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DEVELOPMENT OF DISPERSION STRENGTHENED NICKEL – CHROMIUM ALLOY (Ni-Cr-Th0_2) SHEET FOR SPACE SHUTTLE VEHICLES

PART II

by

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FANSTEEL INC.
METALS DIVISION

prepared for

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16. Abstract

Two dispersion strengthened nickel base alloy systems have been developed for use at temperatures up to 1204°C(2200°F); TD Nickel Chromium (TDNiCr) and TD Nickel Chromium Aluminum (TDNiCrAl). They are considered candidate materials for use on the thermal protection systems of the space shuttle and for long term use in aircraft gas turbine engine applications. Improved manufacturing processes have been developed for the fabrication of TDNiCr sheet and foil to specifications. Sheet rolling process studies and extrusion studies were made on two aluminum containing alloys; Ni-16%Cr-3.5%Al-2%ThO_2 and Ni-16%Cr-5.0%Al-2%ThO_2. Over 1600 kg.(3500 lb.) of plate, sheet, foil, bar and extrusion products were supplied to NASA Centers for technology studies.

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FOREWORD

This report describes the results obtained during the second and concluding part of a development study of dispersion strengthened nickel-chromium alloys.

The developments described herein were conducted primarily at the Baltimore site of the Fansteel Metals Division; supporting chemical analyses were conducted principally at the Fansteel Muskogee plant.

In addition to the listed authors three other Fansteel engineers contributed substantially in the following areas:

J. E. Kennedy	Foil Rolling, Reproducibility Studies, and Creep Testing
J. E. Scheer	TDNiCrAl Process Studies
J. M. Stevens	TDNiCrAl Powder Production

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SUMMARY

Dispersion strengthened nickel base alloys are considered promising candidate materials which can be used without coatings for space shuttle vehicle applications requiring exposure in the 871-1204° C(1600-2200° F) temperature range.

In a previous Part I program processes were developed for the manufacture of TDNiCr (Ni-20%Cr-2%ThO_2) in sheet and foil. Standard processes were established for the production of TDNiCr sheet for the gauge range of 0.025-0.102 cm.(0.010-0.040 in.) in sheet sizes of 46 x 91 cm.(18 x 36 in.). A total of 821.1 kg.(1808.6 lb.) of TDNiCr sheet was manufactured and shipped to the NASA Centers or their Contractors for space shuttle technology studies.

A new alloy material was developed during the Part I program, TDNiCrAl (Ni-Cr-Al-ThO_2), in which the aluminum addition provides cyclic oxidation protection under severe test conditions at temperatures up to 1260° C(2300° F).

A Part II program was designed to continue the development of the dispersion strengthened nickel base alloys. The Part II program was modified during the course of the work to broaden the development of the TDNiCrAl alloys.

The goals of the modified Part II program were fivefold:

- Further develop the TDNiCr sheet manufacturing process to produce sheet 61.0 x 152.4 x 0.025-0.102 cm.(24 x 60 x 0.010-0.040 in.) having improved quality.
- Further develop the technology for manufacturing TDNiCr foil.
- Develop the manufacturing technology for producing large sheet sizes, 46 x 122 cm.(18 x 48 in.) of a TDNiCrAl alloy having improved oxidation resistance and high temperature strength comparable to TDNiCr.
- Develop extrusion technology for the extrusion of bar and mill shapes of TDNiCrAl alloys.
- Provide TDNiCr sheet, foil and bar, and TDNiCrAl alloy stock to NASA for use in other NASA technology programs.

The goals of the Part II program were attained and are summarized as follows:

- The process for the manufacture of TDNiCr sheet was scaled up successfully to provide sheet 61.0 x 152.4 x 0.025-0.102 cm.(24 x 60 x 0.010-0.040 in.). The billet size was increased to 68.0 kg.(150 lb.) and a number of process improvements were developed.

- An Alternate Process was developed for thin TDNiCr sheet, 0.025 to <0.038 cm.(0.010 to <0.015 in.) which utilized cold rolling for the finish rolling to gauge. This resulted in improvements in both surface finish and tolerances with some reduction in high temperature strength.
- High quality TDNiCr foil was produced by cold tension rolling. Foil gauges produced ranged from 0.0051 to 0.0127 cm.(0.002 to 0.005 in.) in widths of 46 cm.(18 in.).
- Preliminary sheet manufacturing processes were developed for production of evaluation quantities of two TDNiCrAl alloys, Ni-16%Cr-3.5%Al-2%ThO_2 and Ni-16%Cr-5.0%Al-2%ThO_2. The limited sheet process studies demonstrated that TDNiCrAl sheet can readily be manufactured, but higher rolling temperatures are required than for TDNiCr. The high temperature properties attained on sheet were lower than for TDNiCr.
- The 3.5 percent aluminum level appears to afford the optimum combination of oxidation resistance, ductility and strength for space shuttle applications. The 5.0 percent aluminum level is believed to afford additional oxidation resistance for long term use in applications such as aircraft engines.

- Extrusion process studies demonstrated that TDNiCrAl bar and shapes can be produced by extrusion in a wide range of sizes. Attainment of uniform high strength microstructures will require additional development studies.
- Quantities of TDNiCr and TDNiCrAl products manufactured and supplied to NASA for their technology programs are listed below.
- A total of 448.8 kg.(989.4 lb.) of TDNiCr plate, sheet and foil and 166.4 kg.(366.4 lb.) of extruded TDNiCr bar was manufactured and shipped to the NASA Centers or their Contractors for space shuttle technology studies.
- A total of 136.4 kg.(300.6 lb.) of TDNiCrAl sheet, 144.2 kg.(318 lb.) of TDNiCrAl extruded bar, and 347.9 kg.(767.0 lb.) of TDNiCrAl extruded shapes were manufactured and shipped to NASA for technology studies.
- Five densified billets of TDNiCrAl alloys, 396.5 kg.(874 lb.) were manufactured and shipped to NASA for future extrusion development studies.

INTRODUCTION

Dispersion strengthened nickel base alloys such as TDNiCr (Ni-20%Cr-2%ThO_2) are considered promising candidate materials which can be used without coatings for space shuttle vehicle applications requiring exposure in the 871-1204° C (1600-2200° F) temperature range.(1) The addition of aluminum (3.5 to 5.0%) to the nickel-chromium alloy matrix was shown in the Part I program(2) to provide improved cyclic oxidation protection under severe test conditions at temperatures up to 1260° C(2300° F). Thus, the aluminum modified alloys (designated TDNiCrAl) have potential for use as an improved shuttle material as well as applications in advanced gas turbine engines.

The Part I manufacturing development program was initiated as part of the NASA Space Shuttle Technology Program to further the development of TDNiCr sheet. The goals of the Part I program were met and exceeded. Processes were developed and TDNiCr sheet and foil were manufactured and delivered to NASA Centers for further studies. The new alloy system, TDNiCrAl, was developed which offers outstanding oxidation resistance. Specifically, these accomplishments included the following:

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(1) Blankenship, C. P. and Saunders, N. T.; Development of Dispersion Strengthened Ni-Cr-ThO_2 Alloys for Space Shuttle T.P.S., NASA TMX-68024, Mar. 1, 1972.

(2) NASA CR-120796, Development of Dispersion Strengthened Nickel-Chromium Alloy (Ni-Cr-ThO_2) Sheet for Space Shuttle Vehicles, Part I, 1971.

- A standard process was established for the production of TDNiCr sheet which met the goals of the program for the gauge range 0.025-0.102 cm.(0.010-0.040 in.) in sheet sizes of 46 x 91 cm.(18 x 36 in.).
- A total of 821.1 kg.(1808.6 lb.) of TDNiCr sheet was manufactured and shipped to the NASA Centers or their Contractors for space shuttle technology studies.
- High quality TDNiCr foil in gauges from 0.005-0.013 cm.(0.002-0.005 in.) was fabricated by cold tension rolling in widths exceeding the goal value of 30 cm.(12 in.).
- Alloy studies were made to improve the behavior of TDNiCr at 982-1204°C(1800-2200°F) under the severe oxidational environment to be experienced by the space shuttle. The addition of Al in the Ni-Cr alloy matrix provided cyclic oxidation protection under the most severe test conditions at temperatures up to 1260°C(2300°F). The basic alloy system recommended for further study was Ni-Cr-Al-ThO_2 alloys plus the possible addition of yttrium; the chromium levels to be 12-22%, the aluminum levels 2 to 4%.

A Part II program was designed to continue the development of the dispersion strengthened nickel base alloys. The program was accomplished under a continuation of NASA Contract NAS 3-13490. The original contract for Part I, April 23, 1970 to June 22, 1971 was extended for Part II to October 22, 1972.

The Part II program was further modified during the course of the work to broaden the development of the TDNiCrAl alloys to include extrusion with some decrease in the development of TDNiCrAl sheet alloys. This modification also de-emphasized the development of processing techniques for the production of fastener stock and fasteners from the TDNiCr and TDNiCrAl alloys.

The goals of the modified Part II program were as follows:

- Further develop the TDNiCr sheet manufacturing process to produce sheet 61.0 x 152.4 x 0.025-0.102 cm.(24 x 60 x 0.010-0.040 in.) having improved quality and high temperature ductility.
- Develop improved technology for manufacturing TDNiCr foil.
- Standardize the sheet production for large TDNiCr sheet and provide at least 317.5 kg. (700 lbs.) of sheet and foil for use in other NASA programs.

- Develop manufacturing technology for producing large sheet sizes, 46 x 122 cm.(18 x 48 in.), of a TDNiCrAl alloy having improved oxidation resistance and high temperature strength comparable to TDNiCr.
- Develop extrusion technology for the extrusion of bar and mill shapes of TDNiCrAl alloys.
- Provide at least 363 kg.(800 lbs.) of TDNiCrAl alloy billet and extruded shapes for use in other NASA programs.

The results of this Part II manufacturing technology development program are summarized in this report. All phases of the manufacturing technology development are described in the Discussion Section. Procedures for conducting mechanical property tests, and metallographic techniques including measuring grain size and ThO_2 size are detailed in reference 2. All mechanical property tests reported herein were conducted on test samples having an orientation normal to the rolling direction unless otherwise noted. Material specification requirements as referred to herein are those defined in Fansteel Specifications given in reference 2.

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INTERNATIONAL UNITS

The International System of Units, SI Units is used as the primary system of units in this report. The customary units are given as secondary units, parenthetically following the primary units. In the case of large "Tables", two versions are given, one in SI Units, and one in Customary Units.

The SI Units used in this report together with their symbols are listed below:

Physical Quantity	Name of Unit	Symbol
Length	meter	m
Length	centimeter	cm
Length	micron	μ
Mass	kilogram	kg
Time	second	s
Time	hour	hr
Temperature	degree centigrade	°C
Force	newton	N
Force	meganewton	MN
Stress	meganewtons per square meter	MN/m^2

DISCUSSION

I - TDNiCr SHEET PRODUCTION

1. Standard Process for 0.025-0.102 cm. x 60.96 cm. x 152.4 cm. (0.010-0.040 in. x 24 in. x 60 in.) Sheet

a. Rolling Practice for Manufacture of 60.96 cm.(24 in.) Wide Sheet

The results of the standard process development carried out in the Part I program indicated that the sheet size capability program goals could not be achieved with the 45.4 kg.(100 lb.) heats employed for the recommended finalized process. In order to make 60.96 x 152.4 cm.(24 x 60 in.) sheets over the gauge range from 0.025 to 0.102 cm.(0.010 to 0.040 in.) it was necessary to increase the starting size. The new size selected maintained the same nominal cross section of 8.25 x 21.59 cm.(3-1/4 x 8-1/2 in.), but increased the weight and thus the length by about 50 percent. As a result, the new weight was 68.0 kg. (150 lbs.) and the new length was a nominal 66.04 cm.(26 in.).

