900/1935	6.5	82, 84	136,136	16, 15	-	40 (645)	-
189	RV-9408	60	None	1900/1935	6.5	81, 85	139,141	22, 21	-	16 (644)	-
212	RV-9408	60	None	1910/1950	6	-	-	-	118 (646)	-	-
212	RV-9307	75	None	1910/1950	6	97,98	153,156	19, 20	-	85 (647)	7.5 (663)
221	RV-9307	75	None	1910/1950	6	84	141	16	-	-	-
222	RV-9408	60	None	1900/1947	6.5	79, 82	142,147	23, 21	-	9.5 (678)	-
223	RV-9408	60	1900/3	1925	6.5	82, 83	142,142	20, 23	-	9.5 (679)	-
225	RV-9408	60	1900/10	1935	6.5	77, 81	138,144	22, 24	-	32 (680)	10.5 (681)
227	RV-9408	60	2000/5	1900/1940	6.5	71, 71	113,117	9, 10	-	-	10 (683)
229	RV-9408	60	1900/10	1900	6	86	143	20	-	20.3 (729)	-
271	RV-9408	60	1900/3	1900	6	75	138	21	-	-	-
272	RV-9408	60	1900/3	1900	6	81	140	23	-	26 (769)	-

\*All samples denitrided for 180 min at 2025oF, except runs 271 and 272 which were 60 min at 2050oF

\*\*Three digit numbers in parentheses correspond to creep test codes in Appendix B. All creep test samples were coated with brazing alloy as shown in Appendix B.

TABLE 2-17

SUMMARY OF TENSILE AND CREEP/RUPTURE RESULTS ON  
0.010-IN.-THICK NDS 200 MATERIAL\*

Test Temp., OF	Uncoated			Braze Coated with 100 Filler**		
	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.
RT	75-81	138-140	21-23	93 (129)	111 (154)	6
1100	-	-	-	69 (92)	70 (93)	1.4
1300	-	-	-	43 (60)	51 (72)	11.3
1500	-	-	-	25 (34)	27 (37)	10
1650	-	-	-	19 (26)	21 (29)	9

Test Temp., OF	Stress,*** ksi	Creep Rupture Life in Hours for	
		1% Creep	Rupture****
1575	16	120	662 (768)
1650	15	26	136 (769)
1650	12.5	59	210 (780)
1725	10	19	152 (770)
1725	8	94	345 (779)

\*From duplicate nitride runs with optimized cycle.

\*\*Numbers in parentheses are calculated on original thickness before coating with 100 filler.

\*\*\*Stresses based on original thickness before braze coating.

\*\*\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

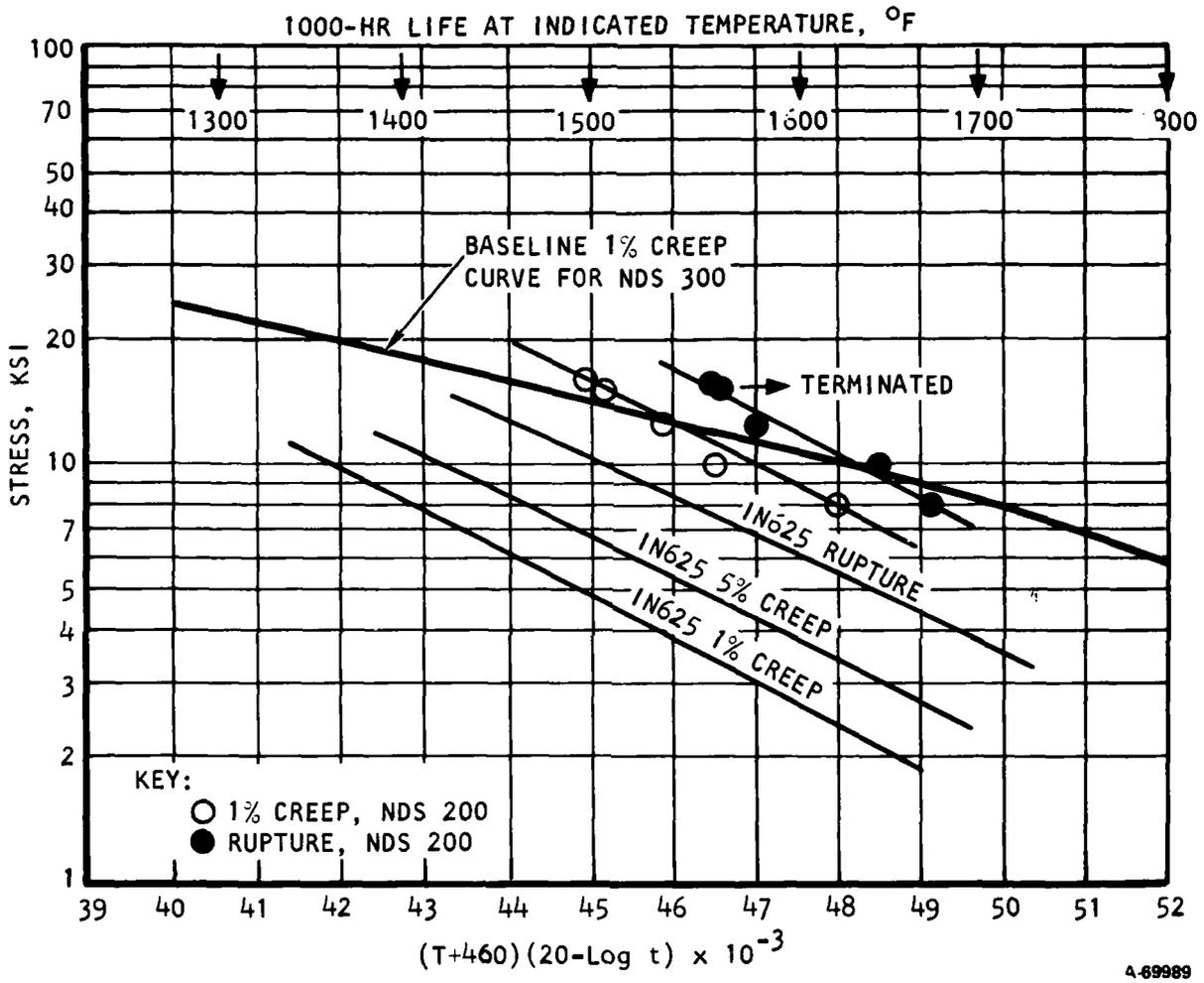


Figure 2-15. Comparison of NDS 200 to IN625 in Creep/Rupture Testing of Braze-Coated Material (IN625 Data from Table 2-5 and Figure 2-8)

continue to be improved, further reduction in the base material price can be expected. The lower titanium 321 stainless, which is commercially available, has the same Cr-Ni-Fe base as 18-12-2, for example, but the current price of 10-mil material is only about \$3 per pound. Since processing costs of 321 and 18-12-2 would be similar, the melting cost addition for 18-12-2 would be on the order of \$2 to \$3 per pound, which is high. There is the potential, though to see this melt cost figure reduced to \$1 to \$2 per pound, which would then put the cost of the 18-12-2 starting material in the \$4 to \$5 per pound range.

The cost of nitriding must be added to the price of 18-12-2 in order to reach a figure for NDS 300 thin sheet. This must necessarily include factors such as write-off and maintenance on the furnace, which are, in turn on a per-pound basis, dependent on the size and geometry of the furnace. The estimates range conservatively from \$2.00 to \$2.50 per pound to nitride thin sheet material. This analysis results in an average cost for NDS 300 of about 82 percent of the current minimum cost of IN625, with a range of 74 to 89 percent. Thus, the increase in strength can be obtained without a cost penalty, and can probably be achieved at a cost saving.

#### 2.4 TASK 2, BRAZING DEVELOPMENT

This task was primarily a matter of selection rather than development. Many filler metals were commercially available; the problems were to: (1) find one that would flow readily on either NDS 200 or NDS 300 while having minimal effect on the strength of the parent metal, and (2) ascertain that braze joint strength would be sufficient for high-temperature operation of plate-fin recuperators. Also, because the heat exchanger plates are fully covered by the filler metal during fabrication, the brazing alloy could suffice as protection for the lower-chromium NDS 200 alloy in lieu of applying a separate postfabrication diffusion coating.

Seven screening runs were made using 23 different filler metals (see Table 2-18) on T-specimens of the NDS 300 material (1900°F nitride). These tests resulted in the selection of the five brazing alloys shown in Table 2-19 for further evaluation.\* In this analysis cyclic oxidation tests were run at 1600° and 1800°F with the five brazing alloys coated onto the NDS 300 baseline material. From early visual results of the cyclic oxidation tests, two filler metals--100B and the DF-4A/4B mix--were chosen to evaluate the effects of the filler metal coating on a heat exchanger plate. For this evaluation NDS 300 material was given the full 30-min nitride processing at 1900°F. These two fillers allowed assessment of three conditions: (1) nonpenetrating, boron-free, high-silicon materials (100B); (2) boron-penetrating alloys (DF-4A/4B); and (3) aluminum-containing alloys (DF-4A/4B). After fusing, the DF-4A/4B-coated specimens were given an additional postbrazing diffusion cycle for 3 hr at 1950°F, as recommended by the supplier.

Cyclic oxidation testing of the braze filler metals in Table 2-19 was conducted, with samples removed for metallographic examination of cross sections after 400 and 1023 hr (i.e., 436 and 1116 heating cycles of 55 min). Qualitative assessments of oxidation resistance made on polished cross sections are given in Table 2-20. These results generally correlate well with visual observations of the specimens during testing, except for the DF-4A/4B

\*The last (sixth) alloy shown, MBF-50, was added later as an alternate to alloy 100.

TABLE 2-18  
BRAZING ALLOY SCREENING TESTS ON NDS 300

Brazing Alloy Designations*	Major Alloy Elements (Ni-base Unless Indicated)	Vendor Suggested Braze, °F	Test Temperatures, °F	Test Results and Observations
AMS 4777	Cr-Si-B	1900 to 2150	1950	Flowed; erosion
AMS 4778	Si-B	1900 to 2150	1950	Flowed; erosion
AMS 4779	Si-B	1975 to 2150	2050	Flowed
AMI 300	Cr-Si-Mn	2025 to 2125	2050	Flowed
AMI DF-4A	Cr-B-Co-Ta	2050 to 2175	2050, 2125	OK at 2125
AMI DF-915/RB2	Cr-B-Fe-Mo-Ta-W	2000 to 2050	2050	Cold; liquated
AMI DF-915/B1	Cr-B-Fe	2100 to 2225	2100	Very cold
AMI DF-915/B2	Cr-B-Fe	1975 to 2050	2050	Cold; liquated
AMI DF-915/B2 5	Cr-B-Fe	1985 to 2050	2050, 2125	Flowed 2125°F with erosion
AMI 915E	Cr-Si-B-Co-Fe	2075 to 2200	2100	OK, slight skull
Microbraz 210	Cr-Si-B-Ni-W (Co-base)	2100 to 2250	2200	Flowed; OK
AMI 100B	Cr-Si	2160 to 2200	2100, 2200	OK; some erosion at 2200°F
AMI DF-3	Cr-B-Co-Ta	2100 to 2200	2200	Flowed with skull; erosion
AMI DF-5	Cr-B-Ta-Al	2150 to 2225	2200	Skulled; cold
Microbraz 150	Cr-B	2075 to 2150	2050, 2100	Flowed at 2100°F; erosion
Microbraz LC	Cr-Si-B-Fe	1975 to 2200	2100	Cold; liquated
AMI 100	Cr-Si	2075 to 2200	2100	OK; needs 250°F
AMI 915	Cr-Si-B-Fe	2050 to 2150	2100	Flowed; heavy erosion
AMI DF-4B	Cr-B-Co-Ta-Al	2100 to 2250	2100	Flowed; heavy skull
AMI 915/RB2+A1	Cr-B-Fe-Mo-Ta-W-Al	2100 to 2225	2200	Heavy skull
AMI S57B	Cr-Si-B-Ni-Ta-Al (Co-base)	2100 to 2175	2200	Heavy skull
AMI DF-6	Cr-B-Ta	2175 to 2225	2150	Flowed; erosion
AMI Fe-Mn-Al-1	Mn-Cu-Al-Si-B (Fe-base)	2175 to 2250	2200	No flow, very heavy skull
AMI Fe-Mn-Al-2	Mn-Cu-Al-Si-B (Fe-base)	2115 to 2175	2200	No flow, very heavy skull
MBF-50	Cr-Si-B	2075 to 2200	2120	Flowed well

\*AMI = Alloy Metals, Inc.    Microbraz = Wall Colmonoy (Trade Name)    AMS = Aerospace Material Specification  
MBF = Metglas Brazing Foil, from Allied Corp.

TABLE 2-19

## BRAZING ALLOYS SELECTED FOR FURTHER STUDY

Code Number*	Brazing Temperature, OF	Composition in Percent by Weight									
		Ni	Cr	Si	B	Co	Ta	Al	W	C	Y
100	2120	71	19	10	--	--	--	--	--	--	--
100B	2120	60	30	10	--	--	--	--	--	--	--
400	2200	17	19	8	0.8	51	--	--	4	0.4	00
915E	2125	58	15	4	2.8	20	--	--	--	--	--
DF-4A/4B	2200	70	14	--	2.7	10	3	1	--	--	0.02
MBF-50	2120	73	18	7	1.4	--	--	--	--	--	--

\*Designations for filler metals provided by Alloy Metals, Inc., Troy, Michigan. DF-4A/4B is a blend of two powders in a ratio that provides nominally 1-percent aluminum. MBF-50 is a designation of Allied Corp.

TABLE 2-20

## RESULTS OF CYCLIC OXIDATION TESTING OF BRAZING ALLOYS\*

Brazing Alloy	Test Temperature, OF	400-hr Condition**	1023-hr Condition**
None	1600	-	G
4A/4B	1600	P	P
100	1600	G	F
100B	1600	E	G-E
400	1600	G-E	G
915E	1600	P	P
None	1800	-	G
4A/4B	1800	P	P
100	1800	G	F-G***
100B	1800	E***	E***
400	1800	G-E	G-E
915E	1800	P	P

\*Tests were conducted in air, using 55 min in the furnace and 5 min in ambient air each hour. The test equipment was automated and ran continuously.

\*\*P = poor

F = fair

G = good

E = excellent

\*\*\*Kirkendall porosity noted.

mixture, which appeared very good externally. The higher-chromium materials (100, 100B, and 400) had the best oxidation resistance, but development of Kirkendall porosity was apparent in alloy 100B after 400 hr and in alloy 100 after 1023 hr at 1800°F (see Figure 2-16 a through c). Alloy 100 behaved well over 1023 hr at 1600°F (Figure 2-16d). Oxidation of uncoated NDS 300 control specimens appeared minor, as shown in Figure 2-16 e and f.

One of the most consistent features associated with these brazing filler metals was the tendency to skull and not flow when high aluminum contents were present, such as occurred with DF-4B and the Fe-based Cu-Al-Si-B-Mn alloys (Table 2-18). This suggests a possible reaction between aluminum in the brazing alloy and excess nitrogen in the NDS 300 material. Regardless of the mechanism, it is apparent that high aluminum-containing filler metals are not compatible with the nitride-strengthened material.

Tensile and creep-rupture tests on the specimens of nitrified NDS 300 braze coated with either 100B or the DF-4A/4B mix were also conducted. Test results are given in Table 2-21. Only the normal changes associated with braze-coating thin material were observed, i.e., higher tensile strength and lower ductility. Creep test results also fell within the established scatter-band for the baseline material. Similarly, brazeability of the new NDS 200 alloy was demonstrated with J8300 (manganese-modified alloy 100) and with alloy 100, which were used as coatings on creep specimens to provide oxidation resistance. It may be concluded, then, that nitride-strengthened material is not changed by filler metal contact any differently than standard materials. This similarity allowed final brazing alloy selection to be based on braze joint strength and oxidation resistance requirements without additional restrictions imposed due to use of nitride-strengthened material. Although creep strength was not affected, the ambient tensile ductility was reduced by about half (to 9 to 10 percent) with boron-free filler metals, and to about one-quarter (4 to 5 percent) with brazing alloys of high boron content.

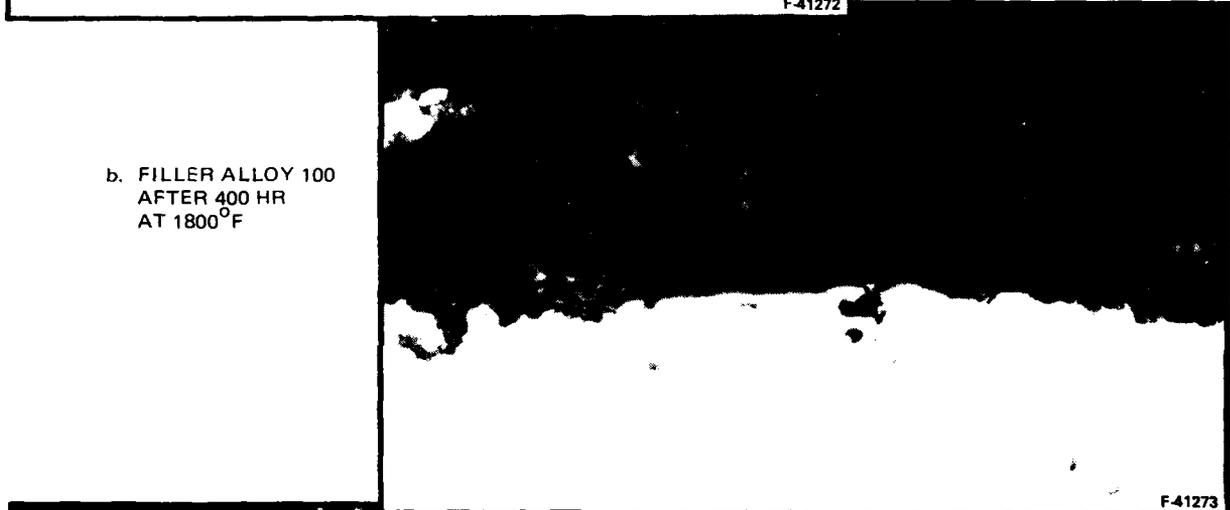
From these test results three strong candidate filler metals were found: AMI 100, 100B, and 400. Each had apparent shortcomings. The 100 alloy has a solidus of about 1975°F, and may be too weak for 1800°F service. AMI 100B (a proprietary composition) contains 30 percent Cr, which somewhat negates the strategic metal reduction in the nitride-strengthened metal, and also shows signs of in-service porosity formation. Alloy 400 would have the highest remelt temperature of the three because it contains boron, which diffuses away; however, alloy 400 is high in cobalt, which is also a strategic element.