While thirty heats of the new larger size were fabricated during the Part I program, very little process optimization could be accomplished. It was, therefore, the objective of this portion of the program to optimize and finalize the TDNiCr sheet manufacturing process to produce sheet 61.0 x 152.4 x 0.025-0.102 cm.(24 x 60 x 0.010-0.040 in.) in size. Specific areas of effort included variations in sintering, canning, and rolling operations as described in the following sections.

(1.) Temperature-Volume Experiment

The roll consolidation technique as employed and developed during Part I combines the consolidation and hot

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roll breakdown operations. Initially it was believed that utilization of this processing technique would enable processing of a billet from the compacted state to the 0.254 cm. (0.1 in.) intermediate gauge plate state of processing in one canning operation. However, early work indicated a necessity for decanning and conditioning at the 2.54 cm.(1.0 in.) slab stage due to end and/or edge cracking and surface imperfections. In an effort to eliminate these difficulties, an additional investigation of variations in canning and rolling parameters was initiated.

During conventional roll consolidation processing, the picture frame portion of the can is fabricated in a manner that yields a cavity whose volume is equal to the volume of the compacted billet it contains at a theoretical density of 100 percent. Since it appears that the frame and its included cavity enlarges during the consolidation step of processing, edge restraints are believed to be reduced thus yielding a cavity larger in volume than the full dense compact it contains. Consequently surface tearing and edge and/or end cracking occur due to lack of can restraint. While more massive can components and/or stronger high temperature can components might be employed to eliminate these problems, resultant handling difficulties and/or cost increases would tend to cancel out yield improvements attained. For this reason, an alternate approach was evaluated. This approach is best described as an "overfill technique".

The standard picture frame component of the consolidation can employs mild steel border members which are 5.08 cm.(2.0 in.) high by 3.81 cm.(1.5 in.) wide. Therefore, the frame cavity volume is 5.08 cm.(2.0 in.) x width x length for a standard fill. The overfill technique evaluated employed a frame cavity volume 25 percent smaller. This was accomplished by using frame border members 3.81 cm.(1.5 in.) square.

The temperature utilized for the sinter and roll consolidation operations were also evaluated since they would determine the thoria size and influence the degree of plasticity of the TDNiCr compact during consolidation.

An experiment was designed utilizing six conventionally compacted TDNiCr 68.0 kg.(150 lb.) billets. The six billets were subdivided into three groups of two billets each which were sintered at 1010° C, 1067° C and 1121° C(1850° F, 1950° F and 2050° F) respectively. Subsequently, one billet sintered at each of the aforementioned temperatures was canned for roll consolidation in a can having a picture frame cavity equivalent in volume to the compact volume at 100 percent of theoretical density. The remaining three billets, one sintered at each temperature, were canned for roll consolidation in cans having picture frame cavities 25 percent less in volume than the compact volume at 100 percent of theoretical density. Each of the six billets was subsequently roll consolidated and hot rolled to the 2.54 cm.(1.0 in.) slab thickness at a temperature identical to its sinter temperature.

These parameters along with heat numbers assigned are shown in Table 1.

Observation during the roll consolidation and hot rolling operations revealed negligible differences in can condition between standard fill and overfilled units at the 2.54 cm.(1.0 in.) stage of processing on Heats 3792 through 3795. The can of Heat 3797 which was the standard filled unit processed at 1121° C(2050° F) looked very nearly similar to the cans of Heats 3792 through 3795, but the can of Heat 3796 was found to be broken open on the trailing edge corners, see Figure 1. This was attributed to the overfill not being contained by the picture frame component of the can since the mild steel edge borders and welded corners were of insufficient strength at the higher consolidation temperature of 1121° C (2050° F). As a result the TDNiCr consolidation of Heat 3796 was not sufficiently restrained and pushed through the can initiating bad edge and end cracks.

After cooling from the final hot rolling, each of the six units was mechanically decanned, visually examined and conditioned for subsequent fabrication to intermediate gauge plate. Samples were secured for thoria size determination and metallographic examination at this stage.

No discernable differences in general surface appearance and/or edge cracking were observed as a result of standard fill versus overfill except as noted above for Heat 3796. However, a definite trend was observed with respect to surface condition as a function of sinter-roll

TABLE 1

TEMPERATURE - VOLUME FILL EXPERIMENTAL PARAMETERS

Heat No.	Sinter Temp. °C	Sinter Temp. °F	Consolidation Temp. °C	Consolidation Temp. °F	Fill Variable
3792	1010	1850	1010	1850	Standard
3793	1010	1850	1010	1850	Overfill
3795	1067	1950	1067	1950	Standard
3794	1067	1950	1067	1950	Overfill
3797	1121	2050	1121	2050	Standard
3796	1121	2050	1121	2050	Overfill

Standard Fill

Overfill

FIGURE 1

STANDARD FILL AND OVERFILLED UNITS AFTER ROLL CONSOLIDATION AND HOT ROLLING TO 2.54 cm.(1.0 in.) AT 1121°C(2050°F) consolidation temperature. It was noted that the degree of surface irregularities decreased as sinter-roll consolidation temperature was increased. Similar results were also observed on various occasions during the Part I phase of the program. It was therefore concluded that plasticity of the TDNiCr sintered compacts improves as temperature is increased. However, the maximum temperature which can be used is limited by thoria growth and was established during Part I at 1093° C±28° (2000° F±50°).

No significant difference in degree of edge and/or end cracking could be observed as a result of sinter- roll consolidation temperature.

Yield data for Heats 3792 through 3797 at the 2.54 cm.(1.0 in.) stage of processing, after conditioning, are shown in Table 2. Also included in Table 2 are thoria size results for the heats in the as-hot rolled condition at a thickness of 2.54 cm.(1.0 in.). As indicated by these data, yield improved for standard filled billets and decreased for overfilled billets as the sinter-consolidation temperatures were increased. This behavior was most likely caused by two interacting variables, improved TDNiCr plasticity, and decreased can restraint with increasing temperature. Consequently, higher yields were experienced on standard filled billets primarily due to greater TDNiCr plasticity, while the overfilled billets exhibited poorer yields primarily as a result of less can restrain in conjunction with the overfilled condition.

TABLE 2

SLAB YIELD AND ThO_2 SIZE AT 2.54 CM.(1.0 IN.)

Heat No.	Sinter Temp.		Roll Temp.		Fill Variable	ThO_2 Size mμ	% Yield at 2.54 cm. (1.0 in.)
	°C	°F	°C	°F			
3792	1010	1850	1010	1850	Standard	15.0	51.7
3793	1010	1850	1010	1850	Overfill	15.0	75.3
3795	1067	1950	1067	1950	Standard	17.0	70.7
3794	1067	1950	1067	1950	Overfill	18.0	74.8
3797	1121	2050	1121	2050	Standard	21.5	79.1
3796	1121	2050	1121	2050	Overfill	20.5	65.3

It may also be noted in Table 2 that thoria sizes in the hot rolled condition at the 2.54 cm. (1.0 in.) thickness increase as the sinter-consolidation temperatures increase. The data were comparable to those obtained for similar sinter-consolidation and/or post hot rolling homogenization heat treatment cycles established during Part I.

Evaluation at the 2.54 cm. (1.0 in.) hot rolled conditioned thickness indicated that the 1121° C (2050° F) sinter-consolidation temperatures and standard fill condition produced the slab of highest yield having a thoria size comparable to Part I material subjected to a post hot rolling homogenization temperature of 1093° C (2000° F). In addition, it must be concluded that processing of a billet from the compacted state to the 0.254 cm. (0.1 in.) intermediate gauge plate stage of processing in one canning operation is not feasible. While surface condition in the hot rolled state at the 2.54 cm. (1.0 in.) thickness was improved appreciably, it was still necessary to condition the slab to some extent prior to further rolling. Higher sinter-consolidation temperatures might further improve surface condition at the 2.54 cm. (1.0 in.) slab thickness, but are not recommended for use since excessive thoria growth could occur.

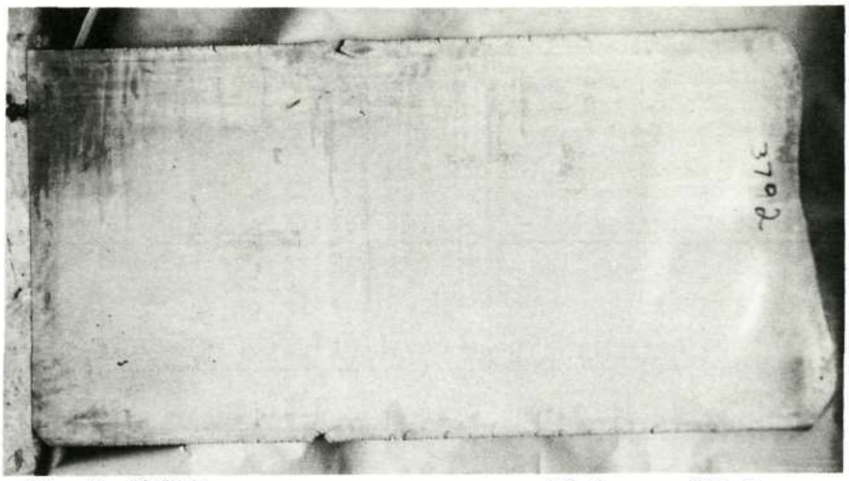
All six of the conditioned 2.54 cm. (1.0 in.) slabs resulting from the temperature-can design investigation were fabricated to a warm rolled gauge of 0.254 cm. (0.1 in.) for evaluation. Heats 3792 and 3793, having thoria sizes of 15 mμ at the 2.54 cm. (1.0 in.) slab thickness, were subjected to a heat treatment of 1093° C (2000° F) for two hours in order to adjust thoria size to a satisfactory level. Heats 3794, 3795, 3796 and 3797 were processed as is since they each contained thoria sizes of an acceptable level, i.e., 18.0, 17.0, 20.5 and 21.5 mμ respectively.

At the 0.254 cm.(0.1 in.) stage of processing, a marked difference in edge cracking was observed as a result of thoria size, see Figure 2. Heats 3792 and 3796 have comparable thoria sizes, while Heat 3795 having a lesser thoria size exhibits the greatest quantity of edge cracking.

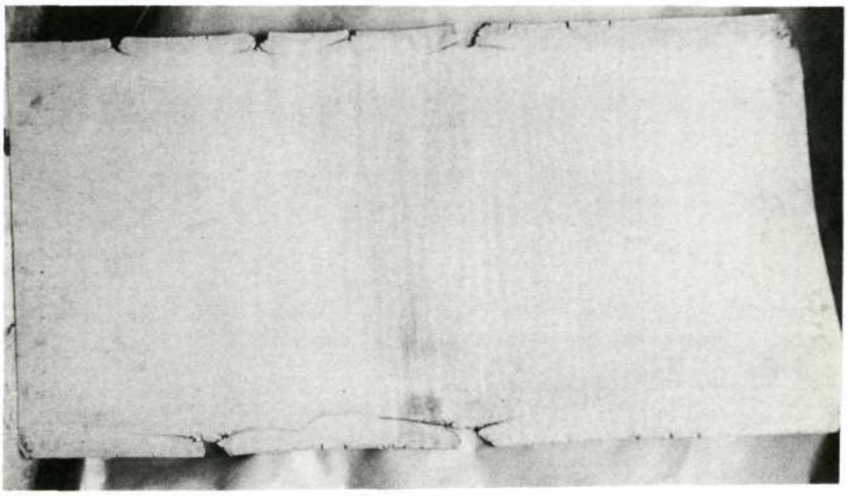
Subsequent to chemical and mechanical conditioning operations at the 0.254 cm.(0.1 in.) intermediate plate stage of processing, samples were obtained for chemical analysis and thoria size determination. These data are summarized in Table 3. Heat 3797 was only fabricated to a thickness of 1.02 cm.(0.400 in.) because it developed excessive cracking as a result of improper handling during breakdown rolling. Therefore, chemistry and thoria size results were obtained at this gauge.

As indicated by the data presented in Table 3, chemical analysis results appear to be typical for TDNiCr. The thoria size results compare favorably with those obtained at the 2.54 cm.(1.0 in.) slab thickness for Heats 3794 through 3797 while those obtained on Heats 3792 and 3793 appear satisfactory considering the 1093° C(2000° F) thoria size control heat treat to which these heats were subjected.

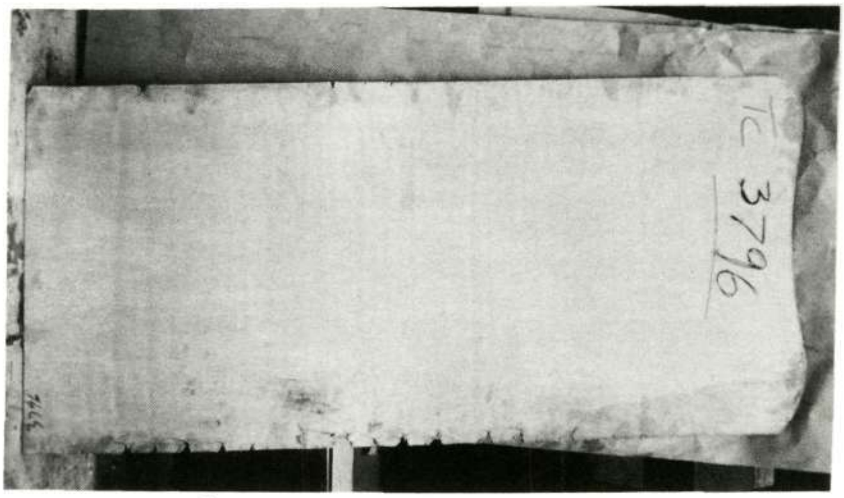
In summary, the standard filled unit in conjunction with a 1093° C±28° (2000° F±50°) sinter and roll con-



Heat 3792 ThO_2 - 20.0 mμ



Heat 3795 ThO_2 - 18.0 mμ



Heat 3796 ThO_2 - 21.0 mμ

FIGURE 2

EFFECT OF THORIA SIZE UPON EDGE CRACKING AT THE 0.254 cm.(0.100 in.) STAGE OF PROCESSING

TABLE 3

CHEMICAL ANALYSIS AND THORIA SIZE OF TDNiCr PLATE

Heat No.	Chemical Constituents in Percent					ThO_2 Size - mμ
	C	Cr	S	ThO_2	Ni	
3792	0.019	19.60	0.005	2.11	Bal.	20.0
3793	0.025	19.56	0.004	2.13	Bal.	21.5
3794	0.030	19.78	0.002	2.18	Bal.	19.0
3795	0.026	19.77	0.005	2.13	Bal.	18.0
3796	0.018	19.55	0.005	2.16	Bal.	21.0
3797	0.026	19.70	0.005	2.18	Bal.	21.0

solidation temperature was found to be optimum. However, no combination of volume-temperature variables evaluated yielded a satisfactory process for fabrication of a TDNiCr billet from the compacted state to the 0.254 cm.(0.1 in.) intermediate gauge plate state of processing in one canning operation.

(2.) Sinter-Roll Consolidation Preheat Combination

Five additional 68.0 kg.(150 lb.) units were initiated. They were identified as Heats 3826 through 3830. These heats were employed for a controlled study to determine the effect of combining the sinter operation with the roll consolidation preheat operation. The primary advantage of combining these operations into one step is the elimination of cooling down the billet after sintering, decanning, recanning and subsequent reheating for roll consolidation. Excessive handling and possible contamination of the billet is thus eliminated.

Previous trial runs utilizing the NASA furnished Pereny Sinter-Consolidation Furnace were carried out during Part I, however, no control billet was employed since standard processing procedures for 68.0 kg.(150 lb.) billets were not established at that time.

The five Heats 3826 through 3830 were compacted utilizing conventional processing techniques. Heat 3826 was selected as the control unit and was canned in a mild steel loose fitting container in preparation for a conventional sinter operation at 1121° C(2050° F) in the Harper Furnace. It was subsequently decanned and recanned in a roll consoli-

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dation can in preparation for a preheat operation at 1121° C(2050° F) in the Pereny Furnace. It was then roll consolidated and hot rolled by standard procedures. Heats 3827 through 3830 were canned in conventional roll consolidation cans in the as-compacted condition. They were then subjected to a 1121° C(2050° F) sinter-roll consolidation preheat cycle in the Pereny Furnace and subsequently roll consolidated and hot rolled to the 2.54 cm.(1.0 in.) stage of processing. Each of the five heats was then mechanically decanned and conditioned as required for further rolling to intermediate gauge plate.

Thoria sizes determined at this stage of processing were found to be as follows:

Heat 3826	18.0 mμ
Heat 3827	17.5 mμ
Heat 3828	17.0 mμ
Heat 3829	18.0 mμ
Heat 3830	17.5 mμ

These results appeared quite satisfactory. Therefore, the five units were canned in a conventional manner for subsequent breakdown rolling at 768° C(1400° F) to intermediate gauge plate thicknesses. Heat 3827 was stopped at approximately 0.76 cm. (0.3 in.) to satisfy a NASA requirement for unrecrystallized 0.64 cm.(0.25 in.) plate stock.

Samples for chemical analysis were obtained at this gauge. The results, which appear quite usual, are shown in Table 4. Total oxygen determinations were also carried

TABLE 4

CHEMICAL ANALYSIS RESULTS OF TDNiCr INTERMEDIATE GAUGE PLATE

Heat No.	Constituents in Percent				
	ThO_2	Cr	C	S	Ni
3826	2.14	19.60	0.02	0.004	Bal.
3827	2.14	19.41	0.02	0.006	Bal.
3828	2.11	19.58	0.02	0.004	Bal.
3829	2.11	19.55	0.02	0.006	Bal.
3830	2.15	19.68	0.02	0.004	Bal.

out on all five heats and were found to be in the 3500 to 4000 ppm range. These are considered to be on the low side of the range for typical TDNiCr product (3500 to 5500 ppm) determined during the Part I Program.

Based upon these results and general observations during the fabrication of Heats 3826 through 3830, the combination of the sintering and roll consolidation preheat operations appeared to be quite feasible and was therefore recommended for inclusion in the standard process.

(3.) Reduction-Grain Size Relationship

Intermediate gauge plate resulting from the Temperature Volume Investigation and the Sinter-Roll Consolidation Preheat Combination evaluation described in the two previous sections was fabricated to 0.102 cm.(0.040 in.) thick sheet for the purpose of evaluation and possible use as production sheet.

At the 0.114 cm.(0.045 in.) thick stage of processing, pilot specimens were obtained from each of the heats (3792 through 3796 and 3826 through 3830) processed. These specimens were heat treated and recrystallized in the production furnace by conventional techniques. Mechanical properties were determined and a metallographical evaluation was carried out. The mechanical property data and average grain diameters for each heat are summarized in Table 5. As indicated by these data, room temperature ultimate tensile strength and yield strength values were found to be marginal in the cases of Heats 3792 through 3796 and 3826. These results along with marginal to poor bend results were attributed to the unusually large average grain sizes determined by metallography.* Average grain sizes, for 0.102 cm. (0.040 in.) sheet fabricated from 2.54 cm.(1.0 in.) slabs having thoria sizes of 18 to 21 mμ size range, typically would be in the 0.030 to 0.060 mm size range. Heats 3792 through 3796 and 3826 had average grain sizes appreciably larger than normal.

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*Metallography procedures defined in reference 2, NASA CR-120796.

TABLE 5

MECHANICAL PROPERTIES AND GRAIN SIZE OF TDNiCr SHEET

		Room Temperature			1093°C			1093°C		
Heat No.	Gauge Cm.	UTS MN/M^2	Y.S. MN/M^2	Elong. %-Cm.	UTS MN/M^2	Y.S. MN/M^2	Elong. %-Cm.	Stress Rupture 20 hrs. @ 37.9 MN/M^2	Min. Bend	Avg. Grain Diameter mm
3792	0.114	762.7	518.8	23.0	126.1	125.4	2.5	OK	3.3 T	0.126
3793	0.114	768.2	514.0	23.3	129.5	128.8	3.5	OK	4.4 T	0.121
3794	0.114	795.8	518.8	21.8	136.4	136.4	3.5	OK	3.1 T	0.106
3795	0.114	752.4	483.7	23.1	130.2	127.5	3.0	OK	4.7 T	0.146
3796	0.114	770.3	486.4	22.0	121.3	119.2	3.0	OK	3.0 T	0.099
3826	0.114	803.4	527.8	24.5	136.4	135.7	2.5	OK	3.5 T	0.095
3827		FABRICATION STOPPED AT 0.640 cm.								
3828	0.114	840.6	540.2	23.5	124.7	113.7	2.5	OK	3.0 T	0.044
3829	0.114	814.4	540.9	25.5	126.1	125.4	2.5	OK	3.0 T	0.059
3830	0.114	821.3	534.7	19.5	126.1	125.4	2.0	3.2 2.1	3.0 T	0.033

TABLE 5

MECHANICAL PROPERTIES AND GRAIN SIZE OF TDNiCr SHEET

		Room Temperature			2000°F			2000°F Stress Rupture		
Heat No.	Gauge In.	UTS KSI	Y.S. KSI	Elong. %-In.	UTS KSI	Y.S. KSI	Elong. %-In.	20 hrs. @ 5500 psi	Min. Bend	Avg. Grain Diameter mm
3792	0.045	110.7	75.3	23.0	18.3	18.2	2.5	OK	3.3 T	0.126
3793	0.045	111.5	74.6	23.3	18.8	18.7	3.5	OK	4.4 T	0.121
3794	0.045	115.5	75.3	21.8	19.8	19.8	3.5	OK	3.1 T	0.106
3795	0.045	109.2	70.2	23.1	18.9	18.5	3.0	OK	4.7 T	0.146
3796	0.045	111.8	70.6	22.0	17.6	17.3	3.0	OK	3.0 T	0.099
3826	0.045	116.6	76.6	24.5	19.8	19.7	2.5	OK	3.5 T	0.095
3827		FABRICATION STOPPED AT 0.250 IN.								
3828	0.045	122.0	78.4	23.5	18.1	16.5	2.5	OK	3.0 T	0.044
3829	0.045	118.2	78.5	25.5	18.3	18.2	2.5	OK	3.0 T	0.059
3830	0.045	119.2	77.6	19.5	18.3	18.2	2.0	3.2 2.1	3.0 T	0.033

In an attempt to isolate the cause for the grain size anomaly encountered during the fabrication of the aforementioned heats, a detailed review of all processing data relevant to 68.0 kg.(150 lb.) TDNiCr units fabricated during the Part I and Part II portions of the program was carried out. No trends were established, nor were any unusual data encountered during processing from the powder stage through the 0.254 cm.(0.1 in.) intermediate plate thickness that could be pinpointed as the cause of grain size anomaly. This conclusion was verified by metallographic evaluation of material at the intermediate plate gauge. Therefore, it was established that the grain size anomaly occurred during fabrication from the intermediate plate stage to final gauge. The only variables contributing during this portion of processing are rolling temperatures and times, roll pass schedules and recrystallization heat treatment cycles. Because both abnormal and normal heats were yielded from single recrystallization heat treat furnace loads, it was believed that the heat treat variable was not the primary cause of the grain size anomaly. Accordingly, work piece temperatures and/or roll pass schedules remained the suspect variables.