A few high-temperature braze peel tests were therefore conducted at 1800°F, as illustrated in Figure 2-17; data are summarized in Table 2-22. For the first round of tests shown at the top of Table 2-22, the boron-strengthened alloys such as 915 and 915E tended to break partly in the braze joints in this test, whereas boron-free alloys 100 and 100B broke in the parent metal at the braze fillet toe (see Figure 2-17 b and c). The second test series (lower half of Table 2-22) was designed to improve brazing flow by mixing in a small amount of a boron-containing filler metal at a level low enough to minimize the parent metal embrittlement effects noted earlier with the DF-4A/4B mix, and to avoid broken braze joints such as seen in Figure 2-17c. This set of samples was fabricated by different personnel, and the sensitivity of the test to preparation technique was evident in the reduced levels of breaking stresses. Although all failures were in the parent metal, apparently there were significantly higher bending moments in this second set due to a slight misalignment of the two sides of the peel test specimen. This does, however,



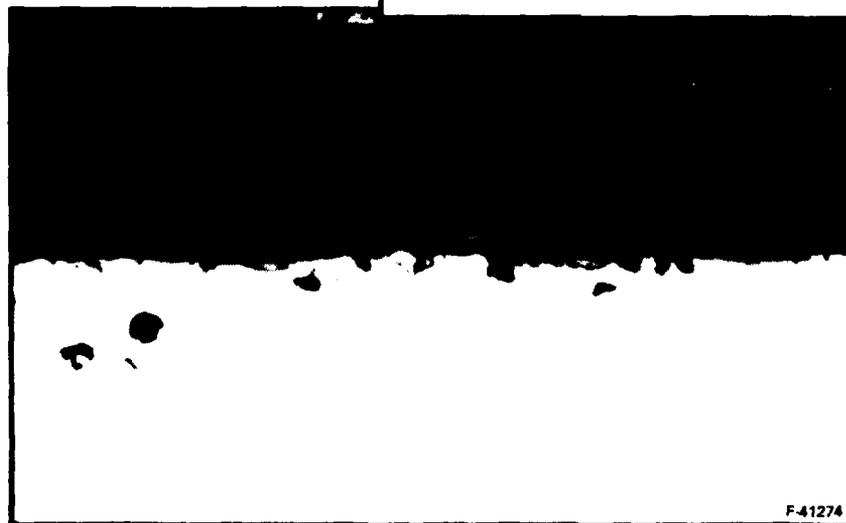
a. FILLER ALLOY 100B  
AFTER 400 HR  
AT 1800°F

F-41272



b. FILLER ALLOY 100  
AFTER 400 HR  
AT 1800°F

F-41273

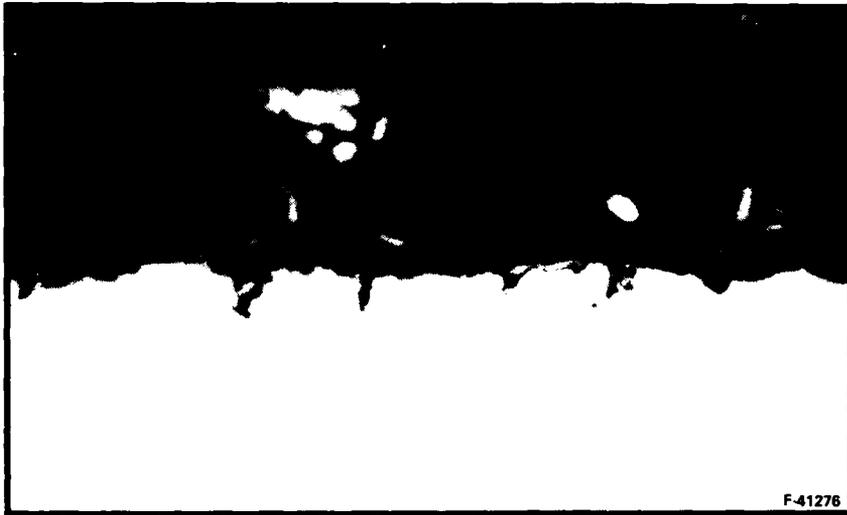


c. FILLER ALLOY 100  
AFTER 1023 HR  
AT 1800°F

F-41274

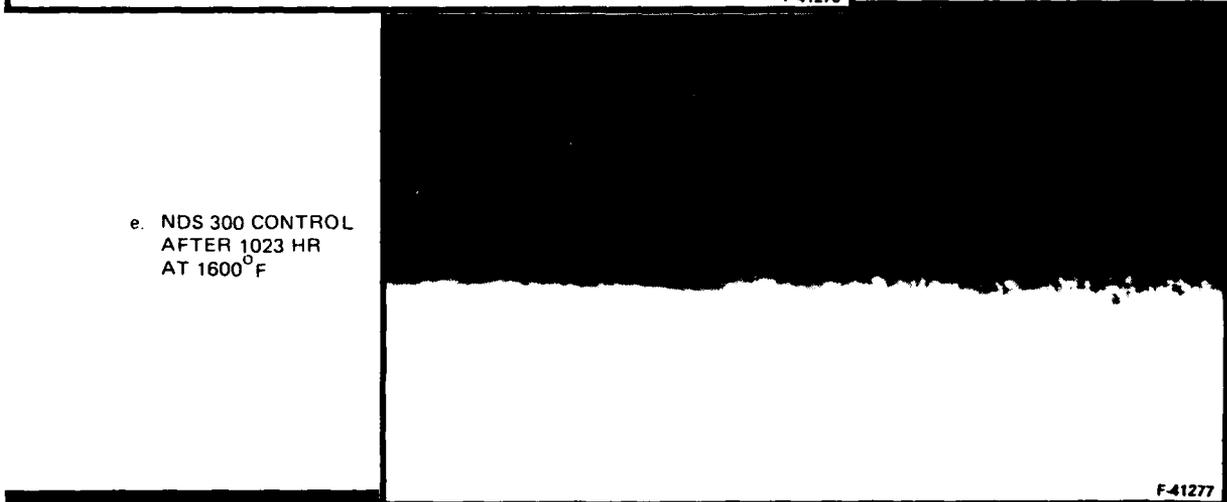
F-41275

Figure 2-16. Cross Sections of Braze-Coated Cyclic Oxidation Test Specimens (Magnification, 250X)



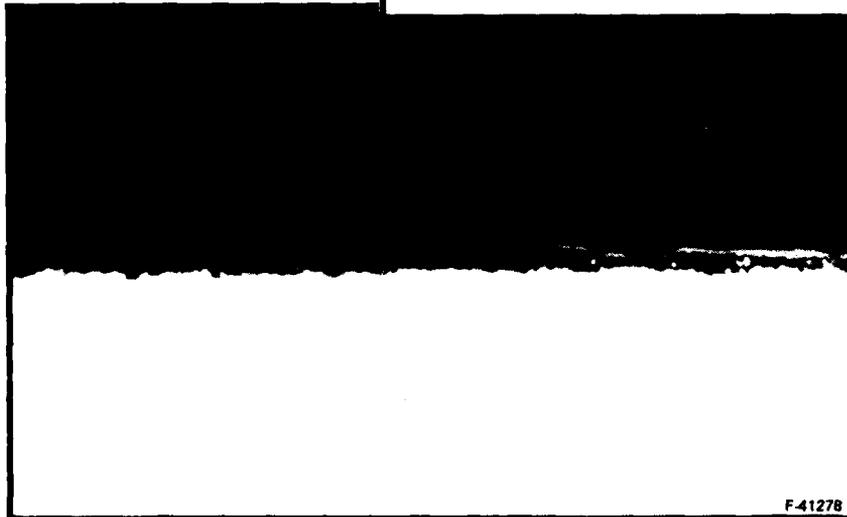
d. FILLER ALLOY 100  
AFTER 1023 HR  
AT 1600 F

F-41276



e. NDS 300 CONTROL  
AFTER 1023 HR  
AT 1600 F

F-41277



f. NDS 300 CONTROL  
AFTER 1023 HR  
AT 1800 F

F-41278

F-41279

Figure 2-16. (Continued)

TABLE 2-21

## TEST RESULTS ON BRAZE-COATED NDS 300\*

Braze Filler Metal	Room-Temperature Properties,			Creep Test Data: Hours to 1% Extension	
	0.2% Yield, ksi	Ultimate, ksi	% Elongation over 1 in.	Hours to 1% Extension	
				1500°F/20 ksi	1650°F/15 ksi
None	83, 88	142, 146	24, 20	71 (585), 187 (586)	
100B	74, 75	97, 111	10, 9	205 (587)	40 (588)
DF-4A/ 4B	100, 110	143, 160	4, 5	280 (590)	281 (591)
None (controls with braze runs)	76, 73	132, 138	21, 28		

\*The 0.010-in.-thick material was given a full 30-min nitride at 1900°F (Code 143, see Appendix A) plus a standard denitride. Brazing alloy coatings were applied using standard plate-fin heat exchanger fabrication procedures before fusing in vacuum per Table 2-19. Tensile test data include brazing alloy thickness, whereas creep loading is based on original cross sectional area. The three-digit numbers in parentheses correspond to creep test codes in Appendix B.

resemble the real-life situation in a fabricated plate-fin heat exchanger, where joint efficiency--based on comparison to parent metal strength--is never unity, even when the filler metal itself does not fail.

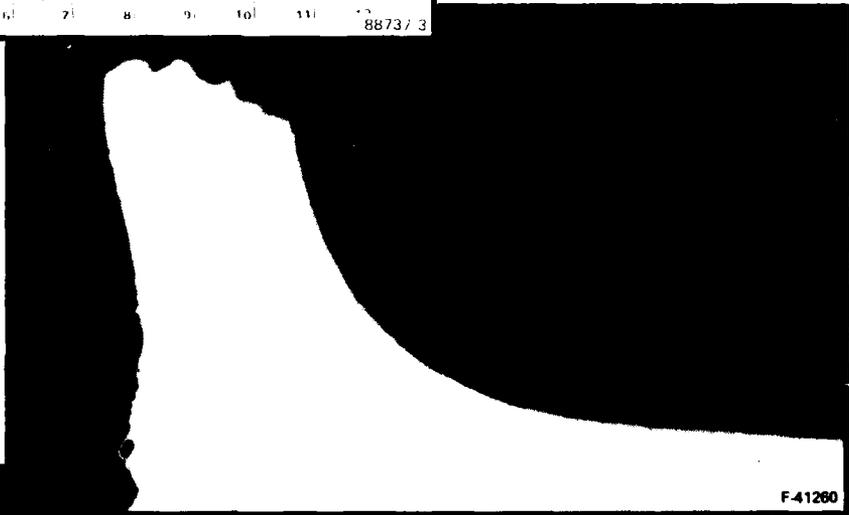
To further test the strength of AMI 100 (19Cr-10Si-bal. Ni) brazed joints in creep, a 1-in.-square section of NDS 300 heat exchanger fin 0.131 in. high, (10 to the inch) was brazed between two parallel-faced 1-in.-diameter studs machined from 347 stainless steel (Figure 2-18). The completed assembly was stress-rupture tested at 1800°F under a load which would simulate a 200 psig recuperator pressure (equivalent to a 2000 psi fin material stress). After 138 hr without failure, the load was increased to give a fin stress of 3000 psi for an additional 2-hr period and was then increased to 4500 psi where it finally failed after an additional 2 hr (142 hr total). Failure was entirely in the bare NDS 300 fins.

This test demonstrated the excellent high-temperature strength of the AMI 100 filler metal for this recuperator application. The backup brazing alloy chosen was Allied's MBF-50, a glassy metal foil modification of AMI 100, which contains about 1.5 percent B and 7.5 percent Si in the same Cr-Ni base. As noted, a small boron addition may be desirable to enhance brazing flow. The use of braze foil may also prove to be of value in manufacturing recuperators.



a. PEEL TEST SPECIMENS  
 (LEFT, READY FOR ASSEMBLY;  
 CENTER, ASSEMBLED WITH  
 2-MIL SHIMS;  
 RIGHT, BRAZED WITH ALLOY 100  
 AND TESTED)

b. FAILURE IN  
 PARENT METAL  
 (ALLOY 100 FILLER)



c. PRIMARY FAILURE IN JOINT  
 (ALLOY 915E FILLER)

F-41262

Figure 2-17. Braze Peel Test Specimen and Modes of Failure--1800°F Test (Magnification, 75X)

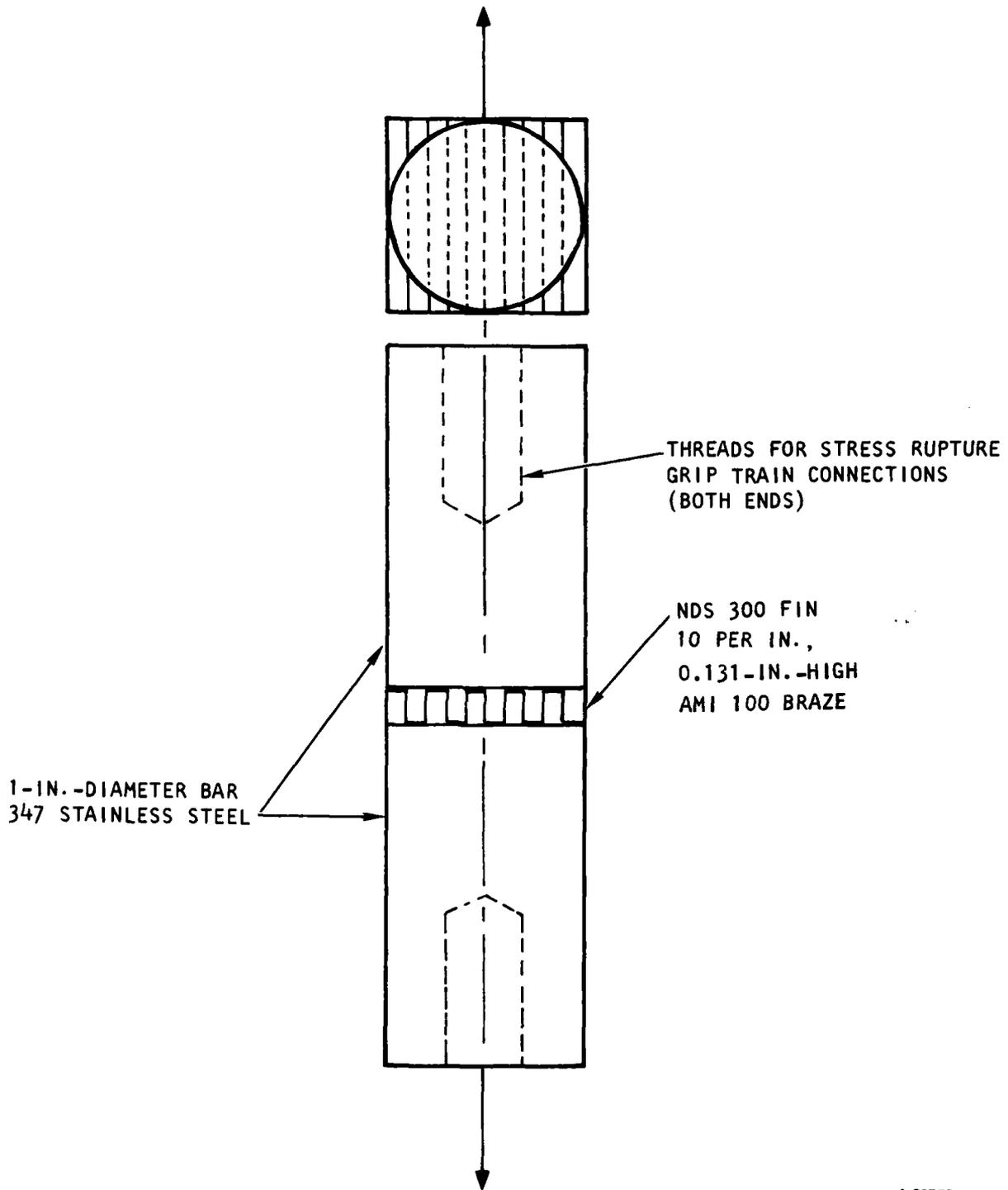
TABLE 2-22  
BRAZE PEEL TENSILE TEST DATA AT 1800°F\*

Brazing Alloy	Breaking Stress, ksi	Failure Location
100B	23.0	Top of fillet
100B	11.9	Top of fillet
915	10.0	Inside braze joint
915E	13.2	Inside braze joint
915E	19.1	Inside braze joint
100	23.0	Top of fillet
100	22.5	Top of fillet
100	13.0	Top of fillet
100	3.6	Tore; bad sample
100+ 10% 4777**	6.7	Top of fillet
100+ 20% 4778**	9.6	Top of fillet
100+ 20% 4778**	11.2	Top of fillet
100+ 20% 4779**	6.9	Top of fillet
100+ 20% 4779**	7.8	Top of fillet (tore)

\*The 0.010-in.-thick NDS 300 was brazed with filler metals per Table 2-18. The base lot of NDS 300 (code 176) had the following properties at 1800°F:

22.6 ksi, 0.2% yield  
25.5 ksi, ultimate  
3% elongation over 1 in.

\*\*Standard alloys per Aerospace Specification (see Table 2-18).



A-69572

Figure 2-18. Sketch of Brazed Fin-Stud Pull Test Assembly (Not to Scale)

## 2.5 TASK 3, COATING DEVELOPMENT

Because the NDS alloys are low in chromium, intrinsic oxidation/hot corrosion resistance was suspect, especially with the NDS 200 composition. It therefore became essential to evaluate coatings for environmental resistance, particularly in the hot end sections of the recuperator. Both aluminum and chromium were evaluated as diffusion (rather than overlay) coatings.

Three sets of specimens of the NDS 300 baseline material were through-nitrided at 1900°F and then sent to Chromalloy Research and Technology in Orangeburg, NY for coating. Two sets were chromized first; one of those sets was then aluminized along with the third set. Results were partially encouraging; although all aluminized specimens were extremely brittle and could hardly be handled, let alone tested, the chromizing treatment appeared to have been successful, with both ambient yield strength and creep strength matching the properties of the uncoated material, as determined in separate control tests (Table 2-23). Ductility was reduced considerably, with all samples failing in the fillet radii. This indicated possible notch sensitivity. The vendor reported that chromized material is normally not significantly embrittled, but the thin gage may have been a factor. The samples analyzed showed very high chromium at the surface (more than 50 percent); this explains the embrittlement, which became even worse after 100 hr at 1650°F. Oxidation testing of the coupons coated along with the mechanical test specimens was cancelled because the samples did not represent the state of the art.

The coating vendor also commented that this embrittlement was normal for thin-gage aluminized material, especially iron-based alloys. However, it seemed that with proper processing the aluminum content could be controlled to a much lower level and still be effective. Therefore, an alternate vendor, Turbine Metal Technology (TMT) of Burbank, CA, was used for coating of the second set of samples which also included NDS 200 material. Turbine Metal Technology also provided chromized samples of both NDS 200 and 300. Test

TABLE 2-23  
TEST RESULTS ON THE FIRST SET OF CHROMIZED NDS 300

Condition	Room-Temperature Properties			Creep Test Data: Hours to 1% Extension	
	0.2% Yield, ksi	Ultimate, ksi	% Elongation over 1 in.	1500°F/20 ksi	1650°F/15 ksi
Bare (control)	87	148	22	46(582)	
	85	144	20	>48(0.3%)(583)	
	80	14b	22	346(584)	
Chromized	79	124	6	240(614)	95(615)
	80	102	4		
Chromized (1650°F creep specimen after 1%)	79	85	1		

\*The 0.010-in.-thick material was through-nitrided for 30 min at 1900°F.

\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

data on this set are given in Tables 2-24 and 2-25 and in Figure 2-19. Microstructural analysis showed that the chromizing treatment produced considerable subsurface porosity, and as a result the chromized material was not creep tested. Aluminized material, which was ductile in tensile tests and also provided oxidation resistance, was significantly lower in creep strength than either the baseline NDS 300 bare material or the brazing-alloy-coated NDS 200 alloy. Microstructural evaluation of failed creep test specimens gave indication that the aluminized portion of the structure had virtually no strength, particularly at 1800°F. In cyclic oxidation tests (Table 2-25) the coatings spalled, cracked, and were generally not protective.

TABLE 2-24

MECHANICAL TEST RESULTS ON THE SECOND SET OF COATED MATERIAL\*

Base	Coating	Room Temperature Tensile			Creep-Test Data:** 1% Extension		
		0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	Temp, °F	Stress, ksi	Hours
NDS 200	Cr	64	81	1		Not tested	
NDS 200	Al	71	107	7	1500	22	3(664)
					1650	15	1.5(676)
NDS 300	Cr	77	123	9		Not tested	
NDS 300	Al	68	116	11	1500	20	23(666)
					1650	15	8(667)
					1800	5	6(677)
					1800	2	12(670)

\*Nitriding was at the nominal 1900°-1950°F for 6.5 min for NDS 200 and 15 min for NDS 300. Denitriding was 3 hr at 2025°F. Coated by alternate vendor (TMT).

\*\*Three-digit numbers in parentheses correspond to creep test codes given in Appendix B.

Concurrently, discussions were under way with the original coating house to improve the chromized coating. Samples were provided for their use in setting coating parameters, and a second set of samples was then coated. The test results in Table 2-26 compare the properties of the coated material with those of the uncoated specimens. (Results are complicated slightly by the fact that a coating of some sort must be applied for creep testing NDS 200 at all temperatures and for NDS 300 at 1800°F; typically, AMI 100 brazing alloy was applied in a manner representative of recuperator plate preparation.) Essentially, there were no apparent problems with the chromized coating on NDS 300, but results on NDS 200 suggested a shortened creep life. The chromium level in both alloys was about 35 percent for a depth of 1.5 mils, dropping off quickly to the base level in NDS 300, whereas the gradient was more gradual in NDS 200. However, since these results were favorable for NDS 300, this treatment was selected for further evaluation in plate-fin segments as discussed in Section 2-7.