It was indicated during the Part I program that reductions per pass increase as temperature increases if roll gap is maintained at a constant setting. Therefore, it may be concluded that variability in reductions per pass from sheet to sheet may only occur as a result of either variability in sheet dimensions, or the operators discretion in an effort to adjust pass reductions to attain sheet shape, since the furnace temperatures are accurately maintained and soak times are sufficient to attain proper work piece temperature for the most extreme working parameters. For these reasons a simple investigation was initiated to determine effect of average reduction per pass upon grain size.

One sheet of Heat 3828, 0.144 x 63.50 x 171.45 cm. (0.045 x 25 x 67-1/2 in.), was warm sheared into two equal pieces, 0.114 x 63.50 x 81.28 cm. (0.045 x 25 x 32 in.). These pieces identified as 3828-A and 3828-B were subsequently rolled at 760° C (1400° F) to 0.084/0.089 cm. (0.033/0.035 in.) using light reductions and heavy reductions respectively, as shown in Table 6. Each of the sheets was canned in aluminized steel by conventional means and reheated after each double pass. As indicated in Table 6, the light reductions yielded an average reduction per pass of 3.3 percent while the heavy reductions yielded an average reduction per pass of 5.4 percent.

After rolling, specimens were obtained from each heat and subjected to metallographic evaluation and tensile testing at both ambient and elevated temperatures. The results along with those obtained on the starting material at 0.114 cm. (0.045 in.) thickness are presented in Table 7. Based upon these results, it may be noted that the lighter reductions per pass utilized on Piece 3828-A yielded an average grain size of 0.095 mm while the heavier reductions per pass typified by piece 3828-B yielded an average grain

TABLE 6

ROLL PASS SCHEDULES FOR TDNiCr SHEET

Heat 3828-A		Heat 3828-B	
Double Pass No.	Percent Reduction	Double Pass No.	Percent Reduction
1	3.0	1	3.0
2	2.9	2	4.4
3	4.3	3	6.4
4	3.7	4	5.5
5	3.6	5	6.6
6	3.2	6	6.2
7	4.8		
8	1.2		
9	3.4		
Average Per Pass	3.3	Average Per Pass	5.4

TABLE 7

MECHANICAL PROPERTIES AND GRAIN SIZE FOR TDNiCr SHEET

		Room Temperature			1093°C				
Heat No.	Gauge cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	Min. Bend	Average Grain Size mm
3828	0.102	840.6	540.2	23.5	124.7	113.7	2.5	3.0T	0.044
3828-A	0.076	780.6	488.5	24.0	120.6	120.6	2.0	4.0T	0.095
3828-B	0.076	815.1	524.3	22.5	124.7	124.7	2.0	2.8T	0.062

		Room Temperature			2000°F				
Heat No.	Gauge in.	UTS ksi	YS ksi	Elong. %-in.	UTS ksi	YS ksi	Elong. %-in.	Min. Bend	Average Grain Size mm
3828	0.040	122.0	78.4	23.5	18.1	16.5	2.5	3.0T	0.044
3828-A	0.030	113.3	70.9	24.0	17.5	17.5	2.0	4.0T	0.095
3828-B	0.030	118.3	76.1	22.5	18.1	18.1	2.0	2.8T	0.062

size of 0.062 mm. Further, it may be noted that mechanical properties obtained on 3828-A having the coarser grained structure are quite similar to those obtained on Heats 3792 through 3796, Table 5. These properties are typified by lower than usual room temperature tensile strength and yield strength accompanied by minimum bend of greater than 3.0T. On the other hand, properties and grain size of 3828-B, fabricated with the heavier reductions, appear quite normal and compare very favorably with results from the Part I program.

While an apparent definite relationship exists between roll pass reductions and resulting grain size, all of the ramifications were not established. Therefore, additional investigations were initiated in an effort to better define the reduction-grain size relationship.

Powder preparation was initiated and completed for the fabrication of additional 68.0 kg.(150 lb.) TDNiCr units. These three units identified as Heats 3869, 3870 and 3874 were utilized for definition of the reduction-grain size relationship.

Heats 3869 and 3870 were compacted, sinter-roll-consolidated and hot rolled at a temperature of 1121° (2050° F) to a 2.54 cm.(1.0 in.) thickness by conventional techniques. Usual decanning, conditioning, and recanning followed. Again conventional procedures were employed to breakdown roll to the intermediate plate stage of processing.

After chemical conditioning, the intermediate gauge plates of Heat 3869 and 3870 were warm sheared into four equal lengths. Two pieces from each heat were then sheared to widths of 50.80 cm.(20 in.) and the remaining two pieces from each heat to widths of 66.04 cm.(26 in.) for Heat 3869 and 64.77 cm.(25.5 in.) for Heat 3870. Each piece was then canned in aluminized steel in preparation for warm rolling.

One pair of narrow and wide sheets from Heat 3869 were subsequently rolled to 0.114 cm.(0.045 in.) on the Schloemann mill* using roll gap increments of 10 percent reductions and a second pair were rolled to the same gauge using roll gap increments of 5 percent reductions. At this stage of processing, samples were obtained for determination of mechanical properties and metallographical evaluation. Next, each pair of sheets was halved and assembled into a two sheet per pack configuration for further rolling to a gauge of 0.056 cm.(0.022 in.) on the Schloemann mill making sure that each pair of narrow and wide sheets were subjected to the same roll gap reduction increment as employed during rolling to the 0.114 cm.(0.045 in.) thickness. After samples were obtained for determination of mechanical properties and metallographical evaluation at the 0.056 cm.(0.022 in.) gauge, each sheet was again halved and assembled into four sheet per pack configurations for rolling to a final gauge of 0.030 cm. (0.012 in.). Each sheet was then subjected to chemical cleaning, recrystallization, wide belt abrasive grinding to

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*Work rolls 88.8 cm.(35 in.) diameter; maximum separating force 2.2 million kg.(4.8 million lbs.).

a gauge of 0.025/0.029 cm.(0.010/0.0115 in.), cold tension rolling a maximum of 5 percent reduction and a final stress relief of 1177° C(2150° F) for two hours. Samples were then obtained for testing purposes.

A similar experiment was also carried out employing Heat 3870 and the United rolling mill* with roll gap increments of 5 and 2-1/2 percent reductions. By this means, it was anticipated that the effect of reduction upon grain size and sheet quality could be evaluated.

Table 8 summarizes the results of mechanical property testing and the metallographical evaluation carried out on Heats 3869 and 3870 at a finish gauge of 0.102 cm. (0.040 in.).

These data indicate that if constant roll gap settings are maintained, average percent reductions per pass increase as sheet width is decreased. As a result, average roll pass reductions were decreased during sheet size scale up studies as product sheet width was increased from 45.72 cm. (18 in.) to 60.96 cm.(24 in.). The decreased roll pass reductions during fabrication from the intermediate plate gauge to a finish gauge of 0.102 cm.(0.040 in.) were found to be detrimental to mechanical properties and microstructure as indicated by the data shown in Table 8. For example, it may be noted that macro grain sizes increase as average reductions per pass decrease, resulting in marginal to failing room temperature tensile and yield values. Further, it should be noted that ductility as reflected by bend testing also

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*Work rolls 88.8 cm.(35 in.) diameter; maximum separating force 1.4 million kg.(3.0 million lbs.).

TABLE 8

MECHANICAL PROPERTIES AND GRAIN SIZE OF 0.102 cm. TDNiCr SHEET

				Room Temperature			1093° C						
Heat No.	Gauge cm.	Width	Ave.% Red. Per Pass	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	1093° C Stress Rupture Hrs. @ 37.9 MN/m^2	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size cm.
3869-1	0.102	W	6.2	808.2	516.8	20.5	147.4	147.4	3.5	>20.0	3.5	0.061	0.102
3869-2	0.102	N	8.1	838.5	536.7	20.7	133.7	119.9	2.4	>20.0	3.0	0.038	0.076
3869-3	0.102	W	3.5	810.3	489.9	21.5	114.4	114.4	2.5	>20.0	3.5	0.082	0.152
3869-4	0.102	N	3.7	811.0	516.8	20.0	119.2	116.4	2.5	>20.0	3.5	0.064	0.152
3870-1	0.102	W	2.9	766.9	519.5	24.5	124.5	124.5	2.5	>20.0	4.0	0.140	0.254
3870-2	0.102	N	2.9	790.9	533.3	25.0	122.6	122.6	1.0	>20.0	3.5	0.100	0.178
3870-3	0.102	W	1.2	751.0	518.8	25.0	119.8	119.8	2.5	>20.0	5.0	0.250	0.510
3870-4	0.102	N	1.3	745.5	505.0	24.0	117.8	113.0	3.0	>20.0	4.0	0.160	0.300

MECHANICAL PROPERTIES AND GRAIN SIZE OF 0.040 in. TDNiCr SHEET

				Room Temperature			2000° F						
Heat No.	Gauge in.	Width	Ave.% Red. Per Pass	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	2000° F Stress Rupture Hrs. @ 5.5 ksi	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size in.
3869-1	0.040	W	6.2	117.3	75.0	20.5	21.4	21.4	3.5	>20.0	3.5	0.061	0.04
3869-2	0.040	N	8.1	121.7	77.9	20.7	19.4	17.4	2.4	>20.0	3.0	0.038	0.03
3869-3	0.040	W	3.5	117.6	71.1	21.5	16.6	16.6	2.5	>20.0	3.5	0.082	0.06
3869-4	0.040	N	3.7	117.7	75.0	20.0	17.3	16.9	2.5	>20.0	3.5	0.064	0.06
3870-1	0.040	W	2.9	111.3	75.4	24.5	18.5	18.5	2.5	>20.0	4.0	0.140	0.10
3870-2	0.040	N	2.9	114.8	77.4	25.0	17.8	17.8	1.0	>20.0	3.5	0.100	0.07
3870-3	0.040	W	1.2	109.0	75.3	25.0	17.4	17.4	2.5	>20.0	5.0	0.250	0.20
3870-4	0.040	N	1.3	108.2	73.3	24.0	17.1	16.4	3.0	>20.0	4.0	0.160	0.12

decreases as reductions per pass decrease. These characteristics were observed on all materials fabricated to a finish gauge of 0.102 cm.(0.040 in.) utilizing average roll pass reductions of 6.2 percent or less. Piece 3869-2 which was subjected to 8.1 percent per pass average reductions, on the other hand, satisfied all mechanical property requirements.

Since it was indicated during the Part I studies that 0.102 cm.(0.040 in.) sheet having an average micro grain size of less than 0.030 mm will have marginal stress rupture properties and the data presented in Table 8 indicates that average micro grain sizes of 0.061 mm or greater result in minimum bends of greater than 3.0 T, it was concluded that the standard process should include the stipulation that fabrication from intermediate plate to 0.102 cm.(0.040 in.) gauge shall utilize average roll pass reductions of greater than 6.2 percent and less than 8.2 percent in order to attain an optimized microstructure yielding satisfactory mechanical properties.

Tables 9 and 10 summarize the results of mechanical property testing and the metallographical evaluation carried out on Heats 3869 and 3870 at the finish gauges of 0.051 cm.(0.020 in.) and 0.025 cm.(0.010 in.).

While the rolling from 0.102 cm.(0.040 in.) to a finish gauge of 0.051 cm.(0.020 in.) was carried out in a double thickness pack configuration, it may be noted from the average percent reductions presented in Table 11 that again, reductions decrease as sheet width increases when constant roll gap openings are maintained.

It should also be pointed out that during the rolling from intermediate plate gauge to a finish gauge of 0.102 cm.(0.040 in.), the effect of varying average reductions per pass upon microstructure was evaluated based upon similar starting materials whereas during the rolling from 0.102 cm. (0.040 in.) to a final gauge of 0.051 cm.(0.020 in.), the effect of varying average reductions per pass was evaluated upon varying microstructures established during previous rolling. Accordingly, upon referring to Tables 8 and 9, it may be noted that pieces identified as 3869-1A and -1B contain a slightly finer micro and macro grain size than the starting material 3869-1 at 0.102 cm.(0.040 in.) thickness. This change was believed to be the primary cause of the improved room temperature yield strength and minimum bend characteristics. Secondly, pieces identified as 3869-2A and -2B also contain slightly increased room temperature yield strengths and improved minimum bend radii with negligible grain size changes.

In the case of pieces 3869-3A, 3B, 4A and 4B, average macro grain size has remained in the same magnitude, but micro grain size increased slightly. Again an increase in room temperature yield strength may be observed, however, bend radii while showing improvement over that reported for the 0.102 cm.(0.040 in.) material may still be considered

TABLE 9

MECHANICAL PROPERTIES AND METALLOGRAPHY RESULTS OF 0.051 cm. TDNiCr SHEET

Heat No.	Gauge cm.	Width	Ave.% Red. Per Pass	Room Temperature UTS MN/m^2	Room Temperature YS MN/m^2	Room Temperature % Elong.	1093° C UTS MN/m^2	1093° C YS MN/m^2	1093° C % Elong.	1093° C Stress Rupture Hrs. @ 37.9 MN/m^2	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size cm.
3869-1A	0.051	W	5.2	822.7	552.6	20.0	117.8	104.0	2.5	>20.0	2.0	0.041	0.051
3869-1B	0.051	W	5.2	779.3	534.7	16.5	111.6	106.8	2.4	>20.0	1.7	0.043	0.064
3869-2A	0.051	N	6.0	846.1	563.6	19.7	118.5	99.2	2.2	>20.0	1.8	0.045	0.076
3869-2B	0.051	N	6.0	836.4	568.6	19.7	129.5	124.0	2.6	>20.0	1.8	0.046	0.076
3869-3A	0.051	W	4.7	804.1	533.3	24.5	143.9	137.1	3.0	>20.0	3.9	0.088	0.152
3869-3B	0.051	W	4.7	793.8	538.8	22.4	123.3	122.0	2.5	>20.0	2.3	0.108	0.127
3869-4A	0.051	N	5.3	823.4	542.9	25.0	120.6	119.2	2.1	>20.0	2.5	0.077	0.152
3869-4B	0.051	N	5.3	789.6	529.2	22.4	117.8	117.8	2.5	>20.0	3.0	0.086	0.127
3870-1A	0.051	W	1.8	711.7	494.0	25.0	87.7	87.7	2.0	>20.0	5.0	0.400	0.760
3870-1B	0.051	W	1.8	690.4	492.6	24.0	104.0	104.0	2.0	-	4.0	0.400	0.760
3870-2A	0.051	N	1.8	725.5	496.7	27.2	107.5	107.5	2.0	>20.0	5.0	0.200	0.430
3870-2B	0.051	N	1.8	715.9	494.0	25.5	108.9	108.9	0.5	>20.0	5.0	0.200	0.430
3870-3A	0.051	W	1.1	706.4	485.1	20.2	106.1	106.1	5.0	>20.0	5.0	0.200	0.430
3870-3B	0.051	W	1.1	806.1	540.2	23.5	93.7	93.7	2.5	>20.0	5.0	0.200	0.430
3870-4A	0.051	N	1.3	732.4	503.7	22.5	103.4	103.4	2.5	>20.0	5.0	0.160	0.300
3870-4B	0.051	N	1.3	742.1	494.0	22.5	107.5	107.5	2.0	>20.0	5.0	0.160	0.300

TABLE 9

MECHANICAL PROPERTIES AND METALLOGRAPHY RESULTS OF 0.020 in. TDNiCr SHEET

				Room Temperature			2000° F						
Heat No.	Gauge in.	Width	Ave.% Red. Per Pass	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	2000° F Stress Rupture Hrs. @ 5.5 ksi	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size in.
3869-1A	0.020	W	5.2	119.4	80.2	20.0	17.1	15.1	2.5	>20.0	2.0	0.041	0.020
3869-1B	0.020	W	5.2	113.1	77.6	16.5	16.2	15.5	2.4	>20.0	1.7	0.043	0.025
3869-2A	0.020	N	6.0	122.8	81.8	19.7	17.2	14.4	2.2	>20.0	1.8	0.045	0.030
3869-2B	0.200	N	6.0	121.4	82.5	19.7	18.8	18.0	2.6	>20.0	1.8	0.046	0.030
3869-3A	0.020	W	4.7	116.7	77.4	24.5	20.6	19.9	3.0	>20.0	3.9	0.088	0.060
3869-3B	0.020	W	4.7	115.2	78.2	22.4	17.9	17.7	2.5	>20.0	2.3	0.108	0.050
3869-4A	0.020	N	5.3	119.5	78.8	25.0	17.5	17.3	2.1	>20.0	2.5	0.077	0.060
3869-4B	0.020	N	5.3	114.6	76.8	22.4	17.1	17.1	2.5	>20.0	3.0	0.086	0.050
3870-1A	0.020	W	1.8	103.3	71.7	25.0	12.7	12.7	2.0	>20.0	5.0	0.400	0.300
3870-1B	0.020	W	1.8	100.2	71.5	24.0	15.1	15.1	2.0	-	4.0	0.400	0.300
3870-2A	0.020	N	1.8	105.3	72.1	27.2	15.6	15.6	2.0	>20.0	5.0	0.200	0.170
3870-2B	0.020	N	1.8	103.9	71.7	25.5	15.8	15.8	0.5	>20.0	5.0	0.200	0.170
3870-3A	0.020	W	1.1	102.5	70.4	20.2	15.4	15.4	5.0	>20.0	5.0	0.200	0.170
3870-3B	0.020	W	1.1	117.0	78.4	23.5	13.6	13.6	2.5	>20.0	5.0	0.200	0.170
3870-4A	0.020	N	1.3	106.3	73.1	22.5	15.0	15.0	2.5	>20.0	5.0	0.160	0.120
3870-4B	0.020	N	1.3	107.7	71.7	22.5	15.6	15.6	2.0	>20.0	5.0	0.160	0.120

marginal as a result of the somewhat larger than optimum grain size for this gauge.

All the pieces of Heat 3870 were fabricated from 0.102 cm.(0.040 in.) to 0.051 cm.(0.020 in.) utilizing average reductions per pass of less than 2 percent. In all except one instance, 3870-3B, room temperature tensile and yield strengths were found to be less than the Fansteel specification requirements. In addition, elevated temperature tensile strengths were marginal in all instances except one, 3870-3B, which failed specified requirements. As indicated in Table 9, grain sizes, both micro and macro were found to increase as reductions decreased resulting in quite huge grains and consequently poor bend radii in all cases.

In conclusion, it may be hypothesized that 0.102 cm.(0.040 in.) thick TDNiCr sheet can be pack rolled to gauges of 0.051 cm.(0.020 in.) or greater in thickness utilizing average reductions per pass of greater than 4.7 percent, but not exceeding 6.0 percent in order to attain an optimized microstructure yielding satisfactory mechanical properties.

Table 10 summarizes the results of mechanical property testing and the metallographical evaluation carried out on Heats 3869 and 3870 at a finish gauge of 0.025 cm. (0.010 in.).

As indicated by the data included in Table 10, it may be noted that all of the material identified as 3870 contained marginal to poor tensile and yield strengths at both ambient and elevated temperatures. Further, minimum bend results are also margainal to failing. All of these poor properties are caused by the excessively large grained structure typical of this material. Again, referring to Table 9, it may be noted that this same material contained large grains at the 0.051 cm.(0.020 in.) thickness. Therefore, it is surmised that average reductions per pass of less than 2.3 percent during fabrication from 0.051 cm. to 0.025 cm. (0.020 in. to 0.010 in.) was insufficient to enhance the microstructure and thus the mechanical properties. On the other hand, all of the material from Heat 3869 at the 0.025 cm. (0.010 in.) thickness was found to satisfy specification requirements. It should be noted, however, that the eight sheets of 3869-1 and 3869-2 had macro grain sizes slightly smaller than the eight sheets of 3869-3 and 3969-4. This difference was most likely caused by the light average reductions per pass employed during fabrication from the intermediate plate stage to the 0.102 cm.(0.040 in.) thickness. Reductions per pass during subsequent fabrication were apparently heavy enough to inhibit excessive grain size increase but insufficient to enhance the microstructure and yield improved properties. This phenomenon is evidenced in sheets 3869-3 and -4 as compared to 3869-1 and -2 by the somewhat lower room temperature yield strengths normally found to be associated with larger grained material. Therefore, it was concluded that average reductions per pass of 6.2 to 7.3 percent during fabrication from 0.051 cm. to