TABLE 2-25

## CYCLIC OXIDATION TEST RESULTS ON THE SECOND SET OF COATED MATERIALS\*

Base	Coating	Temp, OF	Time, hr	Remarks
NDS 200	Al	1600	415 1008	Sublayer pores, not cracked Pores, parent metal cracked
NDS 200	Al	1800	415 1008	Warts through outer layer <2 mils good metal remaining
NDS 200	Cr	1600	415 1008	Substantial sublayer pores Through oxidized in places
NDS 200	Cr	1800	300	Disintegrated
NDS 300	None	1600	415 1008	Lost 1.5 mils/side Lost 1.75 mils/side
NDS 300	None	1800	415 1008	Lost 2.5 mils/side Lost >3 mils/side
NDS 300	Al	1600	415 1008	Cracks to 1.5 mils deep Cracks plus internal pores
NDS 300	Al	1800	415 1008	Cracks to 1.5 mils deep Cracks, pores, int. oxidation
NDS 300	Cr	1600	415 1008	Pores, oxidized inside Same
NDS 300	Cr	1800	415 1008	Grain boundary attack Gone

\*Cycles were 55 min heating plus 5 min cooling in still air.  
These were samples prepared by the alternate vendor (TMT).

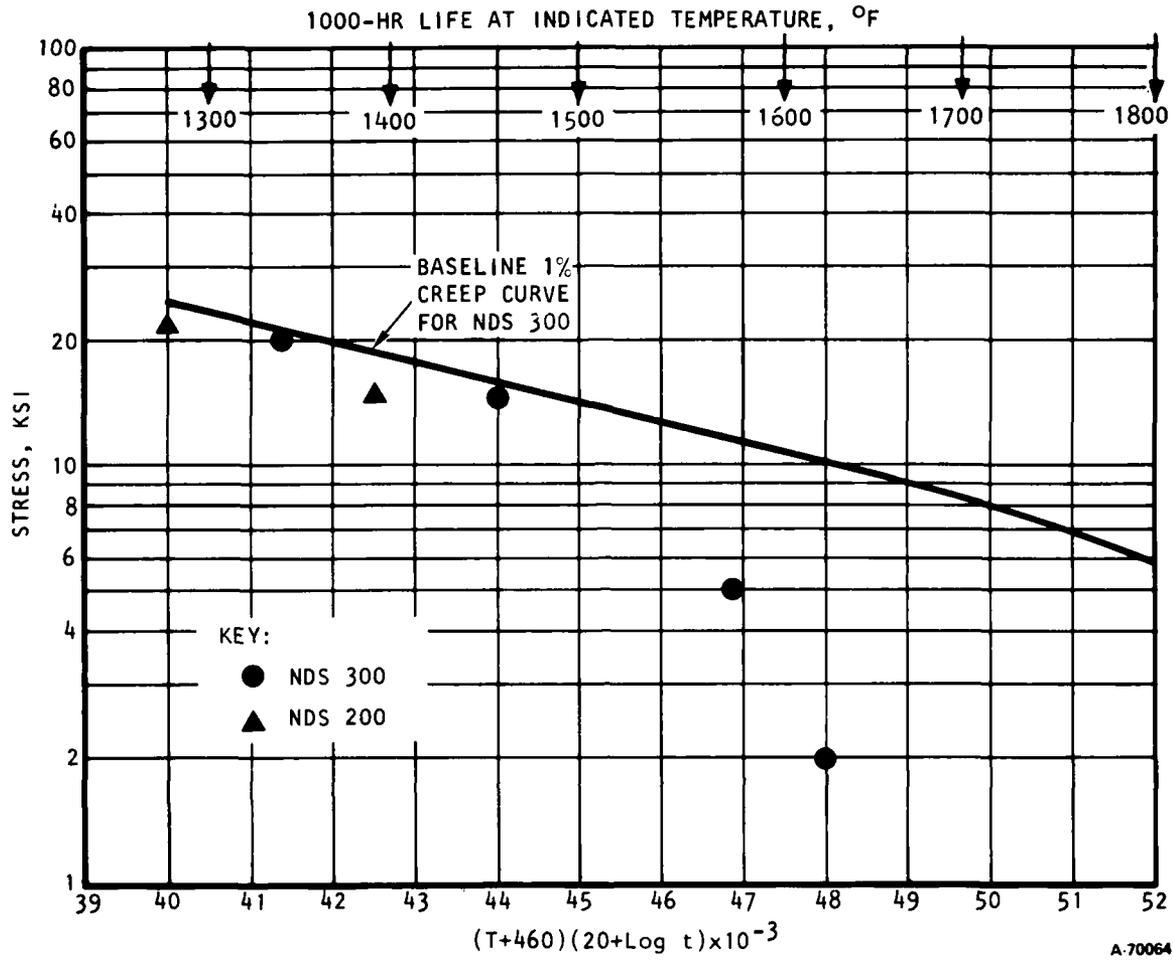


Figure 2-19. Master Plot Comparing 1-Percent Creep Behavior of NDS Alloys after Aluminizing to NDS 300 Baseline Curve

TABLE 2-26

## TEST RESULTS FOR CHROMIZED MATERIALS\*

Material	Room Temperature Tensile			Creep Test Data:**	
	0.2 YS,	UTS,	% Elong.	Hr to 1% Extension	
	ksi	ksi	over 1 in.	1650°F/15 ksi	1800°F/5 ksi
NDS 300	72	143	17	110(720)	11(726)
NDS 300/Cr	82	134	10	159(718)	28(723)
	82	140	14		
NDS 200	86	143	20	20(729)	
NDS 200/Cr	70	93	9	3.7(719)	
	89	113	6		

\*Second set from original vendor

\*\*Three-digit numbers in parentheses correspond to test creep codes in Appendix B.

Since recuperator plates normally must be fully coated with a nickel-based filler metal, an alternate test scheme was developed. The loss of strength reported above for aluminized NDS materials was undoubtedly a result of direct interaction of the aluminum with the TiN dispersoid which is used to provide strength. For recuperator plates that are coated with brazing filler metal, it may be predicted that deep aluminum diffusion will not occur; instead, a nickel-aluminide coating will form only on the brazing alloy and will not effect the strength of the underlying parent metal. This concept would allow coating of brazing alloy and exposed fins and also would work hand-in-hand with use of an alternate gas-side fin material which does not require much strength, yet could be coated for hot corrosion/oxidation resistance. To test this concept, samples of NDS 300 material were coated with AMI 100 and sent to the alternate vendor for aluminizing. Test results are shown in Table 2-27. Group B NDS 300 samples had a heavier filler metal coating than material from run 282. Clearly, results are more promising, although some refinement is needed, perhaps in the brazing alloy, since high silicon is reported to cause adverse interaction with diffusion aluminide coatings.

Test results to this point indicated that NDS 200 would definitely have to be coated for recuperator service, and NDS 300 would probably have to be coated where temperatures exceed 1650°F. However, it was not the purpose of this task to attempt to define and test for hot corrosion; rather, it was to identify coatings that would provide increased hot corrosion resistance without significantly reducing the excellent high temperature strength characteristics of the NDS materials. As has been noted above, protection of the more highly stressed recuperator plates is achieved automatically through the brazing alloy, which is normally applied over 100 percent of the plate area. In this context, the selection of the AMI 100 filler metal described above was

TABLE 2-27

TEST RESULTS FOR BRAZE-COATED ALUMINIZED MATERIALS\*

Material Code	Condition**	Room Temperature Tensile****			Creep Test Data:***		
		0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	Hr to 1%		Hr to Rupture
					16500F/12.5 ksi	17250F/8 ksi	
282	Brazed	81(121)	103(154)	5	324(808)	--	743(806)
282	Brazed and aluminized	77(85)	118(130)	15	21(809)	--	386(807)
B	Brazed	83(109)	127(163)	10	--	122(786)	--
B	Brazed and aluminized	--	--	--	165(825)	129(820)	--

\*Second set from alternate vendor. All samples coated with AMI 100.

\*\*B samples had heavier braze coating before aluminizing.

\*\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

\*\*\*\*Numbers in parentheses are corrected to original area before braze coating.

based, in part, on the excellent oxidation resistance displayed in cyclic testing. Exposed fins must be protected, however, especially in the presence of environmental contaminants. Strength requirements of fins (for pressure containment) are generally low on the air side, however, and, on the gas side, where the potential hot corrosion problem exists, stress levels are close to nil.

Therefore, the use of NDS material for fins probably constitutes an overkill insofar as strength is concerned. Commonly used high-temperature alloys, such as type 310 stainless steel and Incoloy 800H, or perhaps, in the extreme, Inconel 625, would make excellent high-temperature fin materials, depending on final strength requirements. Furthermore, Incoloy 800H, which can contain some free titanium, is also capable of being strengthened by the though-nitriding process. To check briefly the use of Incoloy 800H as a potential fin material, tests were conducted (see Table 2-28) for 10-mil material which had been braze cycled only, braze cycled plus aluminized or nitride strengthened (run 230, Appendix A). Test results for Incoloy 800H that had been aluminized, but not nitride strengthened, indicate creep strength levels slightly below IN625, but still very acceptable for exhaust gas-side recuperator fins. These concepts are presented schematically in Figure 2-20.

A number of coated test coupons were cyclic oxidation tested as described in Section 2.4. The results were mixed. (See the summary of results in Table 2-29 and in Figure 2-21, which shows representative metallographic cross sections.) The aluminide-coated Incoloy 800H performed fairly well; some cracking could be tolerated in gas fins that are only needed for heat conduction. Most diffusion-coated NDS 300 specimens did not hold up nearly as well as the brazing filler metal coatings (described in Task 2) or the AMI 100-coated creep test specimens.

## 2.6 TASK 4, FABRICATION DEVELOPMENT

In addition to brazing requirements, recuperator fabrication is also intimately associated with formability, which was a primary subject of this task. There were two items of concern: (1) forming of fins for panel tests in Task 5, and (2) ensuring that there was sufficient ductility in candidate plate alloys to allow recuperator fabrication. Current thin-sheet materials for recuperators show at least 20-percent elongation in room-temperature tensile tests. This 20-percent level was assumed as an aim property for any new material, although it was recognized that design changes could lead to a lower acceptable ductility level. However, any level below 15 percent would be extremely questionable. An indication of the forming complexity involved may be obtained from Figure 2-22, which shows a section of one current production recuperator.

The first round of information needed to assess the fabricability of the NDS alloys was, therefore, tensile ductility. As an additional measure of formability, the standard ball punch deformation test per ASTM specification E643 was used. Results are summarized in Table 2-30; included are data for both as-nitrided plus denitrided material and material after stretch-leveling on a Baldwin tensile testing machine. Also included are data for 14Cr-4Mo stainless steel, a current AiResearch recuperator plate material. Visual comparisons of NDS 200 and NDS 300 test results may be obtained from Figure 2-23.

TABLE 2-28

TEST RESULTS FOR INCOLOY 800H

Condition	Room Temperature Tensile			Creep Test Data:*** Hr to 1% at			
	0.2% YS, ksi	UTS, ksi	% Elong. over 1 in.	1650°F/2 ksi	1725°F/1 ksi	1800°F/2 ksi	1800°F/4 ksi
Braze-cycled	25	71	24	--	--	18(742)	--
Aluminized*	24	43	16	--	--	3(743)	--
Nitrided	39	84	19	--	--	--	38(694)
Aluminized**	27	60	16	52(816)	15(819)	--	--

\*Group 1 from TMT

\*\*Group 2 from TMT

\*\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B. See also rupture data.

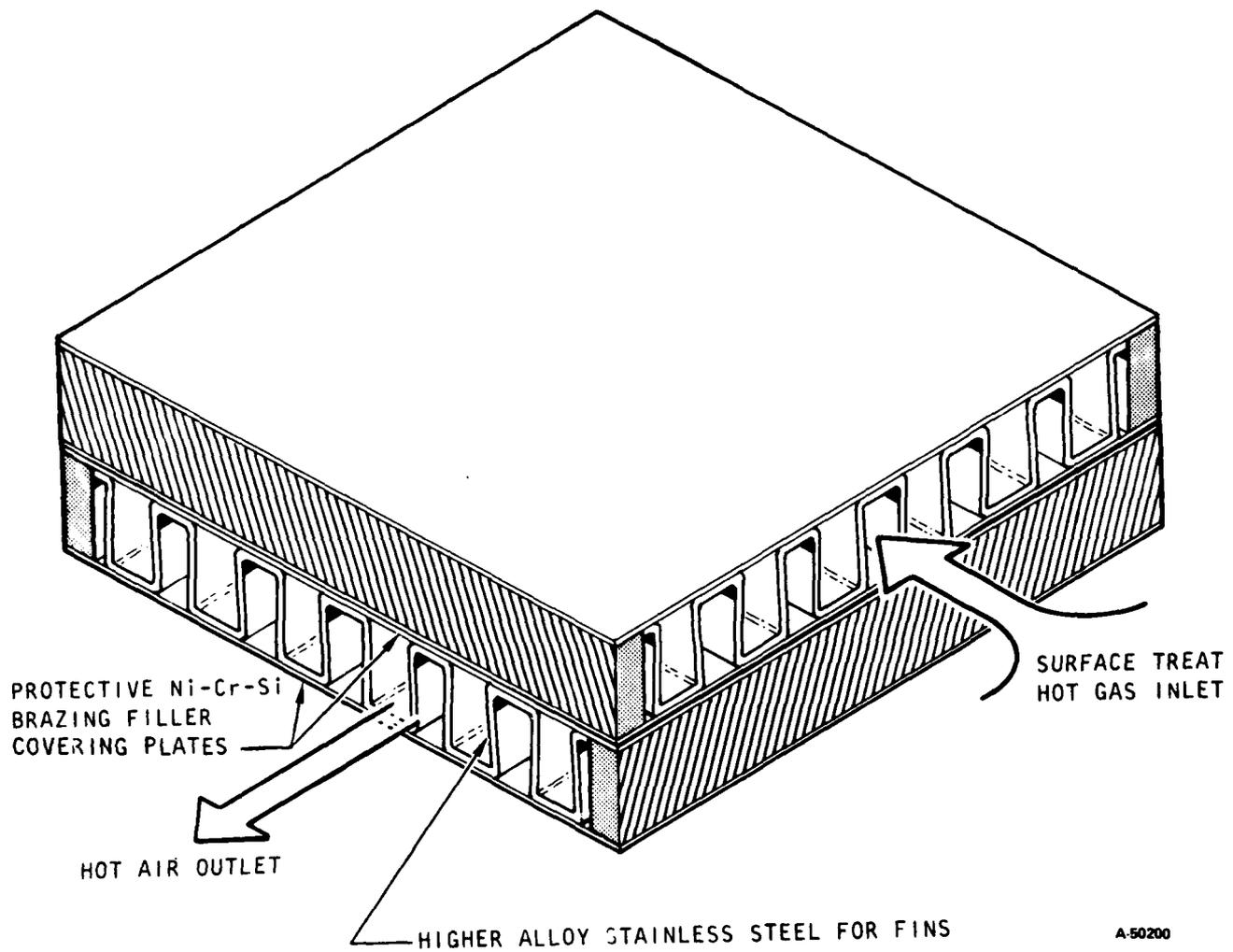


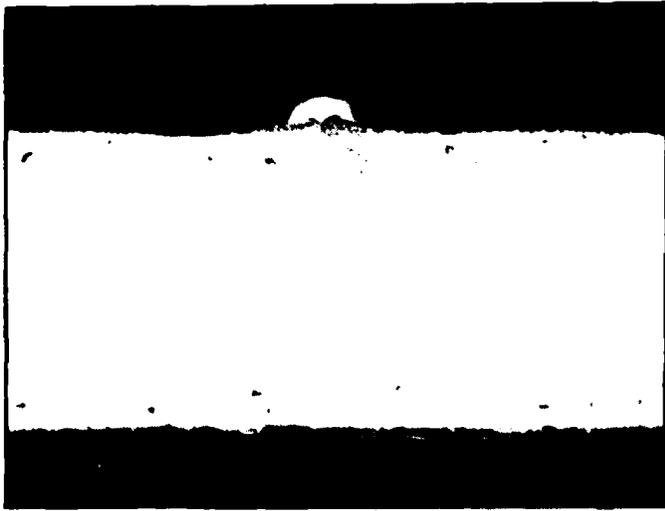
Figure 2-20. Options for Achieving Environmental Protection,  
Singly or in Combinations

TABLE 2-29

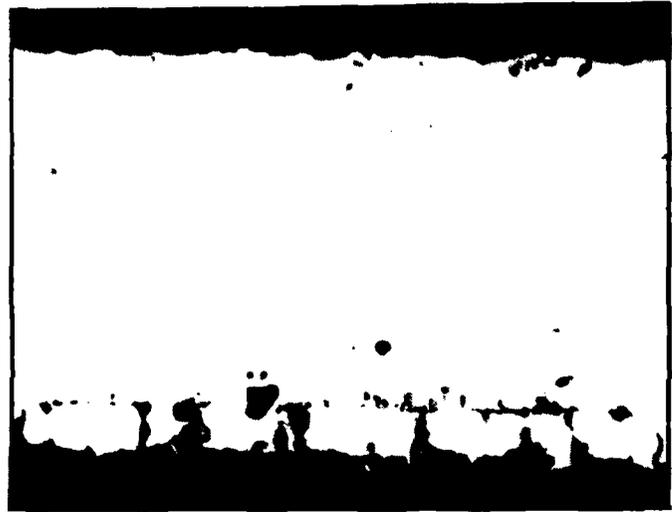
CYCLIC OXIDATION TEST RESULTS  
ON COATED MATERIALS\*

Base	Coating	Temp, OF	Time, hr	Remarks
NDS 200	Cr	1800	436 922	Broke in half Nearly disintegrated
NDS 300	None	1600	701	Warts and internal voids
		1800	476 922	Oxidized through Oxidized through
NDS 300 with AIM 100	None	1600	701	Cracking and mild attack on braze where thin
		1800	701	
NDS 300 with AIM 100	Al	1600	701	Coating separated at braze-NDS interface
		1800	436 922	Heavily oxidized Nearly disintegrated
NDS 300	Cr	1800	436 922	Oxidized through Disintegrated
Incoloy 800H	Al	1800	436 922	Coating cracked; slight attack underneath at longer time

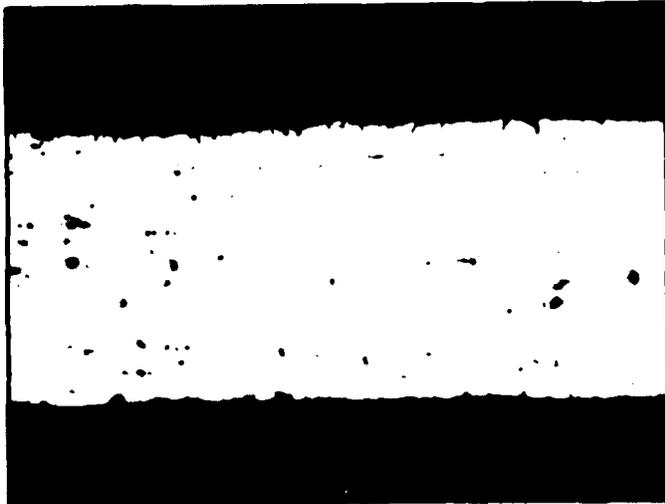
\*Cycles were 55 min heating plus 5 min cooling in still air. Chromized samples were prepared by the original vendor, aluminized by the alternate vendor.



a. NDS 300 CONTROL  
701 HR AT 1600°F



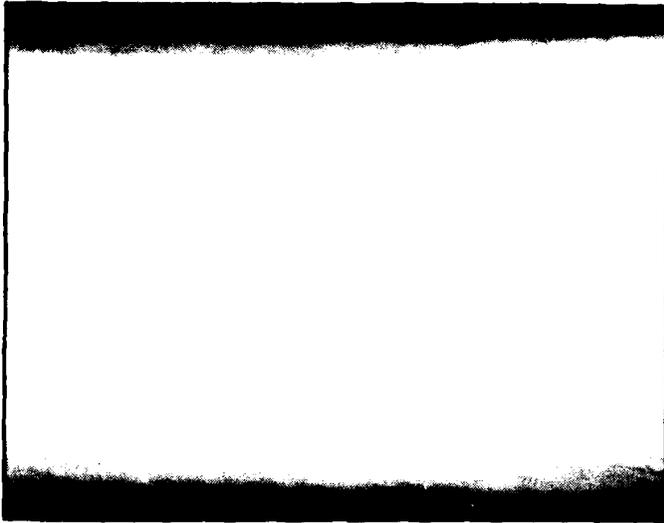
b. NDS 300 COATED  
WITH AMI 100  
701 HR AT 1800°F