TABLE 10

MECHANICAL PROPERTIES AND METALLOGRAPHY RESULTS OF 0.025 cm. TDNiCr SHEET

Heat No.	Gauge cm.	Width	Ave.% Red. Per Pass	Room Temperature			1093° C			1093° C Stress Rupture Hrs. @ 31.0 MN/m^2	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size cm.
				UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.				
3869-1A	0.025	W	6.2	745.5	503.7	20.0	126.8	126.8	-	>20.0	1.5	0.092	0.152
3869-1A	0.025	W		776.0	528.5	17.7	124.0	124.0	2.5	>20.0	3.0	0.117	0.152
3869-1B	0.025	W		738.6	498.3	19.5	152.9	152.9	2.0	>20.0	3.0	0.100	0.152
3869-1B	0.025	W		773.9	538.8	19.6	103.4	103.4	-	>20.0	3.0	0.113	0.152
3869-2A	0.025	N	7.3	752.4	511.3	19.7	139.8	139.8	2.5	>20.0	3.0	0.127	0.178
3869-2A	0.025	N		748.3	514.7	19.7	166.0	166.0	2.0	>20.0	3.0	0.113	0.152
3869-2B	0.025	N		750.3	494.7	22.5	152.9	152.9	2.0	>20.0	3.0	0.086	0.152
3869-2B	0.025	N		758.6	518.1	22.5	145.3	145.3	2.4	>20.0	3.0	0.091	0.152
3869-3A	0.025	W	5.6	743.4	502.5	19.7	150.1	150.1	2.5	>20.0	3.0	0.150	0.152
3869-3A	0.025	W		742.7	491.9	20.0	161.2	161.2	2.5	>20.0	3.0	0.205	0.178
3869-3B	0.025	W		753.8	516.1	20.5	153.8	153.8	2.5	>20.0	3.0	0.194	0.152
3869-3B	0.025	W		706.4	449.2	22.5	142.6	142.6	2.5	>20.0	3.0	0.136	0.203
3869-4A	0.025	N	6.3	729.7	468.5	23.0	115.8	115.8	2.5	>20.0	3.0	0.112	0.229
3869-4A	0.025	N		726.9	490.6	22.5	140.5	140.5	1.5	>20.0	3.0	0.172	0.229
3869-4B	0.025	N		715.9	489.5	20.5	124.0	119.9	2.5	>20.0	3.0	0.103	0.152
3869-4B	0.025	N		744.8	503.0	20.0	141.2	141.2	2.0	>20.0	3.0	0.093	0.152

TABLE 10

MECHANICAL PROPERTIES AND METALLOGRAPHY RESULTS OF 0.010 in. TDNiCr SHEET

Heat No.	Gauge in.	Width	Ave.% Red. Per Pass	Room Temperature			2000° F			2000° F Stress Rupture Hrs. @ 4.5 ksi	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size in.
				UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.				
3869-1A	0.010	W	6.2	108.2	73.1	20.0	18.4	18.4	-	>20.0	1.5	0.092	0.060
3869-1A	0.010	W		112.6	76.7	17.7	18.0	18.0	2.5	>20.0	3.0	0.117	0.060
3869-1B	0.010	W		107.2	72.3	19.5	21.9	21.9	2.0	>20.0	3.0	0.100	0.060
3869-1B	0.010	W		112.3	78.2	19.6	15.0	15.0	-	>20.0	3.0	0.113	0.060
3869-2A	0.010	N	7.3	109.2	74.2	19.7	20.0	20.0	2.5	>20.0	3.0	0.127	0.070
3869-2A	0.010	N		108.6	74.7	19.7	23.8	23.8	2.0	>20.0	3.0	0.113	0.060
3869-2B	0.010	N		108.9	71.8	22.5	21.9	21.9	2.0	>20.0	3.0	0.086	0.060
3869-2B	0.010	N		110.1	75.2	22.5	20.8	20.8	2.4	>20.0	3.0	0.091	0.060
3869-3A	0.010	W	5.6	107.9	72.9	19.7	21.5	21.5	2.5	>20.0	3.0	0.150	0.060
3869-3A	0.010	W		107.8	71.4	20.0	23.1	23.1	2.5	>20.0	3.0	0.205	0.070
3869-3B	0.010	W		109.4	74.9	20.5	22.0	22.0	2.5	>20.0	3.0	0.194	0.060
3869-3B	0.010	W		102.5	65.2	22.5	20.4	20.4	2.5	>20.0	3.0	0.136	0.080
3869-4A	0.010	N	6.3	105.9	68.0	23.0	16.8	16.8	2.5	>20.0	3.0	0.112	0.090
3869-4A	0.010	N		105.5	71.2	22.5	20.1	20.1	1.5	>20.0	3.0	0.172	0.090
3869-4B	0.010	N		103.9	69.6	20.5	18.0	17.4	2.5	>20.0	3.0	0.103	0.060
3869-4B	0.010	N		108.1	73.0	20.0	20.2	20.2	2.0	>20.0	3.0	0.093	0.060

TABLE 10 (CONT'D.)

MECHANICAL PROPERTIES AND METALLOGRAPHY RESULTS OF 0.025 cm. TDNiCr SHEET

Heat No.	Gauge cm.	Width	Ave.% Red. Per Pass	Room Temperature UTS MN/m²	Room Temperature YS MN/m²	Room Temperature % Elong.	1093° C UTS MN/m²	1093° C YS MN/m²	1093° C % Elong.	1093° C Stress Rupture Hrs. @ 31.0 MN/m²	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size cm.
3870-1A	0.025	W	2.3	700.0	490.6	20.0	113.0	113.0	-	>20.0	3.0	0.180	0.380
3870-1A	0.025	W		709.2	507.8	17.5	84.9	84.9	2.5	>20.0	3.0	0.180	0.380
3870-1B	0.025	W		707.1	490.6	22.0	106.8	106.8	-	>20.0	3.0	0.180	0.380
3870-1B	0.025	W		660.1	464.4	22.0	108.2	108.2	1.5	>20.0	3.0	0.180	0.380
3870-2A	0.025	N	2.0	703.1	497.0	17.5	113.0	113.0	1.5	>20.0	3.0	0.180	0.380
3870-2A	0.025	N		686.9	494.0	20.5	113.0	113.0	-	>20.0	4.0	0.180	0.380
3870-2B	0.025	N		707.1	495.4	21.9	105.4	105.4	1.5	>20.0	3.0	0.180	0.380
3870-2B	0.025	N		700.7	485.1	22.5	105.4	105.4	1.5	>20.0	3.0	0.180	0.380
3870-3A	0.025	W	1.0	689.0	488.5	19.7	108.9	108.9	2.5	>20.0	1.5	0.180	0.380
3870-3A	0.025	W		669.0	456.8	25.2	114.4	114.4	-	>20.0	3.0	0.180	0.380
3870-3B	0.025	W		686.9	452.3	22.5	108.9	108.9	-	>20.0	3.0	0.180	0.380
3870-3B	0.025	W		668.3	469.9	22.5	79.2	79.2	2.5		4.0	0.180	0.380
3870-4A	0.025	N	1.0	668.3	472.7	19.9	110.2	110.2	2.4	>20.0	3.0	0.180	0.380
3870-4A	0.025	N		713.8	500.4	22.5	110.9	110.9	2.0		4.0	0.180	0.380
3870-4B	0.025	N		667.0	466.5	23.0	101.3	101.3	-		3.0	0.180	0.380
3870-4B	0.025	N		682.1	446.5	24.0	73.7	73.7	-		3.0	0.180	0.380

TABLE 10 (CONT'D.)

MECHANICAL PROPERTIES AND METALLOGRAPHY RESULTS OF 0.010 in. TDNiCr SHEET

				Room Temperature			2000° F						
Heat No.	Gauge in.	Width	Ave.% Red. Per Pass	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	2000° F Stress Rupture Hrs. @ 4.5 ksi	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size in.
3870-1A	0.010	W	2.3	101.6	71.2	20.0	16.4	16.4	-	>20.0	3.0	0.180	0.150
3870-1A	0.010	W		102.9	73.7	17.5	12.3	12.3	2.5	>20.0	3.0		
3870-1B	0.010	W		102.6	71.2	22.0	15.5	15.5	-	>20.0	3.0		
3870-1B	0.010	W		95.8	67.4	22.0	15.7	15.7	1.5	>20.0	3.0		
3870-2A	0.010	N	2.0	102.0	72.1	17.5	16.4	16.4	1.5	>20.0	3.0	0.180	0.150
3870-2A	0.010	N		99.7	71.7	20.5	16.4	16.4	-	>20.0	4.0		
3870-2B	0.010	N		102.6	71.9	21.9	15.3	15.3	1.5	>20.0	3.0		
3870-2B	0.010	N		101.7	70.4	22.5	15.3	15.3	1.5	>20.0	3.0		
3870-3A	0.010	W	1.0	100.0	70.9	19.7	15.8	15.8	2.5	>20.0	1.5	0.180	0.150
3870-3A	0.010	W		97.1	66.3	25.2	16.6	16.6	-	>20.0	3.0		
3870-3B	0.010	W		99.7	65.6	22.5	15.8	15.8	-	>20.0	3.0		
3870-3B	0.010	W		97.0	68.2	22.5	11.5	11.5	2.5		4.0		
3870-4A	0.010	N	1.0	97.0	68.6	19.9	16.0	16.0	2.4	>20.0	3.0	0.180	0.150
3870-4A	0.010	N		103.6	72.6	22.5	16.1	16.1	2.0		4.0		
3870-4B	0.010	N		96.8	67.7	23.0	14.7	14.7	-		3.0		
3870-4B	0.010	N		99.0	64.8	24.0	10.7	10.7	-		3.0		

0.025 cm (0.020 in. to 0.010 in.) are of the proper magnitude to yield satisfactory mechanical property requirements.

In order to verify the results obtained from the reduction-grain size investigation, Heat 3874 which had been processed to intermediate gauge plate by standard processing techniques was prepared for rolling to a finish gauge of 0.102 cm.(0.040 in.). The plate was chemically cleaned, shear trimmed and cut into two pieces identified as 3874-1 and 3874-2. As indicated in Table 11, piece 3874-1 received an average reduction per pass of 7.5 percent while piece 3874-2 received an average reduction of 6.9 percent. The resulting average micro grain sizes for these pieces were found to be 0.057 and 0.038 mm respectively. Since these grain sizes compared very favorably to those attained on Heat 3869-2 and the mechanical properties were also found to be quite typical for 0.102 cm.(0.040 in.) sheet as indicated in Table 11, the process established utilizing Heat 3869-2 for the fabrication of 0.102 cm.(0.040 in.) sheet was reproduced on Heat 3874.

Heats 3876 and 3917-3 were also fabricated to 0.102 cm.(0.040 in.) sheet utilizing 6 to 8 percent average reductions per pass. Again, optimized average micro grain sizes of 0.040 to 0.044 mm were obtained as shown in Table 11, however, all four pieces of Heat 3876 and the one piece of Heat 3917-3 failed to satisfy minimal stress rupture requirements. The cause of failure was believed to be due to the apparently smaller surface grains as indicated by a macro grain size of 0.051 cm.(0.020 in.) rather than the 0.076 cm. (0.030 in.) typical size of Heats 3874-1, 2 and 3869-2.