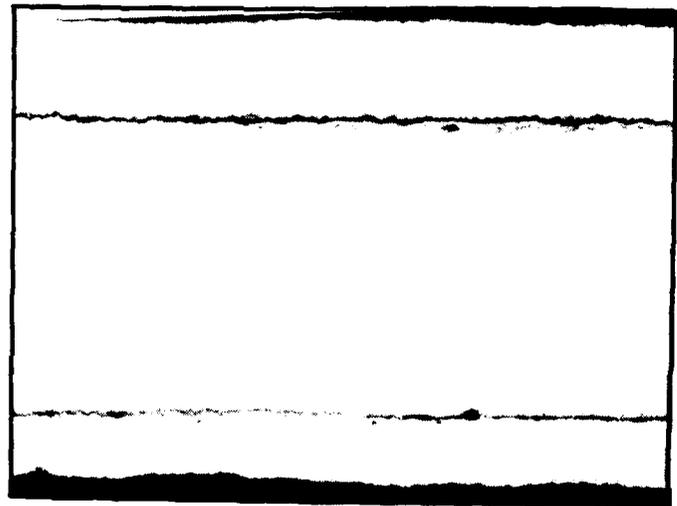
c. NDS 300 CHROMIZED  
BY ORIGINAL VENDOR  
(SECOND TRIAL)  
AS RECEIVED.  
SAMPLE PITTED WHEN  
ETCHED TO SHOW  
CHROMIZED LAYER

F-46017

Figure 2-21. Representative Micrographs of Cyclic  
Oxidation Test Specimens--See Table 2-29  
(Magnification, 200X)



d. NDS 300 WITH  
AMI 100 OVERLAY,  
ALUMINIZED BY  
ALTERNATE VENDOR,  
AS RECEIVED



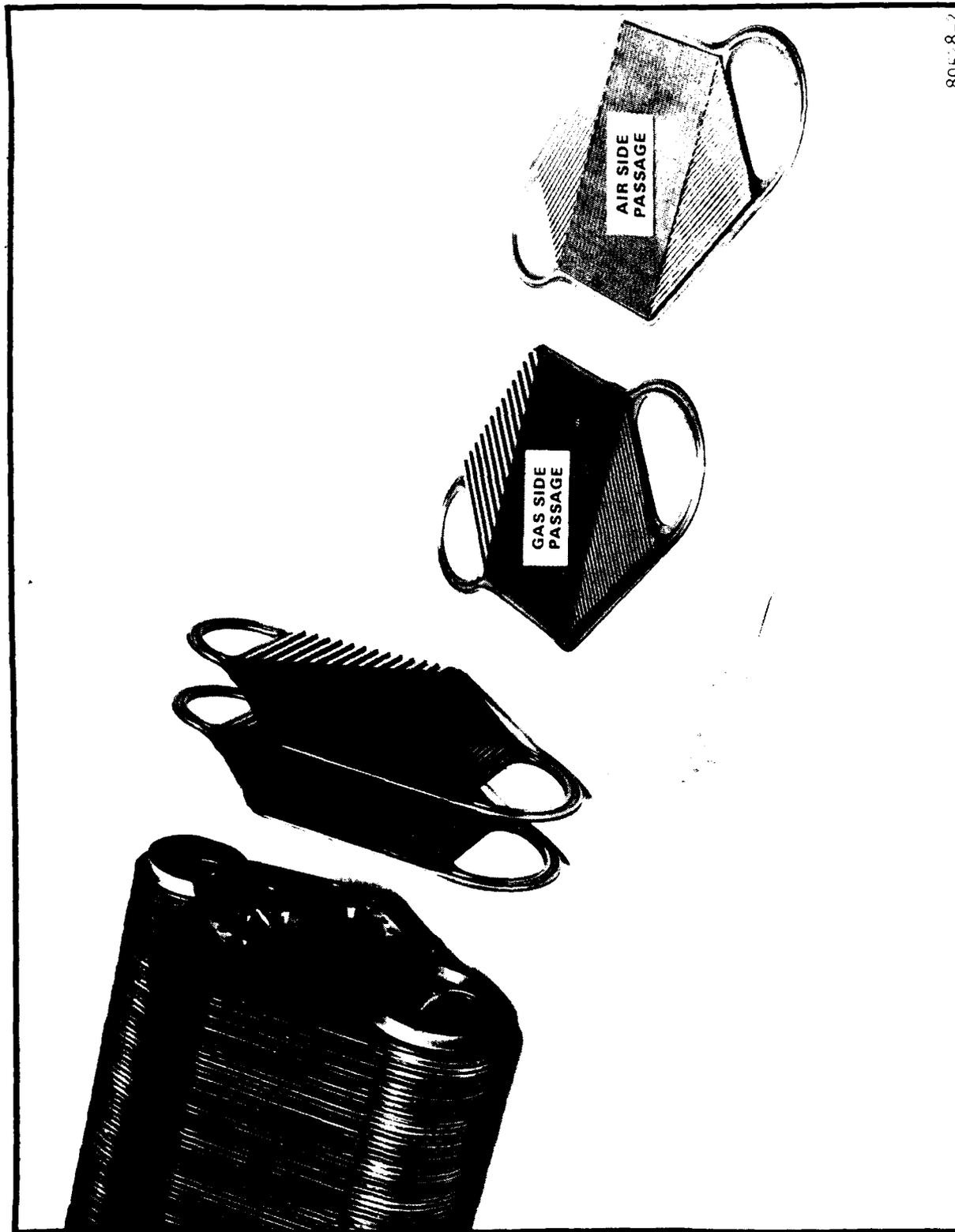
e. NDS 300 WITH  
AMI 100 OVERLAY,  
ALUMINIZED AS  
IN d. ABOVE.  
701 HR AT 1600°F



f. INCOLOY 800H,  
ALUMINIZED AS  
ABOVE FOR NDS 300,  
922 HR AT 1800°F

F-46018

Figure 2-21 (Continued)



80528-2

Figure 2-22. Example of Possible Forming Requirements Associated with Recuperator Fabrication

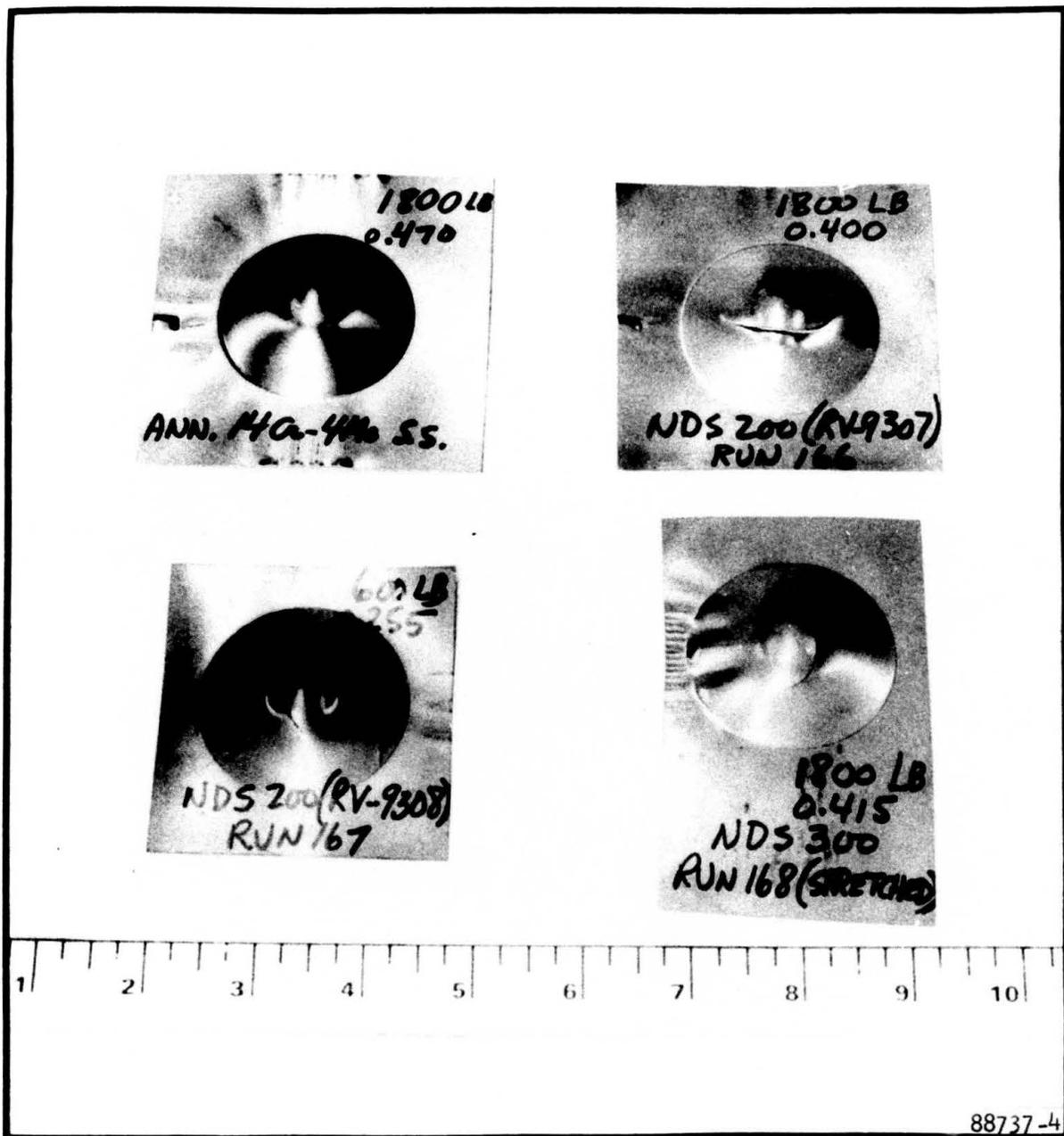


Figure 2-23. Ball Punch Deformation Tests\*

\*Also see Table 2-30

From Table 2-30 it is apparent that tensile ductility levels in excess of 20 percent will be necessary with the NDS alloys. Such ductility is required to achieve the formability attained with 14Cr-4Mo stainless steel.

To evaluate the fin-forming capability of these materials, a small rack capable of holding a 36-in.-long, 3-in.-wide strip for nitriding was prepared (see Figure 2-24). This rack was used to produce NDS 200 and 300 material for tensile, creep, and ball punch testing, as discussed above. Of particular interest was postnitriding flattening and the effect of such an operation on subsequent properties. The introduction of ammonia gas from only one side of the retort (because of space constraints) leads to thermal gradients that cause mild distortion in the sheet. While this probably can be corrected in mass production, the condition had to be corrected before detail fin parts were formed in Task 5. As described earlier, flattening was accomplished by stretching the strip in a Baldwin tensile testing machine. Generally, about 0.5 percent stretch was needed, with 0.1 percent being sufficient for softer materials, and 0.2 percent (the engineering yield point) being the minimum for well-nitrided material. In addition to the normal evaluation by tensile and creep testing (Table 2-31), materials also were subjected to the ball punch deformation test (Table 2-30). Creep test results did not show any deleterious effects due to the postnitridation stretching operation (Table 2-31).

TABLE 2-30  
TEST RESULTS FROM FABRICATION DUCTILITY STUDIES

Material	Nitride Test Code No.	Nitriding Temperature/time, °F/min	Ball Punch		Mechanical Properties		
			Depth, in.	Fail, psi	0.2% Yield, ksi	UTS, ksi	%Elongation over 1 in.
NDS 300	162	1900/30	0.280	750	91, 92	140, 142	11, 16
NDS 300	168	1900/15	0.365	1600	72, 78	135, 137	23, 25
RV-9307*	166	1940/6.5	0.370	1600	83, 88	142, 149	22, 22
RV-9308*	167	1930/7.5	0.255/ 0.398	600/ 1000	81, 82	121, 111	6, 10
14Cr-4Mo	-	None	0.475	1800	67, 68	101, 102	23, 23

\*Heats RV-9307 and RV-9308 are NDS 200 trial compositions, as given in Table 2-11

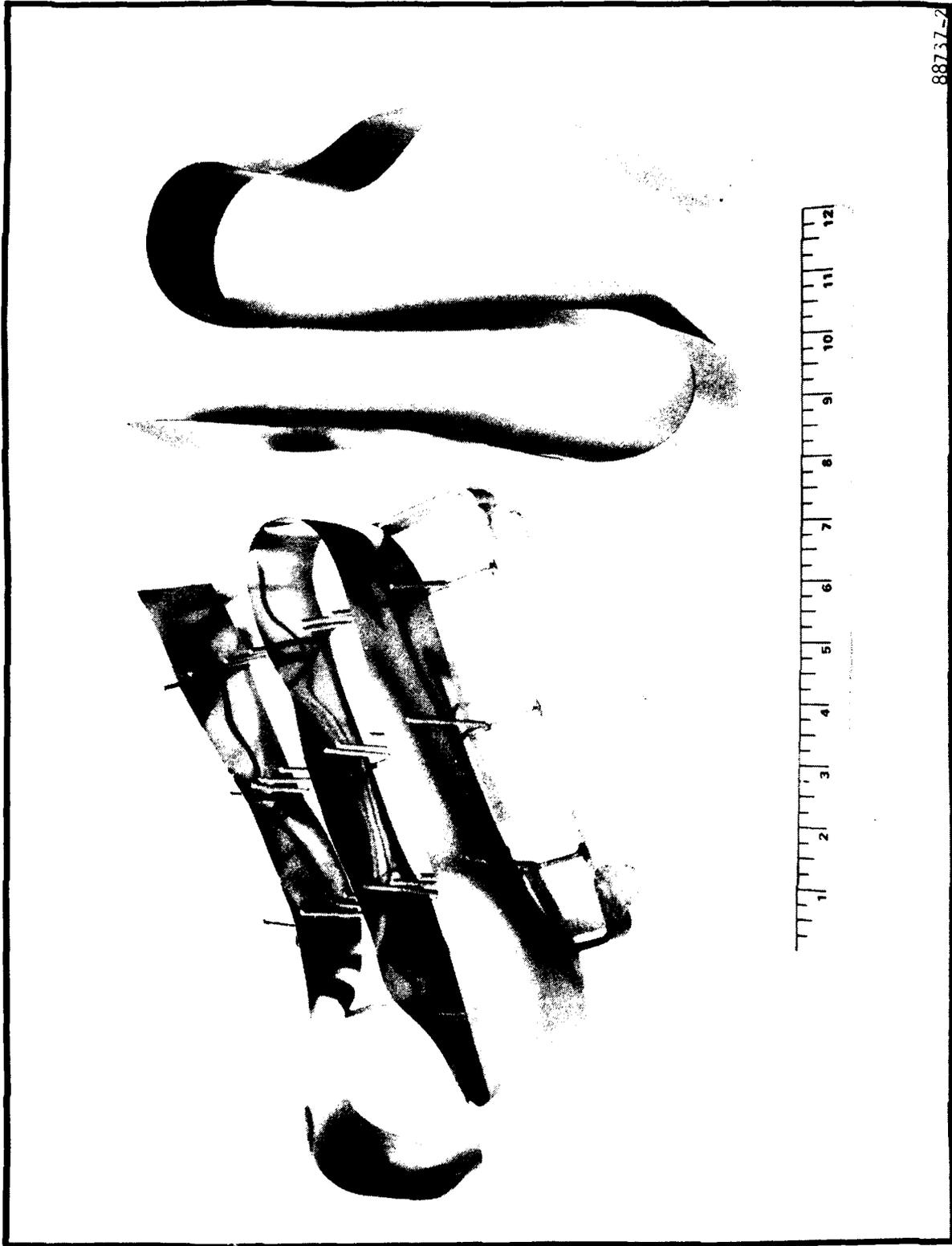


Figure 2-24. Rack Used for Nitriding 36-in.-Long Strip

TABLE 2-31

TEST RESULTS ON STRETCH-FLATTENED NDS 300\*

Nitride Test Code No.	Nitriding Conditions: Temperature/Time OF/min	Amount of Stretch, Percent	Room-Temperature Properties			Creep Test Data:**	
			0.2% YS, ksi	Ultimate, ksi	% Elongation over 1 in.	Hours to 1% Extension at	16500F/15 ksi
162	1900/30	None 0.5	91, 92 100, 103	140, 142 144, 142	11, 16 15, 15	15000F/20 ksi	
168	1900/15	None 0.1	72, 78 77, 80	135, 137 136, 140	23, 25 22, 25	273(611) 130(613)	
169	1925/15	None 0.1	72, 74 70, 74	133, 135 127, 131	24, 25 13, 18	31(612)	
177	1900/15	0.2	82, 83	135, 133	21, 22		98(638)

\*For 0.010-in.-thick material, stretch-flattened in a Baldwin tensile testing machine.

\*\*Three-digit numbers in parentheses correspond to creep test codes in Appendix B.

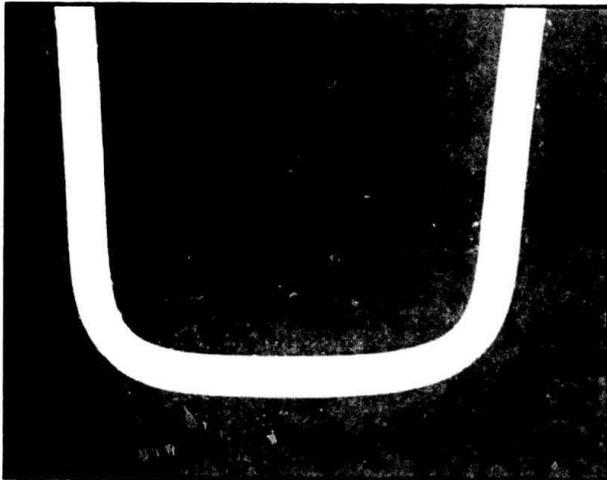
Since NDS 200 would have to be coated for use as fins, the NDS 300 material was selected for fin-forming trials to provide fins for Task 5, Panel Testing. Using 1900°F/15 min nitriding conditions, a total of 22 pieces were prepared to duplicate material of run 177 (Appendixes A and B), each 36-in. long by 1.8-in. wide. These were then batch denitrided at 2025°F for 3 hr in the AiResearch production hydrogen brazing facility, followed by stretching 0.5 percent in a Baldwin Tensile Testing Machine to flatten. All pieces were subsequently formed into heat exchanger fins having plain rectangular geometry, 10 fins per inch, 0.131-in. high. Forming was accomplished without cracking or any other problems. A few pieces of Inconel 625 were run for control. Representative cross sections prepared metallographically are shown in Figure 2-25. As indicated in Figure 2-25b, some fin areas were not completely through nitrided. This was traced ultimately to a broken ammonia distribution tube in the back of the retort. These areas were weaker and tended to narrow during stretch flattening, which provided a way to segregate good versus bad fin.

An experiment was also conducted to determine if the NDS 200 and 300 materials could withstand the forming operations associated with recuperator plate manufacture (see Figure 2-22), since this represents a more severe case than fin forming. A series of samples was prepared from both NDS 200 and NDS 300 using nitriding procedures which would give a range of tensile ductility. These were then subjected to forming experiments using production plate dies. Results, shown in Table 2-32 and Figure 2-26, indicate that acceptable forming can be achieved over a fairly wide range of ductility, thereby allowing use of materials nitrided to the higher strength levels.

Subsequent cycling of formed pieces through a typical 2100°F furnace brazing cycle did not lead to any microstructural or hardness changes in the material that would suggest possible loss of strength as a result of mechanical working (compare Figure 2-26 b and c). Thus, it appears that formability requirements for recuperator fabrication have been met with the NDS materials, with some degree of latitude in processing.

## 2.7 TASK 5, PANEL TESTING

The final step in evaluating the NDS alloys in the laboratory prior to fabricating an actual recuperator module was the testing of small, single-sandwich, plate-fin panels for pressure containment in both burst and creep modes. Panel geometry and assembly is shown in Figure 2-27. The panel geometry selected (i.e., overall external dimensions, including closure bars) was 3 by 4 in., with gas flow in the long direction. This size was picked because of a 3-in. length limitation in the nitriding retort, and also to make maximum use of the available raw NDS alloy stock. The fin was the plain rectangular type--10 to the inch, 0.131-in. high--described above for Task 4. Because of the relatively low fin density, braze joint stress was high, as were pillowing stresses in the plate between the fins. Since typical fin stresses in a recuperator are way below the strength of NDS 300, even at 1800°F, this test was designed to analyze the braze joints and the plates under creep conditions.