TABLE 11

ROLL PASS REDUCTIONS AND MECHANICAL PROPERTIES FOR 0.102 cm. TDNiCr SHEET

Heat No.	Gauge cm.	Ave.% Red. Per Pass	Room Temperature			1093° C			1093° C Stress Rupture Hrs. @ 37.9 MN/m²	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size cm.
			UTS MN/m²	YS MN/m²	% Elong.	UTS MN/m²	YS MN/m²	% Elong.				
3874-1	0.102	7.5	860.5	596.0	22.0	126.8	126.8	1.5	>20.0	3.0	0.057	0.076
3874-2	0.102	6.9	857.0	602.2	20.0	125.4	125.4	1.5	>20.0	3.0	0.038	0.076
3876-1A	0.102	6.7							2.7	3.0	0.040	0.051
3876-1B	0.102	6.7							5.2	3.0	0.041	0.051
3876-2A	0.102	6.5							12.6	3.0	0.042	0.051
3876-2B	0.102	6.5							8.5	3.0	0.042	0.051
3917-3	0.102	6.9							2.1	3.0	0.044	0.051

ROLL PASS REDUCTIONS AND MECHANICAL PROPERTIES FOR 0.040 in. TDNiCr SHEET

Heat No.	Gauge in.	Ave.% Red. Per Pass	Room Temperature			2000° F			2000° F Stress Rupture Hrs. @ 5.5 ksi	Min. Bend	Ave. Micro Grain Size mm	Macro Grain Size in.
			UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.				
3874-1	0.040	7.5	124.6	86.5	22.0	18.4	18.4	1.5	>20.0	3.0	0.057	0.03
3874-2	0.040	6.9	124.1	87.4	20.0	18.2	18.2	1.5	>20.0	3.0	0.038	0.03
3876-1A	0.040	6.7							2.7	3.0	0.040	0.02
3876-1B	0.040	6.7							5.2	3.0	0.041	0.02
3876-2A	0.040	6.5							12.6	3.0	0.042	0.02
3876-2B	0.040	6.5							8.5	3.0	0.042	0.02
3917-3	0.040	6.9							2.1	3.0	0.044	0.02

Isolated heavy or light reductions in the last few roll passes before attaining final gauge are suspected of establishing surface grain size. To minimize this effect, an average roll pass reduction of 5 to 6 percent instead of 6 to 8 percent was evaluated during subsequent rolling of sheet to 0.102 cm.(0.040 in.) in an effort to yield slightly larger average grain sizes to improve stress rupture strengths without promoting a loss in ductility as reflected by bend testing.

Heats 3918, 3919 and 3920 were fabricated to 0.102 cm.(0.040 in.) sheet utilizing an average roll pass reduction of 5 to 6 percent. As indicated in Table 12 , the average micro grain size yielded was comparable to those obtained on Heats 3874, 3876, 3917-3 and 3869-2. In addition, the macro grain size was found to be 0.076 cm.(0.030 in.) for all three heats. Since mechanical properties also satisfied requirements in all cases, Table 13, it was concluded that an average roll pass reduction of 5 to 6 percent for the fabrication of TDNiCr sheet from intermediate gauge plate to a gauge of 0.102 cm.(0.040 in.) was optimum.

Material from Heat 3876 which was unsatisfactory for use at 0.102 cm.(0.040 in.) gauge because of failure to satisfy stress rupture requirements was prepared for further fabrication to 0.051 cm.(0.020 in.). The unrecrystallized sheet was trimmed, sized and assembled into aluminized plates in a two sheet per pack configuration. Subsequently, the pack was fabricated at 760° C(1400° F) to a finish gauge of

TABLE 12

GRAIN SIZE VS. GAUGE FOR TDNiCr PRODUCT SHEET

Heat No.	Gauge cm.	Gauge in.	Average Micro Grain Size mm	Average Macro Grain Size cm.	Average Macro Grain Size in.
3918	0.102	0.040	0.040	0.076	0.030
3919	0.102	0.040	0.042	0.076	0.030
3920	0.102	0.040	0.036	0.076	0.030
3876	0.051	0.020	0.037	0.076	0.030
3873	0.025	0.010	0.066	0.102	0.040
3875	0.025	0.010	0.082	0.150	0.060
3917	0.025	0.010	0.088	0.130	0.050
3918	0.025	0.010	0.110	0.150	0.060
3919	0.025	0.010	0.115	0.180	0.070

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TABLE 13

MECHANICAL PROPERTIES AND CHEMICAL ANALYSIS OF TDNiCr SHEET

Heat	Gauge	Room Temperature			1093° C			Stress Rupture Hrs. at 1093° C		Chemistry Weight %			
No.	cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	31.0 MN/m^2	37.9 MN/m^2	ThO_2	Cr	C	S
3918	0.102	821.3	543.6	23.0	155.2	155.2	2.0		>20	1.86	19.87	0.017	0.0057
3919	0.102	814.4	579.4	20.0	124.7	121.3	2.0		>20	2.07	19.75	0.026	0.0045
3920	0.102	841.2	569.3	22.0	126.1	119.2	2.0		>20	1.97	19.85	0.024	0.0048
3876	0.051	887.4	592.5	20.0	148.8	147.4	2.5		>20	1.96	19.92	0.022	0.0051
3873	0.025	800.6	553.3	17.0	97.1	97.1	2.0	>20		1.98	19.69	0.008	0.0051
3875	0.025	766.2	547.1	16.0	111.6	111.6	3.0	>20		1.94	19.80	0.016	0.0057
3917	0.025	766.9	539.5	15.0	129.5	129.5	2.0	>20		1.95	20.25	0.014	0.0050
3918	0.025	761.3	540.9	18.0	99.9	88.9	2.0	>20		1.86	19.87	0.017	0.0057
3919	0.025	742.1	549.8	17.0	83.6	83.6	2.0	>20		2.07	19.75	0.026	0.0045

TABLE 13

MECHANICAL PROPERTIES AND CHEMICAL ANALYSIS OF TDNiCr SHEET

Heat	Gauge	Room Temperature			2000°F			Stress Rupture Hrs. at 2000°F		Chemistry Weight %			
No.	in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	4.5 ksi	5.5 ksi	ThO_2	Cr	C	S
3918	0.040	119.2	78.9	23.0	22.2	22.2	2.0		>20	1.86	19.87	0.017	0.0057
3919	0.040	118.2	84.1	20.0	18.1	17.6	2.0		>20	2.07	19.75	0.026	0.0045
3920	0.040	121.8	82.6	22.0	18.3	17.3	2.0		>20	1.97	19.85	0.024	0.0048
3876	0.020	128.5	86.0	20.0	21.3	21.1	2.5		>20	1.96	19.92	0.022	0.0051
3873	0.010	116.2	80.3	17.0	14.1	14.1	2.0	>20		1.98	19.69	0.008	0.0051
3875	0.010	111.2	79.4	16.0	16.2	16.2	3.0	>20		1.94	19.80	0.016	0.0057
3917	0.010	111.3	78.3	15.0	18.8	18.8	2.0	>20		1.95	20.25	0.014	0.0050
3918	0.010	110.5	78.5	18.0	14.5	12.0	2.0	>20		1.86	19.87	0.017	0.0057
3919	0.010	107.7	79.8	17.0	12.1	12.1	2.0	>20		2.07	19.75	0.026	0.0045

0.051 cm.(0.020 in.) utilizing average roll pass reductions of 5 to 6 percent as established during the work carried out on Heats 3869 and 3870. This process yielded finished 0.051 cm. (0.020 in.) sheet having an average micro grain size of 0.037 mm and an average macro grain size of 0.076 cm. (0.030 in.) as shown in Table 12. Again referring to Table 11, it may be noted that this material at 0.102 cm.(0.040 in.) contained a very similar average micro grain size, i.e., 0.040 to 0.042 mm, but a finer macro grain size of 0.051 cm.(0.020 in.). It appears that average roll pass reductions of 5 to 6 percent yield sheet having the optimum macro grain size necessary to satisfy mechanical property requirements.

Based upon the grain size-reduction relationship investigation, average roll pass reductions of approximately 6 to 7 percent were believed to be optimum for fabrication of 0.051 cm.(0.020 in.) TDNiCr sheet to 0.025 cm.(0.010 in.) by pack rolling procedures. However, it has been shown that average roll pass reductions of 5 to 6 percent were found to be optimum for rolling both 0.102 cm.(0.040 in.) and 0.051 cm.(0.020 in.) sheet from the intermediate gauge plate stage of processing. For this reason, a similar process was evaluated for the fabrication of 0.025 cm.(0.010 in.) sheet. As indicated in Table 12, sheet processed in this manner contained average micro grain sizes ranging from 0.066 to 0.115 mm and macro grain sizes ranging from 0.102 to 0.180 cm. (0.040 to 0.070 in.). Further, all material had mechanical properties which complied to specification requirements as shown in Table 13. Consequently, it has been shown that utilization of average roll pass reductions of 5 to 6 percent reduction during fabrication of 0.025 to 0.102 cm.(0.010 to 0.040 in.) TDNiCr sheet from intermediate gauge plate yields finished product sheet containing an optimized structure necessary for attaining specified mechanical properties.

b. Alternate Process for 0.025 cm.(0.010 in.) Sheet

Cold rolling of TDNiCr sheet was investigated as an alternate process for producing thin sheet having improved gauge control, surface finish, flatness, and yield. In order to accomplish this objective, a slight sacrifice in mechanical properties was experienced. This was, however, minimized. The key variables which influenced the elevated temperature strength of the material after cold rolling were grain size of the recrystallized warm rolled starting material and total percent cold reduction.

Experiments were conducted on both a laboratory and production scale. The latter was required to determine such parameters as roll crown, pass schedules and gauge control, but was impractical for obtaining variations in percent reductions on a particular sheet.

(1.) Laboratory Investigations

Three heats at 0.102 cm.(0.040 in.) covering a wide range of grain sizes were chosen for laboratory scale investigations. The heat numbers were 3473, 3631 and 3418, having respective grain sizes of 0.035 mm, 0.050 mm and 0.4 to 0.6 mm. These heats have a grain size range considerably greater than the range produced by the standard process.

Heat 3473 was fabricated utilizing a 954° C(1750° F) sinter temperature, a 1010° C(1850° F) roll consolidation temperature, a thoria size control heat treatment of 1177° C(2150° F) in the 2.54 cm.(1.0 in.) slab condition and subsequent 760° C (1400° F) rolling to a gauge of 0.102 cm.(0.040 in.), at which point it was subjected to an 1177° C(2150° F) recrystallization heat treatment.

Heat 3631 was fabricated in an identical manner to Heat 3473 except that it was subjected to a 1093° C(2000° F) heat treatment at the 2.54 cm.(1.0 in.) slab size for thoria size control. This heat is considered to have been fabricated in a manner comparable to the standard process.

Heat 3418 was fabricated in an identical manner to Heats 3473 and 3631 except that it did not receive any thoria size control heat treatment at the 2.54 cm.(1.0 in.) slab size.

All cold rolling studies were carried out on a 4-High rolling mill having 10.2 cm.(4.0 in.) diameter work rolls and 30.5 cm.(12.0 in.) diameter back-up rolls.

Intermediate heat treatments as required were performed by the standard process in the Sunbeam Production Furnace. Final heat treatments on finished gauge material utilized for test purposes were performed by both standard process in the Sunbeam Production Furnace and in a laboratory furnace which consisted of 2 hours at 1177° C(2150° F) without any program heat-up or furnace cool.