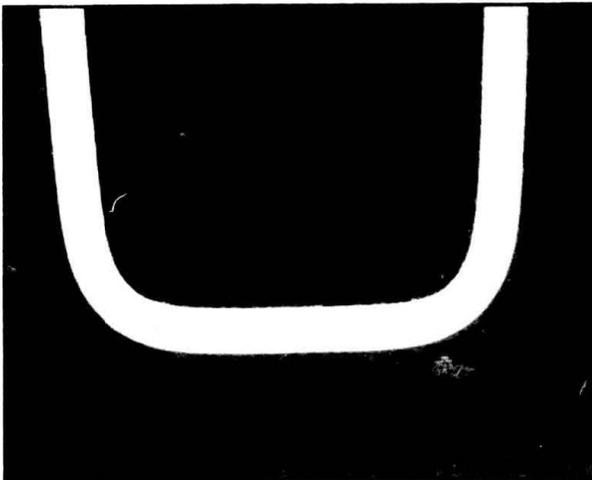


25X



100X

a. FULLY NITRIDED



25X



100X

b. INCOMPLETE NITRIDING

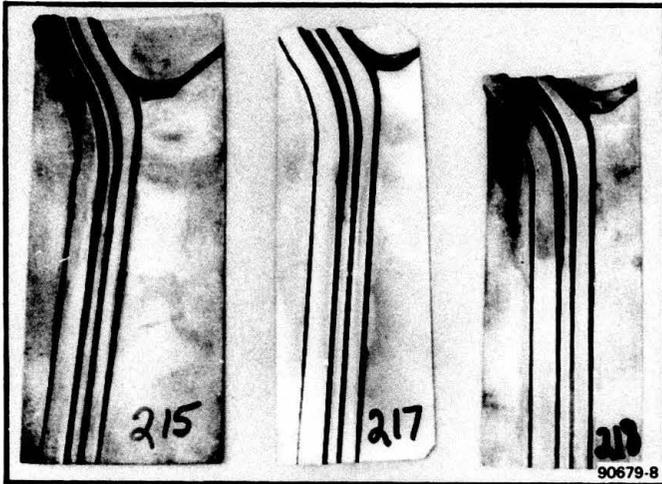
F-46026

Figure 2-25. Sample Microsections of NDS 300 Fin Material  
(Magnifications as Shown)

TABLE 2-32

## RECUPERATOR PLATE FORMABILITY TEST RESULTS

Base	Nitride Test Code No.	Nitriding Conditions	Cup Depth, in.	Forming Press
NDS 200	214	6 min, 1930 avg. (slow heat)	0.355	OK
NDS 200	215	6.5 min, 1940 avg. (fast heat)	0.275	OK
NDS 200	219	5 min, 1926 avg. (slow heat)	0.290	OK
NDS 300	216	15 min, 1940 avg. (hydrogen heat)	0.355	OK
NDS 300	218	10 min, 1987 avg. (hydrogen heat)	0.325	OK
NDS 300	217	15 min, 1925 avg. (ammonia heat)	0.350	OK



a. SAMPLES FORMED IN MANIFOLD  
HOOP-TO-PLATE JUNCTION  
(SEE FIGURE 2-22)



b. NDS 300, RUN  
216, AS FORMED

100X

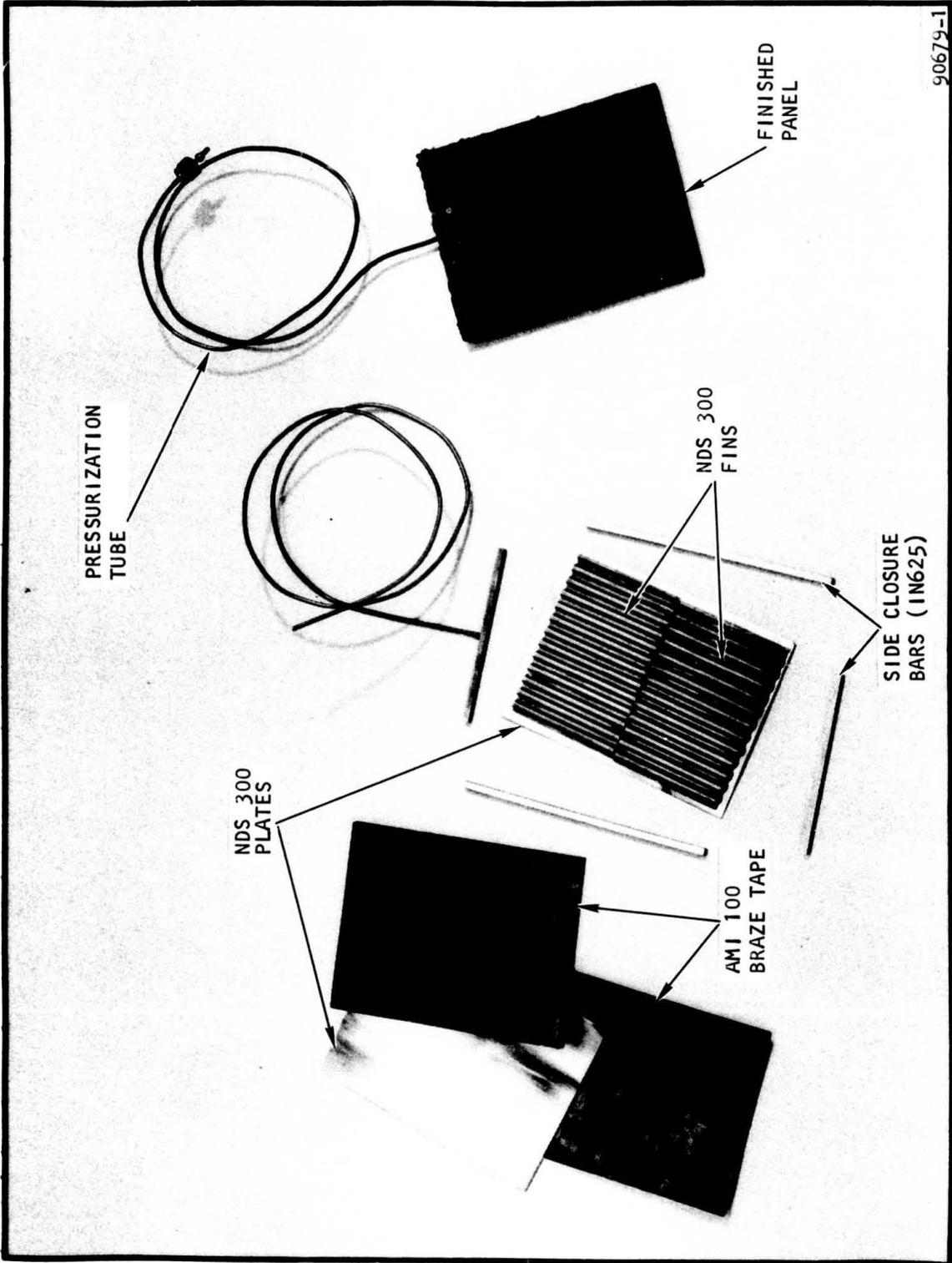


100X

c. NDS 300, RUN  
216, BRAZE  
CYCLED

F-46024

Figure 2-26. Results of Plate-Forming Experiments of NDS Materials  
(See Table 2-32)



F-47178

Figure 2-27. Assembly of Heat Exchanger Panels from NDS 300 Plates and Fins

Early test panels utilized either NDS 200 or 300 for plates. While NDS 300 fins were being fabricated, early tests utilized a standard production Inconel 625 fin. The panels were brazed with AMI 100 filler metal at 2100°F in a vacuum. Several trials were necessary to define brazing parameters. Two panels were then brazed, one each with NDS 200 and NDS 300 plates. The latter was the first good panel; it was pressurized to burst at ambient conditions, failing at 1220 psig. Failure initiated at a small unbrazed area near a corner. Note that 1220 psig is over six times the maximum operating pressure generally associated with a recuperated gas turbine engine, i.e., a maximum pressure ratio of 12:1, or 180 psig. (This was the only panel pressure tested with IN625 fins.)

The second panel fabricated with NDS 200 plates was proof tested to 1000 psig at ambient; then pressurized to 200 psi with argon at 1800°F. Failure occurred in 25 hr, apparently initiated by a combination of pillowing of the NDS 200 plates between the fins and an area of poor brazing caused by insufficient filler metal. The pillowing of the NDS 200 plates apparently was aggravated by the lower creep strength of this material at 1800°F as discussed above in Task 1b. This increased the peel stress on the braze joints, which in turn began to separate.

At this point, emphasis was placed on NDS 300 as discussed in Task 1, and there was no further use of NDS 200 for plates. Several panels were then built and tested, with mixed results. On one panel using all NDS 300 parts with AMI 100 filler a burst of over 2000 psi was achieved at 1650°F. However, a subsequent creep test at 1650°F and 1000 psi on a second panel resulted in nearly instant failure. Results with Inconel 625 plates and fins under the same test conditions were similar. Metallographic examination indicated braze joint failure; stress evaluation suggested pillowing stresses between fins were extremely high and not representative of actual recuperator operation. Accordingly, test pressure levels for NDS 300 panels were selected at 200 psi at 1725°F and 1800°F, and 400 psi at 1650°F; several identical panels fabricated from Inconel 625 were tested for comparison. AMI 100 (as brazing tape) and MBF-50 foil were used to fabricate two sets of NDS 300 test panels.

Test results for the panels are given in Tables 2-33 and 2-34. Photographs and sections prepared metallographically are presently in Figures 2-28 through 2-30. Most of the NDS 300 panel failures were due to braze joint separation, which was primarily the result of preparation of the NDS 300 plates, where distortion was created by uneven heating conditions during nitriding. The IN625 panel appeared well brazed; failure was in the braze joints due to very high peeling stresses developed when the IN625 plates pillowed out of plane between the fins. This mode of failure was not observed with the NDS 300. In the NDS 300-IN625 comparison test with MBF-50 braze foil at 1650°F and 400 psi internal pressure, the NDS 300 panel exhibited more than three times the life, and failure was apparently due to a brazing defect. This is not to suggest problems with either the strength or application of the filler metal, rather, the problem was with fit up and assembly of the NDS 300 samples. The distortion problems with NDS 300 material as produced in the laboratory retort have been discussed previously with respect to fin material, which was successfully stretch-flattened prior to forming. The size requirements of the plates for the single-sandwich heat exchangers precluded flattening operations other than hand bending, i.e., panel assembly was more susceptible to operator error than was assembly with IN625 parts. These processing problems are considered minor, but are a major factor for selection

TABLE 2-33

TEST RESULTS ON SINGLE-SANDWICH HEAT EXCHANGER PANELS  
BRAZED WITH MBF-50 (METGLAS) FOIL

Material*	Temperature, OF	Pressure,** psig	Life (Hours at Temp. and Press.)	Remarks
IN625	1650	400	191.4	Extreme distortion due to creep caused braze joint peel
NDS 300	RT	1995	Burst " } " }	These three panels were pressurized slowly until burst occurred
	1647	1335		
	1722	1670		
NDS 300	1650	400	653.4	Failed at undercut (cold) braze joint in center of panel
NDS 300	1725	400	145.6	Both panels failed in defects in plate-to-header bar braze joints
	1725	250	104.9	
NDS 300	1800	200	292.7	Both of these failures were in the brazing alloy at fin-to-plate joints near a header bar
	1800	200	63.5	

(the above test was pressurized with air instead of argon)

\*Inconel 625 plates and fins were from 0.010-in.-thick material. NDS 300 plate material was 0.010-in.-thick, processed by the treatment A. Fin material was also 0.010-in.-thick, processed per treatment B. Both IN625 and NDS 300 fins were plain rectangular, 10 per inch, 0.131-in. high.

\*\*Burst tests were conducted with water or nitrogen and creep tests with argon, except for the one air test noted last in the list above.

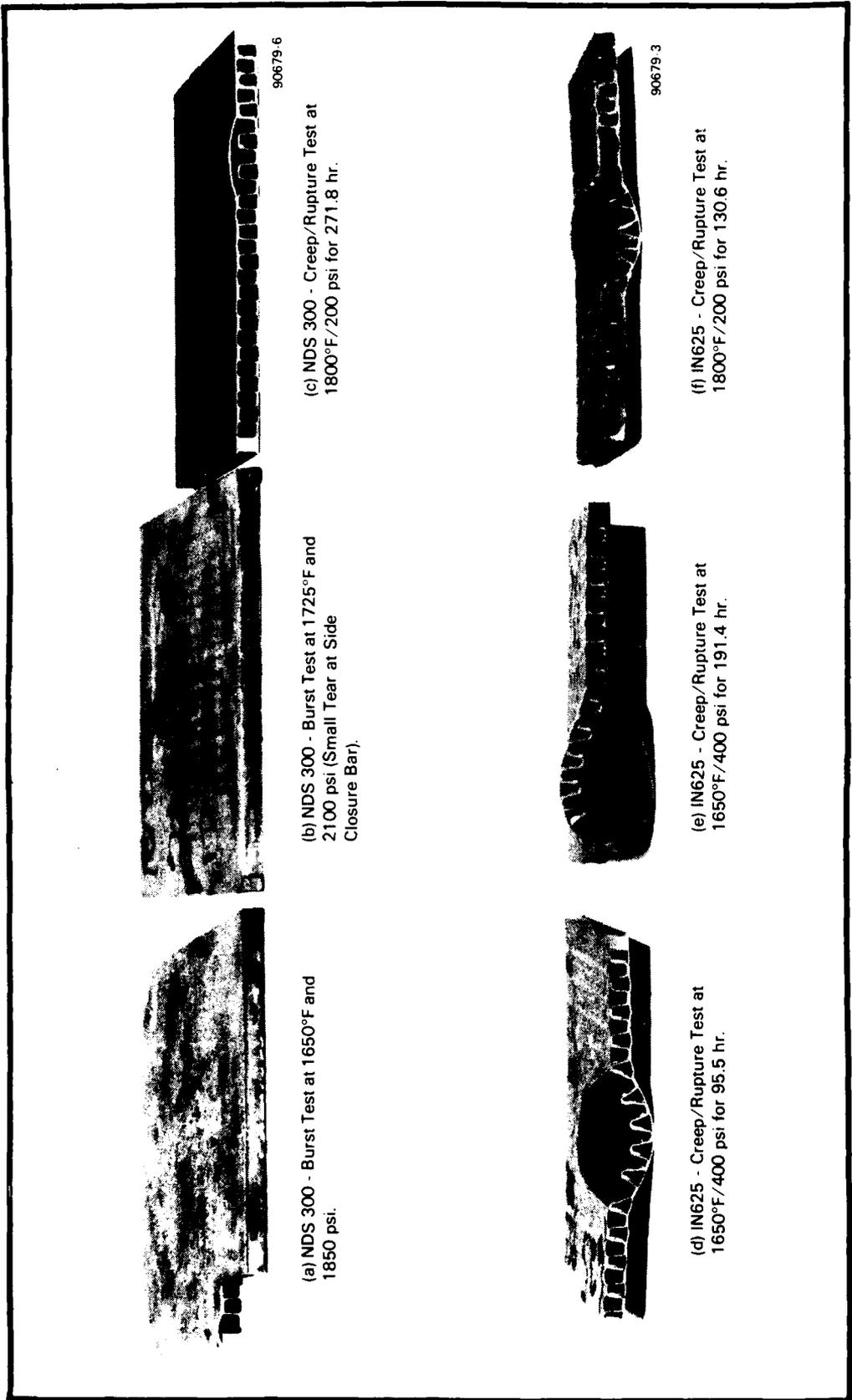
TABLE 2-34

TEST RESULTS ON SINGLE-SANDWICH HEAT EXCHANGER  
PANELS BRAZED WITH AMI 100 TAPE

Material*	Temperature, of	Pressure,** psig	Life (Hours at Temp. and Press.)	Remarks
IN625	1650	2150	>0.25	Burst test. Pillowed extensively
IN625	1650	400	95.5	
IN625	1800	200	130.6	
	1800	200	825.2	Air.
NDS 300	RT	640	Burst	Poor braze (large area)
	RT	1295	Burst	Poor braze (medium area)
	RT	2250	Burst	Small unbrazed area
	1650	1060	Burst	
	1650	1850	Burst	
	1651	1760	Burst	
	1725	2100	Burst	
NDS 300	1650	400	823.1	
NDS 300	1725	200	973.1	Terminated without failure
NDS 300	1800	200	2.7	Air. Poor braze
	1800	200	271.8	Small unbrazed area
	1800	200	834.5	Air.

\*See Table 2-33 footnote.

\*\*Burst tests were with water or nitrogen, creep tests were with argon except where indicated.



(a) NDS 300 - Burst Test at 1650°F and 1850 psi.

(b) NDS 300 - Burst Test at 1725°F and 2100 psi (Small Tear at Side Closure Bar).

(c) NDS 300 - Creep/Rupture Test at 1800°F/200 psi for 271.8 hr.

(d) IN625 - Creep/Rupture Test at 1650°F/400 psi for 95.5 hr.

(e) IN625 - Creep/Rupture Test at 1650°F/400 psi for 191.4 hr.

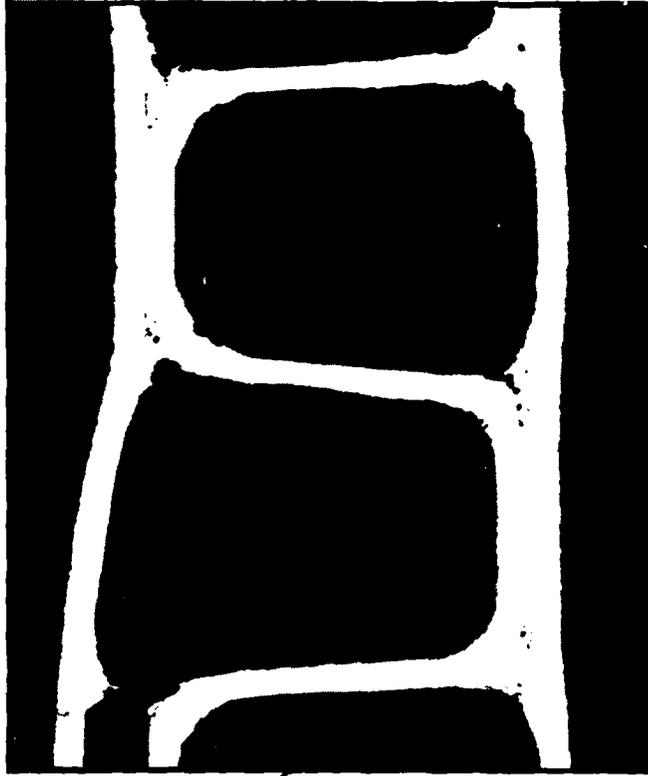
(f) IN625 - Creep/Rupture Test at 1800°F/200 psi for 130.6 hr.

F-45992

Figure 2-28. Sections of NDS 300 and IN625 Test Panels (All Were Brazed with AMI 100 Filler Metal Except (e) Which Was Brazed with MBF-50 Foil)



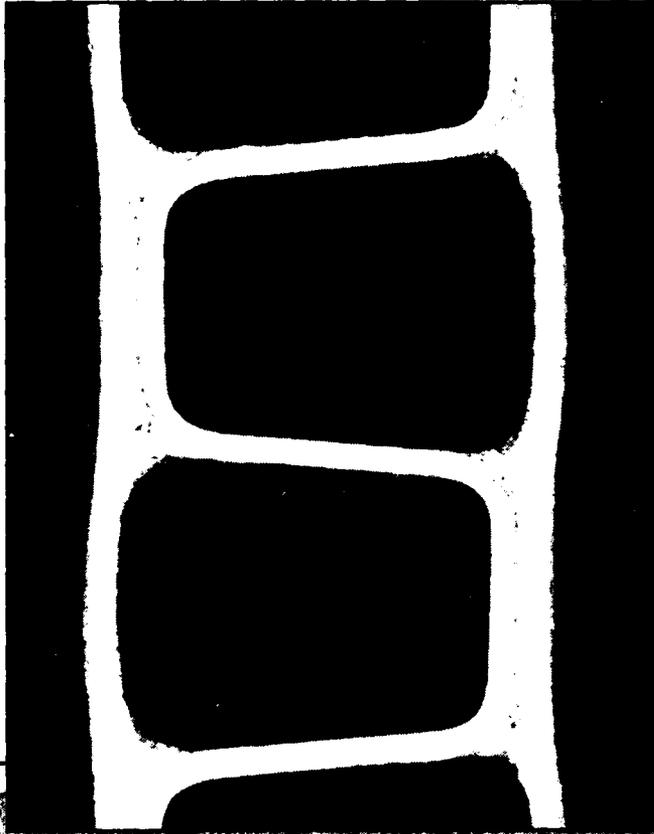
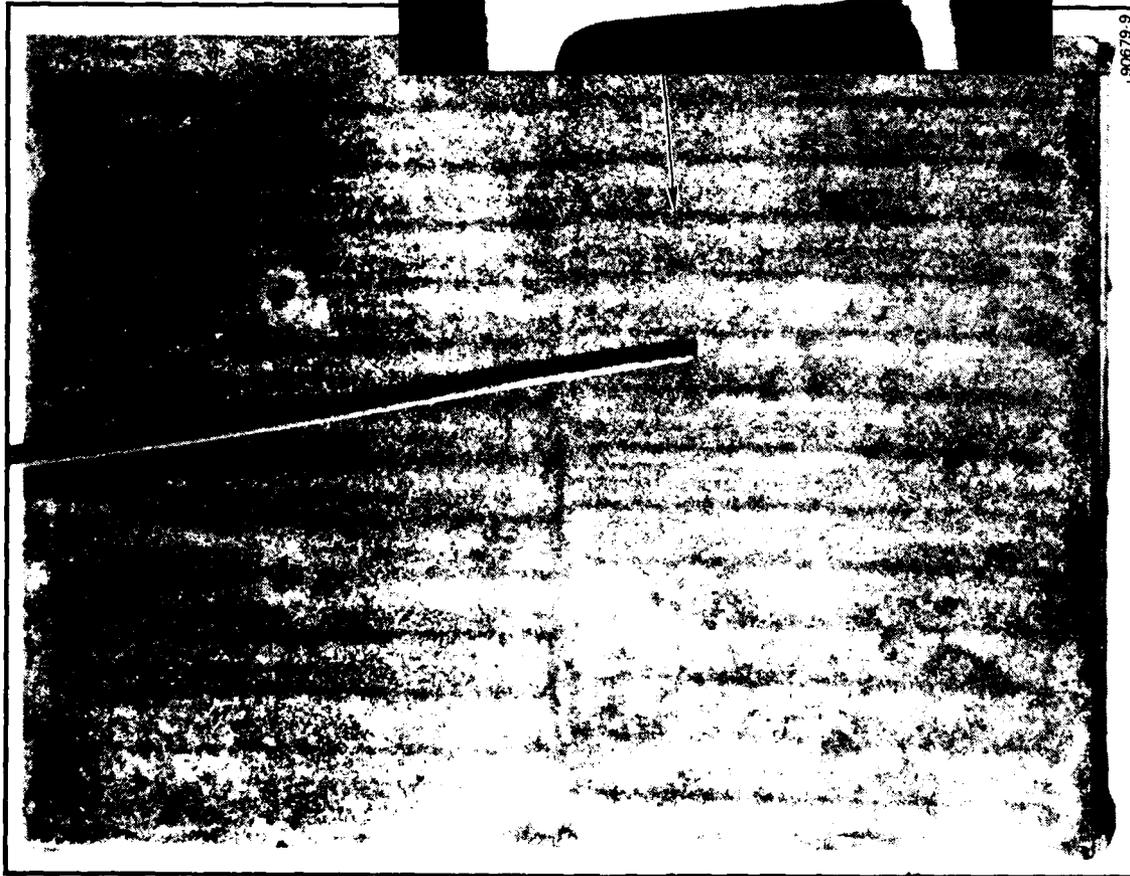
90679.11



14X

F-46027

Figure 2-29. NDS 300 Panel Brazed with AMI 100 Filler Metal and Tested for 823.1 Hours at 16500F with 400 psig Argon



14X

F-46025

.90679.9

Figure 2-30. NDS 300 Panel Brazed with AMI 100 Metal and Tested for 973.1 Hours at 17250F with 200 psig Argon (Did Not Fail--Photograph Shows Termocouple Still in Place)

of a continuous line process instead of batch treatment. Furthermore, if necessary, material can be roller flattened after nitriding at minimal cost.

It was originally planned to evaluate coatings on the heat exchanger panels. Without changing to a very elaborate (and costly) assembly, coating would have been limited to the outside surfaces of the panels, since there was no commercially available technique for coating application through the small pressurization tubes. While coating the outside panel surfaces would provide information on coating applied over a brazed NDS material surface (all panels were braze coated externally to simulate a heat exchanger plate and to provide oxidation resistance for testing), no data would be acquired on the effect of the coatings on brazed plate-fin joints.

The answer to this dilemma was the open stud-fin sample described in Task 2, Brazing Development, and shown schematically in Figure 2-18. A set of stud-fin samples were prepared using either the AMI 100 or MBF-50 filler metals, and were tested both bare and after chromizing by the original vendor. A few tests also were made using AMI 100 as a coating (applied as powder in a resin binder) which was fused simultaneously with stud-fin brazing. Samples prepared using Inconel 625 fins (same geometry as NDS 300) were tested for comparison both bare and coated with AMI 100 power.

Tensile and rupture test results are presented in Tables 2-35 and 2-36, respectively. Tensile results with the two filler metals show that tensile strength of chromized NDS 300\* is apparently not reduced from the bare material. Failure stresses were lower than would be expected from simple ultimate tensile strength comparison, however, because of the complexity of stress distribution in such an assembly. This fractional strength effect is observed for virtually all materials and must be accounted for in design. The total cross-sectional area of the supporting fin was 0.076 sq in. Hence, maximum fin stresses would have been of the order of 50 to 70 ksi at room temperature and 13 to 18 ksi at 1650°F. Since the fin geometry in a heat exchanger panel gives 0.10 sq in. of cross-section per square inch of panel, burst test numbers in Tables 2-33 and 2-34 can most simply be multiplied by 10 to give comparison stress values. The comparison at 1650°F is very good; at ambient, higher stresses achieved with stud fins undoubtedly are related to the more rigid structure of the stud versus a 10-mil plate, since the latter can bow out between fins and create high peel stresses on the braze.

Rupture results on stud-fin specimens (Table 2-36 and Figure 2-31) were as mixed as the panel test results, but again demonstrated that NDS 300 is markedly superior to IN625 and that the filler metals chosen can withstand typical operating stresses over the design life of a military gas turbine engine. Test results on chromized samples were disappointing, but the coating may not have been representative of simple creep test specimens prepared earlier as shown in Figure 2-21c for the oxidation test coupon prepared with creep test specimens reported in Table 2-26. The AMI 100 braze coating did appear to provide significant protection, and would be a single operation combined with recuperator brazing. This is evident in Figure 2-32 which presents micrographs of a representative group of stud-fin rupture specimens.

\*Chromium level had been increased to 33 percent at the surface and 21 percent at depth of 1.5 mils. The base NDS 300 alloy has a chromium content of 18 percent.

TABLE 2-35

## TENSILE TEST RESULTS ON STUD-FIN SPECIMENS\*

Fin Material	Condition	Temp., OF	Breaking Load, lb	
			MBF-50 Foil	AMI 100 Tape
NDS 300	Bare	RT	3650	3440
	Chromized	RT	6200	4670
	Bare	1650	-	1075
	Chromized	1650	1380	972
IN625	Bare	RT	4820	5000

\*Fins were 10 per inch, 0.131-in.-high, 0.010-in.-thick material, brazed between 347 stainless steel studs as shown in Figure 2-18. The chromizing treatment was applied after final assembly.

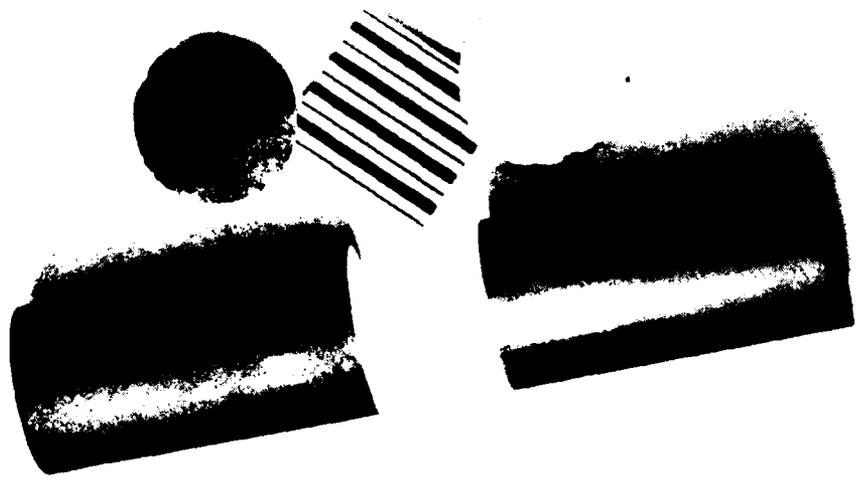
TABLE 2-36

## CREEP/RUPTURE TEST RESULTS ON STUD-FIN SPECIMENS

Fin Material	Brazing Alloy	Coating	Temp., °F	Fin Stress, ksi	Termination at:		Ruptured at: hr	Remarks
					Time, hr	Fin Height,* in.		
NDS 300	AMI 100	None	1650	2	746	0.134	--	--
	AMI 100	None	1650	4	--	--	640	--
	MBF-50	None	1650	4	--	--	348	Fin not through-nitrided
	AMI 100	None	1800	2	138	NM	142	Step-loaded to 4.5 ksi
	AMI 100	None	1800	2	1001	NM	--	Broke on cooldown
	AMI 100	AMI 100	1650	2	266	NM	--	Broke on cooldown
	AMI 100	AMI 100	1800	2	1001	0.157	--	Brazing alloy separation
	AMI 100	Chromized	1650	2	1191	0.135	--	--
	AMI 100	Chromized	1650	4	--	--	442	--
	AMI 100	Chromized	1725	2	--	--	422	--
	AMI 100	Chromized	1800	2	--	--	168	--
	MBF-50	Chromized	1650	2	1195	0.133	--	--
	MBF-50	Chromized	1650	4	--	--	80	--
	MBF-50	Chromized	1725	2	--	--	1134	--
	MBF-50	Chromized	1800	2	--	--	481	--
IN625	AMI 100	None	1650	4	1497	0.166	--	Badly oxidized
	MBF-50	None	1650	4	1010	0.156	--	Badly oxidized
	AMI 100	None	1800	2	--	--	319	Badly oxidized
	AMI 100	AMI 100	1650	2	745	0.140	--	--
	AMI 100	AMI 100	1800	2	580	0.171	--	--

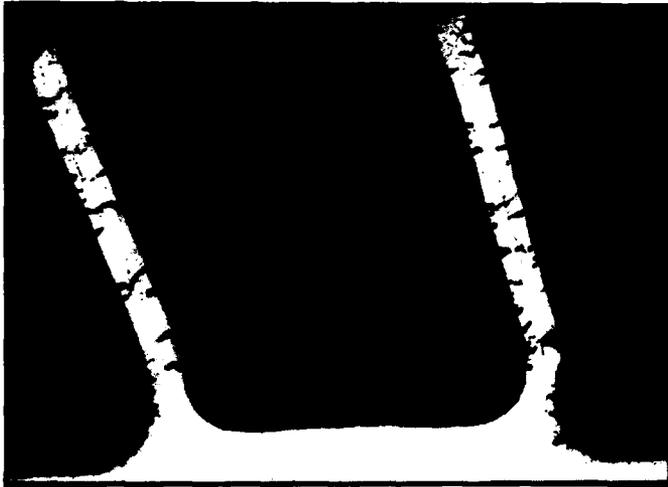
\*Starting fins were nominally 0.131-in. high, 10 to the inch.

NM = not measured



Sub 75-7

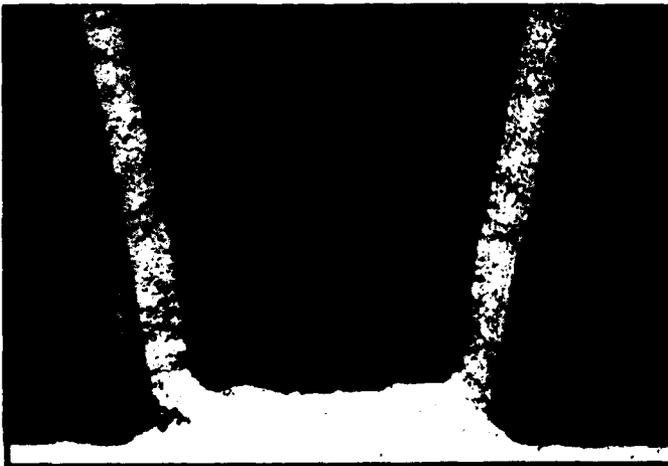
Figure 2-31. Stud-Fin Specimens: Left--Studs, Fin, and MBF-50 Foil for Assembly; Center--Unfailed, Chromized 16500F/2 ksi Creep Test Specimens that Were Terminated after 1191 and 1195 Hr; Right--Chromized 17250F/2 ksi Creep Test Specimen that Failed at 1134 Hr (See Table 2-36)



a. IN625 BARE;  
FAILED 319 HR



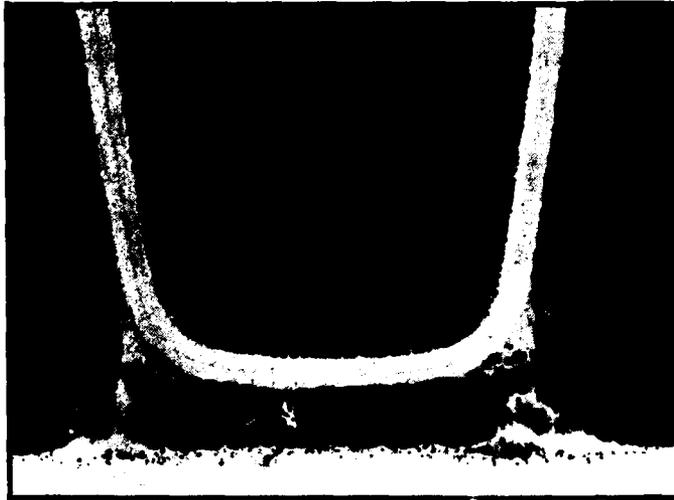
b. NDS 300 BARE;  
FAILED 1001 HR



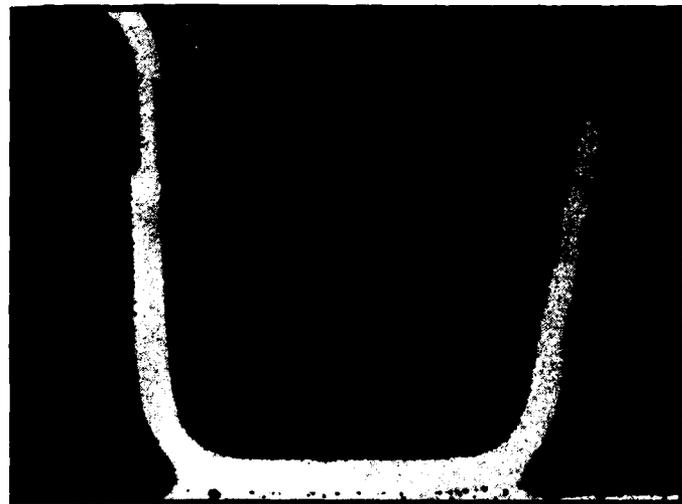
c. IN625 COATED WITH  
AMI 100 BRAZE;  
TERMINATED 580 HR

F-46019

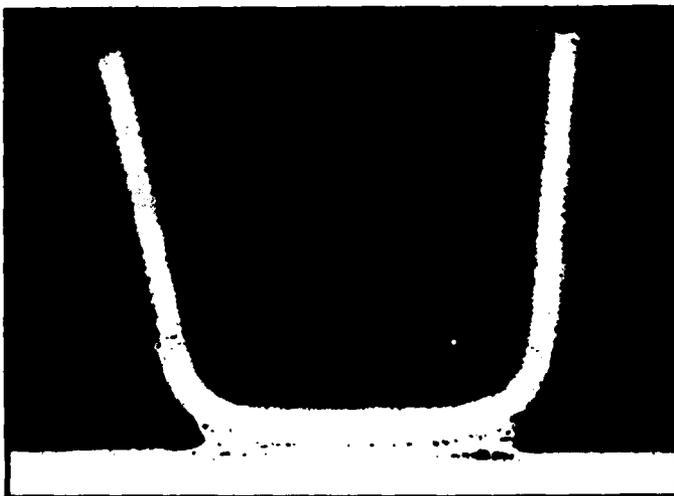
Figure 2-32. Cross Sections of Stud-Fin Specimens, Comparing IN625 to NDS 300 after Stress Rupture Testing at 1800°F--Item (f) was at 1725°F--Under a Fin Stress of 2 ksi for the Times Shown; (Magnification, 25X)



d. NDS 300 COATED WITH  
AMI 100 BRAZE;  
TERMINATED AT 1001 HR



e. NDS 300 CHROMIZED;  
FAILED 168 HR



f. NDS 300 CHROMIZED;  
FAILED 1134 HR  
(1725°F TEST)

F-46020

Figure 2-32 (Continued)

Both bare NDS 300 and IN625 were prone to intergranular oxidation where extensive creep damage occurred (Figure 2-32 a and b). This would be a particular problem with IN625 since creep strength is so low, and, as described in Task 1a above, the material becomes very brittle. The primary problem with NDS 300 was with grain boundary attack of bare fin material which was subjected to a bending moment due to the configuration of the assembly. This has to be expected in practice, and gives further call for a coated NDS 300 fin or an alternate material as discussed in Task 3.

As discussed previously, AMI 100 appears viable as a protective coating for NDS 300. However, because of a high creep rate, this coating technique does not appear to work for IN625 (see Figure 2-32c). Although surface oxidation is visible in Figure 2-32d, AMI 100-coated NDS 300, the fin is essentially intact, and, instead, the braze alloy has crept close to separation. The brazing alloy is still very intact after 1134 hr at 1725°F, and as such the 100 filler metal appears to be an acceptable choice where service at 1800°F under full recuperator pressure is limited to a few hundred hours (compare Figure 2-32 e and f).

### 3. GENERAL DISCUSSION

The NDS 300 material has demonstrated a marked strength advantage, both in tensile properties and in creep, over existing commercial sheet alloys such as Inconels 625 and 617. Projections show the NDS 300 material to be nominally 30 percent less costly than either of these Inconel alloys. The treatment process has been shown to be repeatable; initial production should be readily achievable by treatment of coils on a continuous line. The NDS 300 material has also been found to have the fabrication characteristics necessary for application to a gas turbine recuperator. Some adjustments in nitride treatment appear possible if needed for improved formability.

The NDS 200 composition, although requiring considerably more development, is potentially a three-fold-faster nitriding material than NDS 300. Also, NDS 200 contains a significantly lower level of strategic materials, especially chromium. It appears that better possibilities to duplicate the creep strength of NDS 300 can be obtained by adjusting the titanium content and the prenitride processing. In the long run, a composition with nickel and manganese contents between NDS 200 and 300 levels and with a lower chromium content than the 18 percent in NDS 300 would seem to provide the best combination of mechanical strength and speed of production.

The selected brazing alloy, which is based on the proven J8100 filler metal, appears adequate, but it could be stronger. Future programs should look more closely at higher melting point filler metals, especially those that have small additions of molybdenum or tungsten for high temperature strength. However, care will have to be exercised so that the NDS metal strength is not comprised by too high a brazing temperature.

Coatings for high temperature environmental resistance should be avoided if possible because of added cost. Aluminide-coated NDS alloys have been shown to lose significant strength, and these coatings should not be applied over bare NDS material where high strength is required. For low-stress exposed areas such as heat exchanger fins, the use of alternate alloys such as Incoloy 800H or Inconel 625 is recommended. For extreme environmental problems, an aluminide coating may possibly be applied over a braze-coated NDS material; however, further research is needed to determine the compatibility of such a coating with a nickel brazing filler metal. As an alternative, the use of a brazing filler metal as coating may be of value as demonstrated in this program for both creep and stud-fin test specimens with AMI 100 filler. If this filler metal proves wanting, a higher chromium-content (30 percent) version, AMI 100, could solve the problem and has been shown to be compatible with NDS 300 material.

The pressures (stresses) employed for panel and stud-fin specimen testing were selected to accelerate the test (shorten life) compared to an actual recuperator on an engine in the field. Although somewhat confounded by the difficulty in preparing NDS 300 test specimens, test lives close to the potential recuperator design life of several thousand operating hours were realized, at up to three times the life of comparison samples fabricated from Inconel 625. Even then, failures generally were related to the brazing alloy. In an operating unit, thermal stresses will prevail, however, and this is where the NDS materials should excel. With their high yield and creep strengths, NDS alloys should remain rigid during thermal transients, thus minimizing low-cycle fatigue-related failures.

#### 4. SUMMARY

The objectives of this program were to demonstrate the feasibility of: (1) applying the nitride-dispersion-strengthened (NDS) metal to a gas turbine recuperator by establishing NDS material production requirements, (2) reducing strategic alloy content (Co, Cr, Ni, Mo, W) in the material, (3) fabricating a plate-fin recuperator by furnace brazing, and (4) coating hot-end areas for environmental resistance.

The results of the two-year program have shown that significant reductions in processing time from the original projections based on an established longer-time minimum treatment process can be achieved for NDS 300, an 18Cr-12Ni-2Ti base material, without a major loss in strength. In addition, NDS 200, an alloy lower in chromium (12 percent) and nickel (8 percent), but higher in manganese, was developed. NDS 200 had slightly lower creep strength than NDS 300, but strategic element content was also markedly lower, with chromium content reduced to the 12-percent level. Nitriding processing time of the new NDS 200 alloy was but one-third of the minimum time found for NDS 300, making it an ideal candidate for recuperator plate material to be processed on a continuous line basis. The NDS 300 material was selected as the "go-forward" material, with NDS 200 in reserve as second generation product improvement. Extensive property testing on NDS 300 showed that the material was several times stronger in creep strength than Inconel alloys 617 and 625, with a projected lower cost.

Several brazing alloys were evaluated for use in fabrication of a recuperator from NDS alloys. Filler metals AMI 100 (based on the proven J8100 alloy) and MBF-50 were selected as the two strongest candidate alloys based on strength and on cyclic oxidation tests, as well as general brazing characteristics. The 100 alloy was used in both powder and tape form, while the MBF-50 was available as a foil.

Coating studies showed that NDS 300 could be chromized without loss of strength, but not aluminized, whereas strength loss with either coating was observed with NDS 200. Coating with filler alloy 100 also proved successful on NDS 300. Use of a standard alloy such as Incoloy 800H for exposed surfaces, such as fins, showed promise, either aluminized or nitride strengthened.

Fabrication studies on NDS 200 and NDS 300 showed that ductility levels similar to currently used recuperator plate material could be achieved by control of nitride processing within a fairly wide range. Fin-forming and plate-forming experiments did not reveal any pronounced cracking tendencies, nor were there any structural changes apparent as a result of subsequent heating through a furnace brazing cycle.

Plate-fin single-sandwich and stud-fin specimens were prepared from both NDS 300 and IN625 for direct comparison in burst and creep tests at ambient to 1800°F. While results were somewhat clouded by fitup problems with NDS 300 because of the extreme high temperature stiffness of the alloy, the overall picture was one of confirmation of the creep test results, which showed marked superiority of the NDS 300 material. Strength of the 100 filler metal for application to recuperator fabrication was also confirmed for times on the order of 1000 hr at 1650° to 1800°F.

## 5. CONCLUSIONS AND RECOMMENDATIONS

As a result of the work on this program, the following conclusions may be drawn:

- (1) NDS alloys have very high levels of mechanical strength, with creep strength several times that of Inconel alloys 625 and 617 and are projected to be less costly.
- (2) NDS alloys appear capable of continuous line production and have the fabrication characteristics required of recuperator materials.
- (3) By reducing alloy content (especially Cr and Ni) and adding manganese, an NDS 200 alloy was created. NDS 200 is slightly lower in creep strength than NDS 300, but allows nitride processing at a rate that is several times faster.
- (4) For 1800°F service, NDS 300 recuperator fins will require coating; however, strength levels are considerably higher than necessary, thereby allowing room for the use of an alternate, more environmentally resistant material.
- (5) The chosen braze filler metal was AMI 100, which is based on J8100 alloy (19Cr-10Si-Ni), with MBF-50 (a boron-modified foil version) as backup. This alloy also proved to be an excellent coating for high temperature oxidation resistance of NDS 300. The 100 brazing alloy proved adequate in high temperature creep/rupture tests on small plate-fin heat exchanger assemblies.
- (6) Diffusion coating technology (aluminizing and chromizing) for thin, iron-based alloys is not well developed. Alternate approaches, such as selective hot-end use of a more oxidation- and hot-corrosion-resistant alternate material or version of NDS 300, may be necessary.

Overall, the program goals were met and continuation of work is recommended as follows.

- (1) In the first stage, a prototype treatment facility should be constructed so that NDS 300 material can be produced and evaluated for uniformity of mechanical strength, including tensile, creep, and fatigue properties.
- (2) Once this facility has been established, sufficient material should be produced to allow fabrication and testing of a prototype recuperator.

(3) Concurrently, work should be conducted on second generation materials, a process that should include:

- (a) Development of NDS 200
- (b) Selection and application of improved brazing alloys
- (c) Improved application of diffusion coatings

## 6. REFERENCES

1. Kindlimann, L.E., and G.S. Ansell, "Kinetics of the Internal Nitridation of Austenitic Fe-Cr-Ni-Ti Alloys," Metallurgical Transactions, Vol. 1, January 1970, pp. 163-170.
2. Rapp, R.A., "Kinetics, Microstructures, and Mechanism of Internal Oxidation - Its Effect and Prevention in High Temperature Alloy Oxidation," Corrosion, Vol. 21, December 1965, pp. 382-401.
3. Kindlimann, L.E., and G.S. Ansell, "Internally Nitrided Steel Powder and Method of Making," U.S. Patent 3,650,729, March 21, 1972.
4. Kindlimann, Lynn E., and Alexander B. Greene, "Method of Improving Nitride-Strengthened Stainless Steel Properties," U.S. Patent 3,615,904, October 26, 1971.
5. Floreen, S., "Chromium Substitution in Stainless Steels," Work Performed by INCO Research and Development Center, Inc., for U.S. Bureau of Mines under Contract No. J0295070, Report BuMines OFR 110-81, November 1980.

APPENDIX A  
NITRIDING RUN DATA

APPENDIX A  
NITRIDING RUN DATA

RUN NO.	MATERIALS	RETORT*	HEATUP GAS	CONDITIONS				REMARKS
				NITRIDING		DENITRIDING		
				TIME MIN.	TEMP °F	TIME MIN.	TEMP °F	
105	18	R	H2	15	1900	120	2000	
106	18	R	H2	10	2000	120	2000	
107	18	R	H2	25	1950	120	2000	
108	18	R	H2	15	1950	120	2000	
110	18	R	H2	30	1900	180	2025	
118	18	R	NH3	25	1900	180	2025	
120	18	R	NH3	30	1900	180	2010	
124	18	R	NH3	30	1900	120	2025	
127	18, 7, 8	T	NH3	12.5	1900	180	2025	
132	18, 7, 8	T	NH3	12.5	1900	180	2025	
134	18	R	NH3	15	2000	180	2025	
135	18	R	NH3	30	1900	180	2025	
136	7, 8	R	NH3	20	1900	180	2025	
137	18	R	NH3	30	1900	180	2025	
138	18	R	NH3	30	1900	180	2025	
139	18	R	NH3	30	1900	180	2025	
140	7, 8	R	NH3	7	2000	180	2025	
141	7, 8	R	NH3	10	1900	180	2025	
142	7, 8	R	NH3	7	2000	180	2025	
143	18	R	NH3	30	1900	180	2025	
144	18	T	NH3	10	2000	180	2025	
145	7	T	NH3	5	2000	180	2025	
146	8	T	NH3	5	2000	180	2025	
147	7	T	NH3	4	2000	180	2025	
148	18	T	NH3	10	2050	180	2025	
149	18	T	NH3	15	1950	180	2025	
150	7	T	NH3	5	2000	180	2025	
151	7, 8	R	NH3	5	2000	180	2025	
152	8	T	NH3	6	2000	180	2025	
153	8	T	NH3	5	2000	180	2025	
154	7	T	NH3	8	1900	120	2000	
155	7	T	NH3	10	1900	180	2000	
156	7	T	NH3	6	1900	180	2000	
157	7	T	NH3	4	1900	180	2000	
158	8	T	NH3	4	1900	180	2000	
159	8	T	NH3	6	1900	180	2000	
160	7	R	NH3	4	1885	180	2000	
161	7, 8	R	NH3	10	1910	180	2000	
162	18	R	NH3	30	1870	180	2000	
163	7	R	NH3	5	1950	180	2000	
164	7	R	H2	5	1955	180	2000	
165	7	R	H2	6.5	1930	180	2000	
166	7	R	H2	6.5	1940	180	2000	

\* R = FURNACE RETORT; T = TUBE RETORT

RUN NO.	MATERIALS	RETORT*	CONDITIONS					REMARKS
			HEATUP GAS	NITRIDING		DENITRIDING		
				TIME MIN.	TEMP °F	TIME MIN.	TEMP °F	
167	8	R	H2	7.5	1925	180	2000	
168	18	R	H2	15	1900	180	2000	
169	18	R	H2	15	1925	180	2000	
170	7A, 7B, 7C	R	H2	6.5	1945	180	2025	
171	7A, 7B, 7C	R	H2	6.5	1930	180	2025	
172	7A, 7B, 7C	R	H2	6.5	1940	180	2025	
173	7A, 7B, 7C	R	H2	6.5	1920	180	2025	
174	7, 7B	T	NH3	5	1900	120	2025	
175	18	T	NH3	15	1900	120	2025	
176	18	R	H2	15	1925	180	2025	
177	18	R	H2	15	1900	150	2025	
178	7, 7C	T	NH3	4	1925	180	2025	
179	18	T	NH3	15	1900	180	2025	
180	18	T	NH3	15	1900	180	2025	
181-186	18	R	H2	15	1920	180	2025	
187	18	T	NH3	10	1975	180	2025	
188	7, 7C	T	NH3	5	1900	180	2025	
189	7C	R	H2	6.5	1920	180	2025	
190-207	18	R	H2	15	1920	180	2025	
208	18	R	NH3	15	1920	180	2025	
209	18	R	NH3	15	1920	180	2025	
210	7C	R	H2	5	1925	180	2025	
211	7C	R	H2	5	1925	180	2025	
212	7, 7C	R	H2	6	1930	180	2025	
213	7C	R	H2	6	1935	180	2025	
214	7C	R	H2	6	1935	180	2025	
215	7C	R	H2	6.5	1940	180	2025	
216	18	R	H2	15	1935	180	2025	
217	18	R	NH3	15	1925	180	2025	
218	18	R	H2	10	1990	180	2025	
219	7	R	H2	5	1925	180	2025	
220	7	R	H2	6	1900	180	2025	
221	7C	R	H2	6	1930	180	2025	
222	7C	R	H2	6.5	1925	180	2025	
223	7C	R	H2	6.5	1925	180	2025	PREANNEALED 1900F/3 MIN.
224	7C	R	H2	6.5	1925	180	2025	PREANNEALED 1900F/5 MIN.
225	7C	R	H2	6.5	1930	180	2025	PREANNEALED 1900F/10 MIN.
226	7C	R	H2	6.5	1930	180	2025	
227	7C	R	H2	6.5	1920	180	2025	PREANNEALED 2000F/5 MIN.
228	7C	R	H2	6	1900	180	2025	PREANNEALED 1900F/10 MIN.
229	7C	R	H2	6	1900	180	2025	PREANNEALED 1900F/10 MIN.
230	800H	R	NH3	30	1970	180	2025	
231	18	R	H2	15	1900	180	2025	

\* R = FURNACE RETORT; T = TUBE RETORT

RUN NO.	MATERIALS	RETORT*	CONDITIONS					REMARKS
			HEATUP GAS	NITRIDING		DENITRIDING		
				TIME MIN.	TEMP °F	TIME MIN.	TEMP °F	
232	18	R	H2	15	1900	180	2025	
233	18	R	H2	15	1900	SEE	NOTE	
234	18	R	H2	15	1900	SEE	NOTE	
235	7C	R	H2	4	1880	60	2050	
236	18	R	H2	15	1900	120	2025	OLD VERSUS NEW MATERIAL.
237	18	R	H2	15	1900	120	2025	OLD VERSUS NEW MATERIAL.
238	18	R	H2	15	1900	20	2100	
239	18	R	H2	15	1900	20	2100	
240	18	R	H2	15	1900	20	2100	
241	7C	R	H2	6	1900	60	2050	PREANNEALED 1900F/3 MIN.
243	18	R	H2	15	1900	20	2100	
244	18	R	H2	15	1900	20	2100	
245	18	R	NH3	15	1900	20	2100	
246	18	R	NH3	15	1900	20	2100	
247	18	R	NH3	10	1985	20	2100	
248-261	18	R	NH3	16.5	1900	20	2100	A MATERIAL
262-265	18	R	H2	15	1900	20	2100	B MATERIAL
266-270	18	R	NH3	10	1975	20	2100	C MATERIAL
271-272	7C	R	H2	6	1900	60	2050	PREANNEALED 1900F/3 MIN.
273-281	18	R	NH3	16.5	1900	20	2100	A MATERIAL
282-283	18	R	H2	5	1900	20	2100	B MATERIAL
284	18	R	NH3	10	1950	20	2100	
285	18	R	H2	15	1900	20	2100	B MATERIAL
286	18	R	H2	17.5	1900	20	2100	PREANNEALED 1900F/3 MIN.
287	18	R	H2	20	1900	20	2100	
288	18	R	H2	20	1900	20	2100	

\* R = FURNACE RETORT; T = TUBE RETORT

NOTES FOR RUNS 233 AND 234 DENITRIDING (SEE APPENDIX B)

A = 2025 F/60 MIN  
 B = 2025 F/120 MIN  
 C = 2100 F/60 MIN

APPENDIX B  
CREEP/RUPTURE TEST DATA FOR ALL  
MATERIALS INDEXED BY TEST NUMBER

**APPENDIX B  
CREEP/RUPTURE TEST DATA FOR ALL MATERIALS INDEXED BY TEST NUMBER**

TEST NO.	MATERIAL TYPE	NITRIDE RUN NO.	MATERIAL CONDITION	COATING	TEMP F	STRESS KSI	1% HRS	2% HRS	TERM %	FAIL HRS	ELONG %	REMARKS
566	18-12-2	105			1500	20	603			603	2.7	WENT THRU 1% AT FAILURE. TERMINATED AT 43 HR. EQUIPMENT PROBLEM.
567	18-12-2	105			1650	14						
568	18-12-2	106			1500	20	189		1.0			TERMINATED. PAD FAILURE FAILED DURING COOLDOWN.
569	18-12-2	106			1650	14	>160					
571	18-12-2	108			1650	14	62					
572	18-12-2	124			1650	20	1.5			28.6	3.45	
573	18-12-2	124			1650	15	228			257	2.27	
574	18-12-2	134			1500	20	171			937	2.27	
575	18-12-2	134			1650	15	22			256	3.18	
576	12-8-2	136		ANI 300	1500	20	0.16			13	4.5	
577	7-10-2	136		ANI 300	1500	20				2.6	4.5	
578	18-12-2	135			1500	20	304			725	2.27	
580	12-8-2	142		ANI 300	1500	20	1			32.7	2.7	
581	7-10-2	142		ANI 300	1500	20	5			6.3	2.7	
582	18-12-2	137			1500	20	46			92.7	4.5	
583	18-12-2	138			1500	20			0.33			48 HRS. FURNACE FAILURE.
584	18-12-2	139			1500	20	346			1812	4.4	
585	18-12-2	143			1500	20	71		1.0			TERMINATED.
586	18-12-2	143			1500	20	187			744	3.81	
587	18-12-2	143		ANI 100B	1500	20	205			1443	5.5	
588	18-12-2	143		ANI 100B	1650	15	40			122	5.45	
589	12-8-2	145		ANI 300	1500	20	25.5			35	3.6	
590	18-12-2	143		ANI 4A/4B	1500	20			0.5			TERMINATED AT 283 HR.
591	18-12-2	143		ANI 4A/4B	1650	15			1.0			TERMINATED AT 281 HR.
592	12-8-2	147		ANI 300	1500	20	4.3			4.3	.9	
593	7-10-2	146		ANI 300	1500	20				26.2	3.1	
594	12-8-2	161		ANI 300	1500	20	15			33.5	3.4	
595	7-10-2	161		ANI 300	1500	20				1.8	1.8	
609	12-8-2	166		ANI 300	1500	20	98		1.5			TERMINATED AT 166 HR. TERMINATED.
610	12-8-2	166		ANI 300	1650	15	42		1.0			WENT THRU 1% AT FAILURE.
611	18-12-2	168			1500	20	273			273	1.36	
612	18-12-2	169			1500	20	31			41.8	3.6	
613	18-12-2	168			1500	20	130			131	2.26	
614	18-12-2	138		CHRONIZED	1500	20	240			240	2.18	WENT THRU 1% AT FAILURE.
615	18-12-2	139		CHRONIZED	1650	15	95		1.1			TERMINATED AT 116 HRS.
616	12-8-2	171	FINAL REDUCTION 42%	ANI 300	1650	20	16.5			20	3.27	
618	12-8-2	171	FINAL REDUCTION 50%	ANI 300	1500	25	19.5			30.4	3.2	
619	12-8-2	171	FINAL REDUCTION 50%	ANI 300	1650	20	9			22.4	3.8	
620	12-8-2	171	FINAL REDUCTION 57%	ANI 300	1500	20	128	360		574	4.54	
621	12-8-2	171	FINAL REDUCTION 57%	ANI 300	1650	20	7.5			27.3	3.4	
623	12-8-2	172	FINAL REDUCTION 42%	ANI 300	1650	15	63	144		294	2.27	
624	12-8-2	172	FINAL REDUCTION 50%	ANI 300	1650	20	31			74.2	1.8	
626	12-8-2	172	FINAL REDUCTION 57%	ANI 300	1650	20	76.5			96.4	4.5	

TEST NO.	MATERIAL TYPE	NITRIDE RUN NO.	MATERIAL CONDITION	COATING	TEMP F	STRESS KSI	1% HRS	2% HRS	TEMP %	FAIL HRS	ELONG %	REMARKS
629	12-8-2	173	FINAL REDUCTION 42%	AMI 300	1500	20	94			302	2.27	
630	12-8-2	173	FINAL REDUCTION 42%	AMI 300	1650	15	85			137	2.09	
631	12-8-2	173	FINAL REDUCTION 75%	AMI 300	1500	20	111	177		298	5.45	ORIGINAL MATERIAL (CONTROL).
632	12-8-2	173	FINAL REDUCTION 75%	AMI 300	1650	15	30			52	5.1	CONTROL.
633	12-8-2	173	FINAL REDUCTION 57%	AMI 300	1500	20	136			208	2.25	
634	12-8-2	173	FINAL REDUCTION 57%	AMI 300	1650	15	24	76		147	4.54	
638	18-12-2	177			1650	15	97.5			119	1.8	
639	18-12-2	177			1800	7.5				>18		TERMINATED, EQUIPMENT PROBLEMS.
643	18-12-2	176			1800	7.5	170			173	3.0	
644	12-8-2	189	FINAL REDUCTION 60%	AMI 100	1650	15	16			73.9	4.0	NEW HEAT.
645	12-8-2	189	FINAL REDUCTION 57%	AMI 100	1650	15	40.5			123	4.0	
646	12-8-2	212	FINAL REDUCTION 60%	AMI 300	1500	20	118			817	4.5	NEW HEAT.
647	12-8-2	212	FINAL REDUCTION 75%	AMI 300	1650	15	85			185	3.6	ORIGINAL MATERIAL.
648	18-12-2	177	FIN MATERIAL		1500	20	48			111	1.8	CENTER 1 NIL NOT NITRIDED.
649	18-12-2	177	FIN MATERIAL		1650	15	27			177	2.27	
650	18-12-2	208	GOOD AREA		1650	15	57.1			76.2	1.8	
651	18-12-2	209	GOOD AREA		1800	7.5	64.7			106	2.3	
652	18-12-2	209	SUSPECT AREA		1650	15	4.5			18.5	6.8	COLOR DIFFERENCE.
653	18-12-2	182	SUSPECT AREA		1650	15	92.5			95.3	1.4	COLOR DIFFERENCE.
654	18-12-2	183	SUSPECT AREA		1800	15				52		SHOULD HAVE BEEN 7.5 KSI.
655	18-12-2	177			1650	30				<1		ACCIDENTALLY OVERLOADED.
663	12-8-2	221	FINAL REDUCTION 75%	AMI 100	1800	7.5	7.5			80		RAN OVERTEMP TO 1840.
664	12-8-2	220	FINAL REDUCTION 75%	ALUMINIZED	1500	22	3			10.3	8.1	
666	18-12-2	208		ALUMINIZED	1500	20	23			34.5	3.6	
667	18-12-2	208		ALUMINIZED	1650	15	8			12.8	5.45	
670	18-12-2	208		ALUMINIZED	1800	2	11.5			325	50	
676	12-8-2	220	FINAL REDUCTION 75%	ALUMINIZED	1650	15	1.5			5.1	11.8	
677	18-12-2	208		ALUMINIZED	1800	5	6			13		
678	12-8-2	222	FINAL REDUCTION 60%	AMI 100	1650	15	9.5			80.6	5.45	NEW HEAT.
679	12-8-2	223	NEW HT, PREANNEALED	AMI 100	1650	15	9.5	41		108	6.35	1900/3 MIN. PREANNEAL
680	12-8-2	225	NEW HT, PREANNEALED	AMI 100	1650	15	32		1.8			1900/10 MIN. PREANNEAL (TERM. AT 74 HR.)
681	12-8-2	225	NEW HT, PREANNEALED	AMI 100	1800	7.5	10.5					1900/10 MIN. PREANNEAL (TERM AT 19 HR.)
683	12-8-2	227	NEW HT, PREANNEALED	AMI 100	1800	7.5	10					2000/5 MIN. PREANNEAL (TERM AT 17 HR.)
694	INCD 800H	230			1800	4	38			40	2.0	NBS TREATMENT APPLIED.
702	18-12-2	177	FIN MATERIAL	AMI 100	1800	2			1.8			TERMINATED AT 1607 HR.
703	18-12-2	177	FIN MATERIAL	AMI 100	1800	5	24	144		317	3.6	
704	18-12-2	232			1650	15	102			160	2.3	PLATE RUN
714	18-12-2	233	1 HR DENITRIDE		1650	15	120		1.2			TERMINATED AT 141 HR
715	18-12-2	234	2 HR DENITRIDE		1650	15	94		1.0			TERMINATED.
716	18-12-2	233	2100F DENITRIDE		1650	15	92		1.0			TERMINATED. 1 HR DENITRIDE.
718	18-12-2	110		CHROMIZED	1650	15	159			159	1.8	WENT THRU 1% AT FAILURE.
719	12-8-2	229	FINAL REDUCTION 60%	CHROMIZED	1650	15				3.7	1.0	PREANNEALED 1900/10 MIN.
720	18-12-2	110			1650	15	110			110	1.8	WENT THRU 1% AT FAILURE.

TEST NO.	MATERIAL TYPE	NITRIDE RUN NO.	MATERIAL CONDITION	COATING	TEMP F	STRESS KSI	1% HRS	2% HRS	TERM %	FAIL HRS	ELONG %	REMARKS
721	18-12-2	237			1500	20			0.29			TERMINATED AT 603 HRS. NEW HEAT.
722	18-12-2	236			1650	15	19			78.8	2.27	NEW HEAT.
723	18-12-2	110		CHROMIZED	1800	5	28		3.0			TERM AT 188.5 HRS.
724	18-12-2	110			1800	14				1.1	1.8	SHOULD HAVE BEEN 5.0 KSI.
725	18-12-2	233	FAST DENITRIDE		1650	15	24		1.3			TERMINATED AT 43.2 HR. 20 MIN. DENITRIDE.
726	18-12-2	110		AMI 100	1800	5	11		1.5			TERMINATED AT 40.7 HRS. ALUMINIZE CONTROL.
729	12-8-2	229	FINAL REDUCTION 60%	AMI 100	1650	15	20.3					CHROMIZE CONTROL. TERMINATED.
730	18-12-2	236		AMI 100	1650	15	45		2.0			TERMINATED AT 115 HR.
731	18-12-2	237		AMI 100	1800	5						TERMINATED AT 22.7 HR.
732	18-12-2	110		CHROMIZED	1800	2	82		1.8			TERMINATED AT 669 HR.
733	1N625		BRAZE CYCLED		1650	5	2.5			254	45	
734	1N625		BRAZE CYCLED		1800	2	6.5			312	50	FAILED WHEN UNLOADED.
735	1N625		BRAZE CYCLED		1500	10	14			472	22	
736	1N625		BRAZE CYCLED		1650	2.5	22.5					TERMINATED 23.2 HR.
737	1N625		AS RECEIVED		1650	5	17			45.6	61	
740	18-12-2	243			1800	5	15		3.6			TERMINATED 238 HR.
741	18-12-2	243		AMI 100	1800	5	58		2.7			TERMINATED AT 191 HR.
742	1NCD 800H				1800	2	18		23			TERMINATED AT 166 HR.
743	1NCD 800H			ALUMINIZED	1800	2	3.0		25			TERMINATED AT 142 HR.
744	18-12-2	243		AMI 100	1650	15	120			192	3.1	
745	18-12-2	245			1650	15	50		1.4			TERMINATED AT 117 HR.
746	18-12-2	244			1800	7.5			1.3			TERMINATED AT 90.4 HR.
747	18-12-2	245	"A"		1800	7.5	12			89.9	1.2	
748	18-12-2	247			1650	15	51		1.2			TERMINATED AT 89 HR.
749	18-12-2	253	"A"		1650	15	44		1.2			TERMINATED AT 163 HR.
755	18-12-2	256	"A"		1725	10	208		1.3			TERMINATED AT 248 HR.
756	18-12-2	257	"A"	NBF-50	1725	11.94	39		1.0			TERMINATED AT 39 HR.
757	18-12-2	263	"B"		1725	10			<1.0			TERMINATED AT 239 HR.
758	18-12-2	270	"C"		1725	10	148			156	3.1	
759	18-12-2	274	"A"		1725	11.92	16		1.0			TERMINATED AT 19 HR.
760	18-12-2	274	"A"		1800	7.5	34			162	2.7	PIN BENT - 1% POINT IS N.G.
762	18-12-2	273	"A"		1575	16	261	1200	1.8			TERMINATED AT 1226 HR.
763	1N625			AMI 100	1650	5	10			1102	50	HUNG UP - RUPTURE POINT N.G.
764	1N625			AMI 100	1500	10	12			1158	20	
765	18-12-2	265	"B"	AMI 100	1725	10	28		1.7			TERMINATED AT 88 HR.
766	18-12-2	265	"B"	AMI 100	1800	7.5	13.5			245	6.3	ONE PERCENT POINT SUSPECT.
767	18-12-2	274	"A"	NBF-50	1800	7.5			1.5			TERMINATED AT 92 HR.
768	12-8-2	272	PREANNEALED	AMI 100	1575	16	120	360		662	4.5	1900/3 MIN. PREANNEAL.
769	12-8-2	272	FINAL REDUCTION 60%	AMI 100	1650	15	26			136	4.5	
770	12-8-2	272	FINAL REDUCTION 60%	AMI 100	1725	10	19	65		152	4.5	
775	18-12-2		"A"	AMI 100	1575	14	168	578	3.5			TERMINATED AT 1097 HR.
776	18-12-2		"A"	AMI 100	1650	12.5	109			593	4.5	
777	18-12-2		"A"	AMI 100	1725	8	60			529	4.5	

TEST NO.	MATERIAL TYPE	NITRIDE RUN NO.	MATERIAL CONDITION	COATING	TEMP F	STRESS KSI	1% ELONG HRS	2% ELONG HRS	TERM %	FAIL HRS	ELONG %	REMARKS
779	12-8-2	271	PREANNEALED	AMI 100	1725	8	93.8	235		345	4.1	1900/3 MIN. PREANNEAL.
780	12-8-2	271	FINAL REDUCTION 60%	AMI 100	1650	12.5	59	126		210	3.6	
781	1N625			AMI 100	1725	8.0	0.5	1.0		12	30	
782	1N625			AMI 100	1575	14	1.0			42	34.5	
783	18-12-2	"B"		AMI 100	1725	10	55		1.0			TERMINATED.
784	18-12-2	"B"		AMI 100	1575	16		912	2.0			TERMINATED AT 912 HR.
785	18-12-2	"B"		AMI 100	1650	12.5	352	640	2.5			TERMINATED AT 867 HR.
786	18-12-2	"B"		AMI 100	1725	8	122	336	2.5			TERMINATED AT 770 HR.
787	18-12-2	"A"		AMI 100	1575	12.5			1.6	1330		TERMINATED AT 2012 HR.
788	1N625			AMI 100	1800	5				21.2	24.5	
789	18-12-2	"A"			1800	5				314	1.5	
790	18-12-2	"A"		AMI 100	1800	5			23.6			TERMINATED AT 1070 HR.
791	18-12-2	"A"		AMI 100	1800	5			30.9			TERMINATED AT 1070 HR.
792	18-12-2	"C"			1800	5				400	4.5	
795	18-12-2	"C"		AMI 100	1650	12.5	106			499	3.6	
796	18-12-2	"C"		AMI 100	1575	14	365		3.1			TERMINATED AT 932 HR.
799	18-12-2	"C"		AMI 100	1725	8	27			648	3.1	
800	18-12-2	"C"			1650	12.5	77			544	2.3	
801	18-12-2	"C"			1725	8	184			379	2.3	
802	18-12-2	"C"		AMI 100	1800	5				649	11.8	
803	18-12-2	284			1725	8	14.5			525	2.3	INSTRUMENTATION PROBLEM. TEST REPEATED 805.
804	18-12-2	284	NDB "C"	AMI 100	1725	8	142			688	4.0	SLIGHT MODIFICATION OF C-CYCLE.
805	18-12-2	284	NDB "C"		1725	8	121			648	3.0	
806	18-12-2	282	"B"	AMI 100	1800	5				743	4.5	
807	18-12-2	282	"B"	BRAZED + ALUM	1800	5				386	7.2	AMI 100 BRAZE.
808	18-12-2	282	"B"	AMI 100	1650	12.5	324	586	3.0			TERMINATED.
809	18-12-2	282	"B"	BRAZED + ALUM	1650	12.5	21			195	3.2	AMI 100 BRAZE.
810	1NCO 800H			ALUMINIZED	1800	2				328	12.7	FAILED UNDER PAD.
811	1NCO 800H			ALUMINIZED	1650	5	<1			50	31.8	
816	1NCO 800H			ALUMINIZED	1650	2	52	232	4.5			TERMINATED AT 1369 HR.
819	1NCO 800H			ALUMINIZED	1725	1	15	98	6.2			TERMINATED AT 1201 HR.
820	18-12-2	"B"		BRAZED + ALUM	1725	8	129			331	5.4	COATING FLAKED. AMI 100 BRAZE.
821	1N617	BRAZE CYCLED			1800	5				272	27.3	APPROX. 6 HR AT 1845 F
822	1N617			AMI 100B	1800	5				88.8	58.2	
823	1N617	BRAZE CYCLED			1650	7.5	7.2		35			TERMINATED AT 476 HR.
824	1N617	BRAZE CYCLED			1725	4	16.5	40	12.3			TERMINATED AT 476 HR.
825	18-12-2	"B"		BRAZED + ALUM	1650	12.5	165			333	2.3	
826	18-12-2	288		AMI 100	1725	8	194	523	3.0			TERMINATED AT 812 HR.
827	18-12-2	286		AMI 100	1725	8	114		0.9			TERMINATED, EXTENSOMETER SLIP.
828	18-12-2	288		NBF-50	1725	8	178	670	2.3			TERMINATED AT 840 HR.
829	18-12-2	287		AMI 100	1800	5	>182					BROKE ON COOL DOWN AT 420 HR.
830	18-12-2	287		NBF-50	1800	5	245	606	2.7			TERMINATED AT 732 HR.
831	18-12-2	287		AMI 100	1800	5	507	620	2.3			TERMINATED AT 663 HR.

TEST NO.	MATERIAL TYPE	NITRIDE RUN NO.	MATERIAL CONDITION	COATING	TEMP F	STRESS KSI	1% HRS	2% HRS	TERM %	FAIL HRS	ELONG %	REMARKS
832	18-12-2	288		MBF-50	1800	5	100	214	3.2			TERMINATED AT 732 HR.
833	18-12-2	288		AMI 100	1800	5	245	826		916	4.5	
834	18-12-2	286		AMI 100	1800	5	100	>662		875	4.0	
835	18-12-2	286		MBF-50	1800	5	100	560	2.7			TERMINATED AT 731 HR.
836	IN617			AMI 100	1725	8	<1			12.7	43	
837	IN617		BRAZE CYCLED		1725	8	<1			23.7	22	WENT OVER TEMP TO 1740F.

APPENDIX C  
TENSILE TEST DATA FOR NDS 300 MATERIALS

APPENDIX C  
TENSILE TEST DATA FOR NDS 300 MATERIALS

PROCESS CODE COATING	TEST TEMP	MECHANICAL PROPERTIES				REMARKS	
		0.2 YS, KSI COATED	0.2 YS, KSI BASE	UTS, KSI COATED	UTS, KSI BASE		% ELONG OVER 1"
A BARE	70	101.6		155.8		19	
A BARE	70	102.2		155.7		19	
A BARE	70	95.8		153.3		20	
A BARE	70	98.5		150.5		20	
A BARE	70	109.4		159.2		20	
A BARE	70	112.8		160.6		19	
A BARE	70	94.1		153.1		23	
A BARE	70	97.0		152.6		18	
A BARE	70	95.0		150.5		20	
A BARE	70	94.3		147.0		21	
A BARE	70	102.0		153.6		20	
A BARE	70	99.0		151.8		22	
A BARE	70	100.3		153.7		21	
A BARE	70	97.3		149.6		22	
A BARE	70	89.8		145.2		25	
A BARE	70	88.9		145.2		27	
A BARE	70	109.9		162.8		18.5	
A BARE	70	101.5		153.1		20.5	
A BARE	70	94.5		147.2		16	
A BARE	700	72.0		110.2		13	
A BARE	700	72.7		111.3		13.5	
A BARE	1100	60.6		85.0		15	
A BARE	1100	59.8		83.9		15	
A BARE	1300	53.9		64.0		9	
A BARE	1300	57.4		66.6		9	
A BARE	1500	41.8		45.4		4.5	
A BARE	1500	41.0		45.0		4.2	
A BARE	1650	34.9		36.9		3	
A BARE	1650	34.0		36.1		3	
A BARE	1800	29.1		30.7		2.0	
A COATED	70	89.5	125.3	111.5	156.1	8.0	
A COATED	70	91.8	135.8	113.3	167.6	6.0	
A COATED	70	94.0	122.8	125.7	164.2	19.0	
A COATED	70	93.4	122.0	130.9	171.0	12.2	
A COATED	700	89.8	123.9	99.0	136.6	2.2	MBF-50
A COATED	700	75.3	107.6	87.9	125.6	2.0	
A COATED	700	79.4	111.8	88.7	124.9	2.5	
A COATED	700	78.5	112.1	85.8	122.6	1.0	
A COATED	1100	65.9	104.2	70.0	110.7	2.0	
A COATED	1100	63.2	93.5	82.9	122.6	9.0	
A COATED	1100	68.5	108.3	71.7	113.4	1.0	
A COATED	1300	50.6	67.3	59.3	78.7	10.0	MBF-50
A COATED	1300	53.7	71.4	62.2	82.7	7.5	MBF-50
A COATED	1300	43.9	65.0	49.9	73.8	12.0	
A COATED	1300	46.2	69.8	53.5	80.8	4.0	
A COATED	1300	41.0	58.6	47.7	68.2	11.2	
A COATED	1300	45.3	62.9	51.0	70.8	9.0	

PROCESS CODE COATING	TEST TEMP	MECHANICAL PROPERTIES					REMARKS
		0.2 YS, COATED	YS, BASE	UTS, KSI		% ELDNG OVER 1"	
A COATED	1500	29.3	39.1	31.9	42.5	5.0	
A COATED	1500	28.5	41.7	31.2	45.7	6.5	
A COATED	1500	28.4	37.7	30.4	40.3	9.0	
A COATED	1500	29.5	40.8	31.5	43.0	8.2	
A COATED	1650	23.7	35.1	25.8	38.2	9.5	MBF-50
A COATED	1650	25.8	35.8	28.3	39.1	8.0	MBF-50
A COATED	1650	22.5	32.1	24.4	34.9	6.0	
A COATED	1650	23.8	30.4	25.5	32.5	6.0	
A COATED	1650	27.8	38.9	28.7	38.1	5.0	
A COATED	1800	18.4	27.9	19.8	30.0	3.0	
B BARE	70	76.2		135.1		21	
B BARE	70	79.9		137.2		20	
B BARE	70	83.4		143.7		21	
B BARE	70	84.1		143.0		22	
B BARE	70	85.3		142.5		19	
B BARE	70	84.5		142.1		23	
B BARE	70	92.1		146.5		19	
B BARE	70	94.5		150.5		19	
B BARE	70	87.4		142.0		21	
B BARE	70	88.1		142.3		22.5	
B BARE	70	84.8		140.7		25.2	
B BARE	700	64.8		103.9		11	
B BARE	1100	76.6		77.7		11.3	
B BARE	1300	46.7		53.1		5	
B BARE	1300	46.5		53.7		8	
B BARE	1300	50.7		59.8		7.0	
B BARE	1300	50.0		63.2		11.0	
B BARE	1500	37.6		40.2		4	
B BARE	1500	36.2		39.6		2.5	
B BARE	1650	29.3		31.1		2.2	
B BARE	1650	28.9		30.4		3.6	
B BARE	1650	30.5		31.5		2.0	
B BARE	1650	30.3		32.0		2.0	
B BARE	1650	32.5		34.3		2.5	
B BARE	1650	31.8		33.5		2.0	
B BARE	1650	26.8		27.6		5.0	
B BARE	1650	29.8		31.9		2.7	
B COATED	70	85.1	115.6	119.2	162.1	10.6	
B COATED	70	88.3	115.0	131.3	170.7	12.5	
B COATED	70	82.6	101.4	136.8	168.0	12.5	MBF-50
B COATED	70	87.1	117.1	116.5	156.1	8	MBF-50
B COATED	70	82.2	113.5	117.2	160.6	10.5	MBF-50
B COATED	700	69.1	88.1	92.1	117.5	6.0	
B COATED	700	80.3	114.7	95.6	122.3	1.8	
B COATED	1100	57.9	73.8	79.8	102.1	11.0	
B COATED	1100	76.6	99.6	79.9	103.9	1.4	

PROCESS CODE	TEST	MECHANICAL PROPERTIES					REMARKS
		0.2 YS, KSI		UTS, KSI		% ELONG	
COATING	TEMP	COATED	BASE	COATED	BASE	OVER 1"	
B COATED	1300	55.1	72.0	62.7	82.1	5.4	
B COATED	1300	58.2	74.5	68.3	87.4	6.4	
B COATED	1500	32.6	42.7	35.3	46.2	6.5	
B COATED	1500	32.0	43.8	35.9	49.1	5.9	
B COATED	1650	26.8	34.7	27.6	35.8	5.0	
B COATED	1650	26.2	32.6	28.1	35.0	9	MBF-50
B COATED	1650	21.1	32.2	22.6	34.4	4	
B COATED	1650	22.1	31.0	23.5	32.9	4	
C BARE	70	71.1		132.7		29	
C BARE	70	80.8		140.8		30	
C BARE	70	85.2		144.8		29	
C BARE	70	85.5		144.4		28	
C BARE	70	84.4		146.2		23	
C BARE	700	52.7		94		17.5	
C BARE	1100	52.5		82.2		18	
C BARE	1100	49.3		80.7		14	
C BARE	1300	35.5		46.3		15	
C BARE	1300	43.6		53.5		12	
C BARE	1300	43.6		53.5		11.8	
C BARE	1500	32.3		37.5		6.0	
C BARE	1500	33.2		36.6		5.0	
C BARE	1625	30.2		33.8		5.0	
C BARE	1650	27.5		31.2		4	
C BARE	1650	29.0		31.9		4.2	
C BARE	1650	30.3		30.5		4.0	
C COATED	70	86.8	105.9	124.5	151.9	9.0	
C COATED	70	92.3	121.8	124.3	164.1	6.2	
C COATED	1100	61.4	82.9	74.4	100.4	3.0	
C COATED	1100	60.7	82.0	75.5	101.9	5.8	
C COATED	1500	28.4	36.1	33.9	43.0	14.0	
C COATED	1500	26.3	31.4	31.1	41.4	16.0	

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DEVELOPMENT OF A NITRIDE DISPERSION  
STRENGTHENED (NDS) METALLIC ALLOY  
FOR HIGH-TEMPERATURE RECUPERATORS  
L. E. Kindlmann  
AirResearch Manufacturing Company  
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Key Words

Stainless Steel  
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