Cold rolling of all 0.102 cm.(0.040 in.) recrystallized warm rolled sheet was performed both with and without an intermediate heat treatment. There were early indications that greater than 50% cold work might be required to achieve full recrystallization. Since 50% reduction without an anneal is the maximum amount of work possible on the Schloemann single cluster rolling mill due to sheet breakage, all of the heats were subjected to an intermediate heat treatment after 30% cold rolling. Subsequent rolling was performed to achieve 40 to 70 or 75% total reductions.

Heats 3473 and 3631 were rolled in increments of 5% up to 75% total reduction without an intermediate heat treatment. It was found that full recrystallization did not occur below 40% reduction. A partially recrystallized structure possesses poor bend ductility and is metallurgically unstable. For these reasons, samples were procured from 40% to 70% or 75% cold reduction only for the remaining work.

Transverse samples at each gauge were obtained for determination of mechanical properties and metallographic examination. One set of samples was subjected to a laboratory heat treatment and a duplicate set was subjected to the standard production process heat treatment.

Figures 3 through 8 show the trends in the room temperature strengths for each heat rolled without an intermediate heat treatment. Both plant and lab heat treatments are presented. The ultimate tensile strength and yield strength are well above the values for sheet produced by the standard process of warm rolling and heat treating. It is believed that work hardening is responsible for high room temperature strengths below the point of full recrystallization. When complete recrystallization occurs, a finer grain size is produced which may account for the increase in strength over the starting material. Fully recrystallized strength values are typically 896-1034 MN/m^2 (130-150 ksi) for the room temperature ultimate and 620-793 MN/m^2 (90-115 ksi) for the room temperature yield. All heats showed a slight drop in yield strength when full recrystallization occurred. However, Heats 3473 and 3631 with both the lab and plant heat treatments indicated even higher yield strengths for very large reductions ($\geq$65%). As will be discussed later, the grain size exhibited only a small change for these large reductions. Figures 3 through 8 also present the 1093° C(2000° F) tensile strength as a function of percent reduction for both heat treatments. It is noted that beyond a certain reduction for each heat, the 1093° C(2000° F) tensile strength begins to decrease and that this percent reduction is different for each of the heats examined. Heat 3473 with a 2 hour 1177° C (2150° F) heat treatment began to lose 1093° C(2000° F) strength at about 40 percent cold reduction. Heat 3418 with a similar heat treatment did not begin to lose strength until approximately 50 percent. Even with 75 percent reduction this heat maintained a 1093°C(2000°F) yield strength of 117.0 MN/m^2 (17.0 ksi).

There is some indication on Heat 3418 that the yield strength increases with increasing reduction up to 40% reduction. A tensile value of 200.0 MN/m^2 (29.0 ksi) was obtained for the 40 percent reduction with the plant heat treatment. This strength is far outside the scatter band for warm rolled, recrystallized sheet of this grain size. The other two heats, however, did not indicate this trend; the strength up to recrystallization was approximately constant. Since these heats were not as recrystallization resistant as 3418, the benefits of changes in grain morphology or the introduction of work hardening may have been negated by partial recrystallization. All 1093°C(2000°F) tensile results are, however, above 68.9 MN/m^2 (10.0 ksi).

Minimum bend radius was found to exhibit the greatest changes as a result of cold rolling (see Figures 3 through 8). Minimum bend radius increased almost up to the point where full recrystallization took place. Approximately 5 to 10 percent below recrystallization, bend radius results were scattered; that is, some areas would bend at 3T while others would fracture. However, when full recrystallization was reached, a drastic drop in the minimum bend radius was

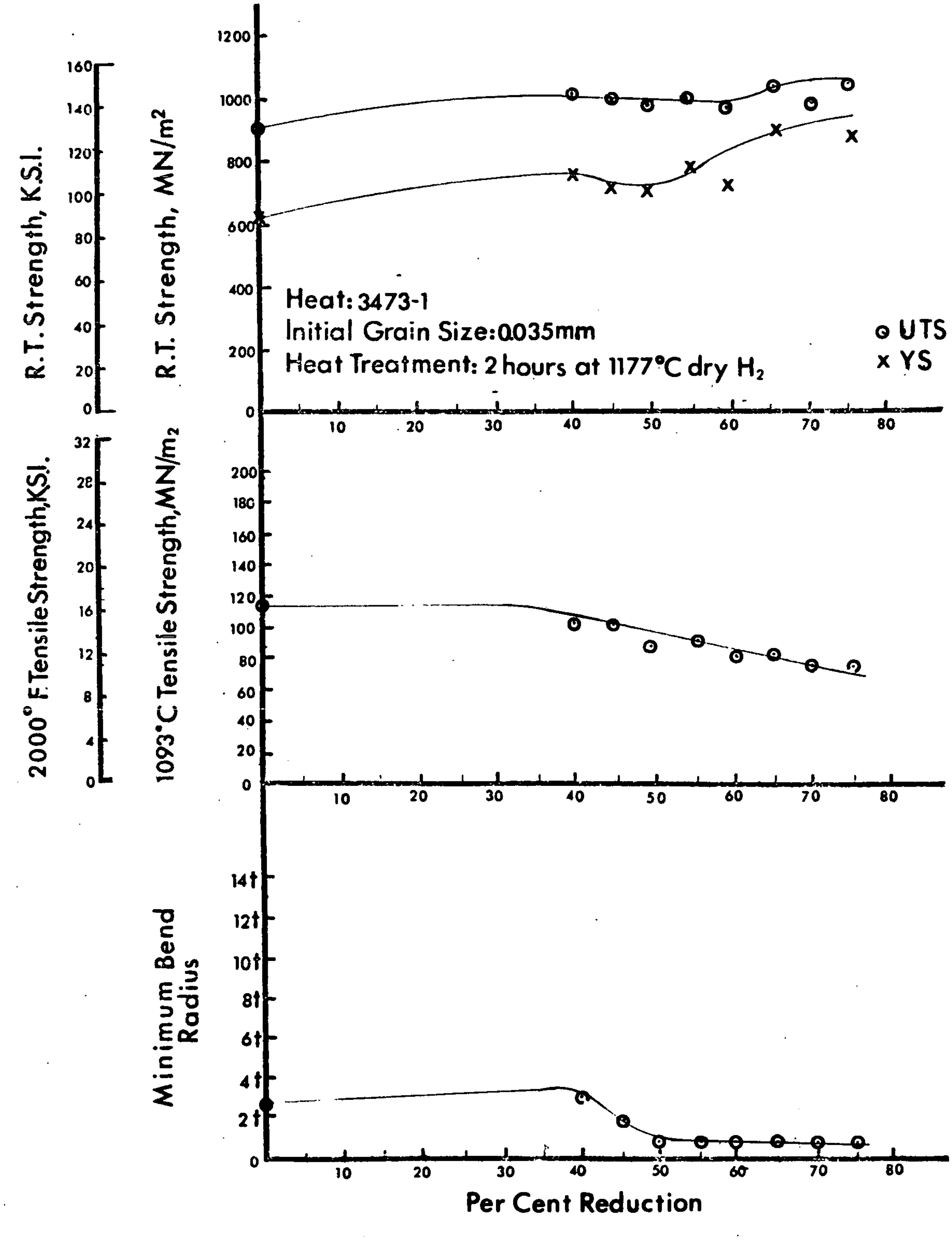


FIGURE 3

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3473

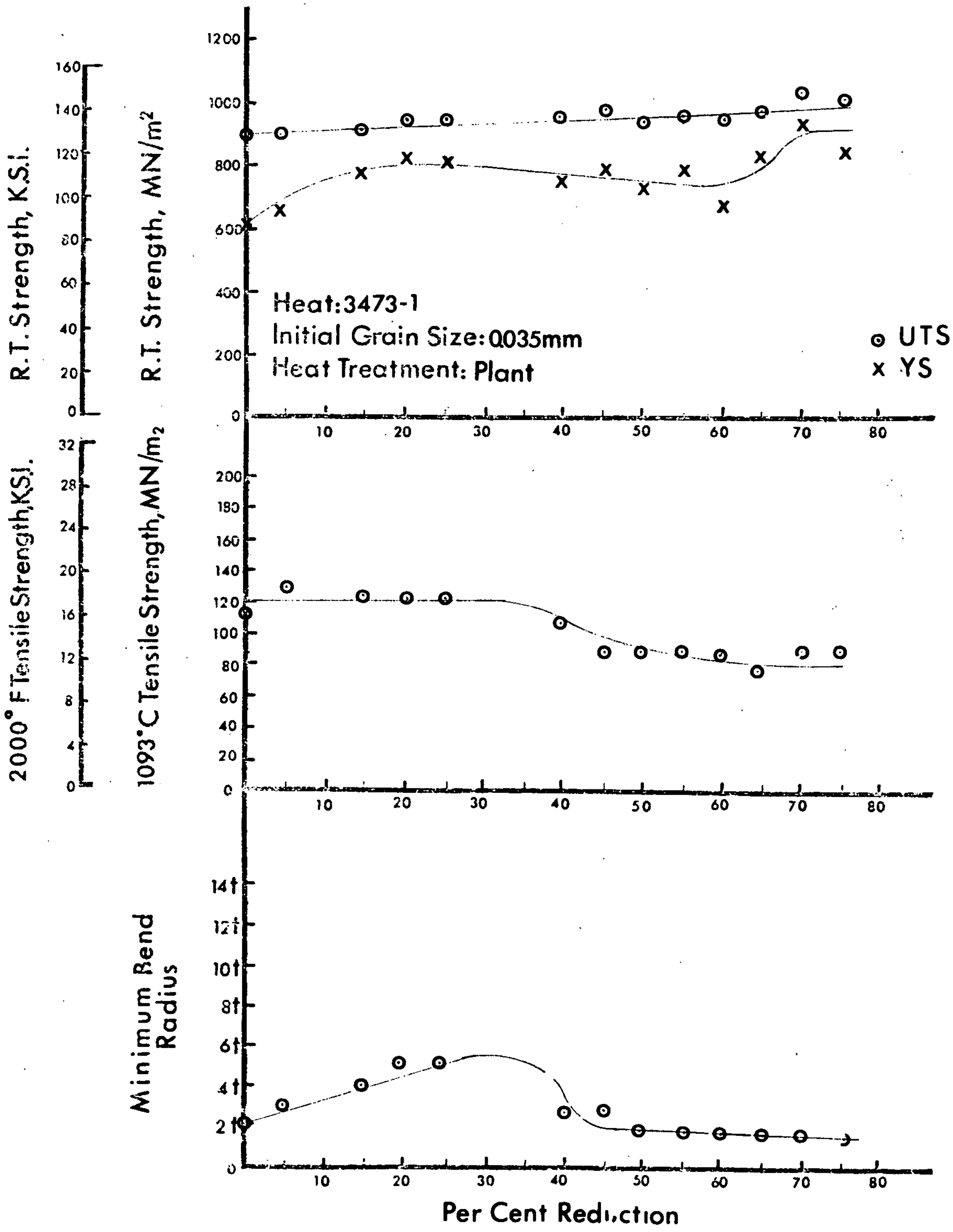


FIGURE 4.

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3473

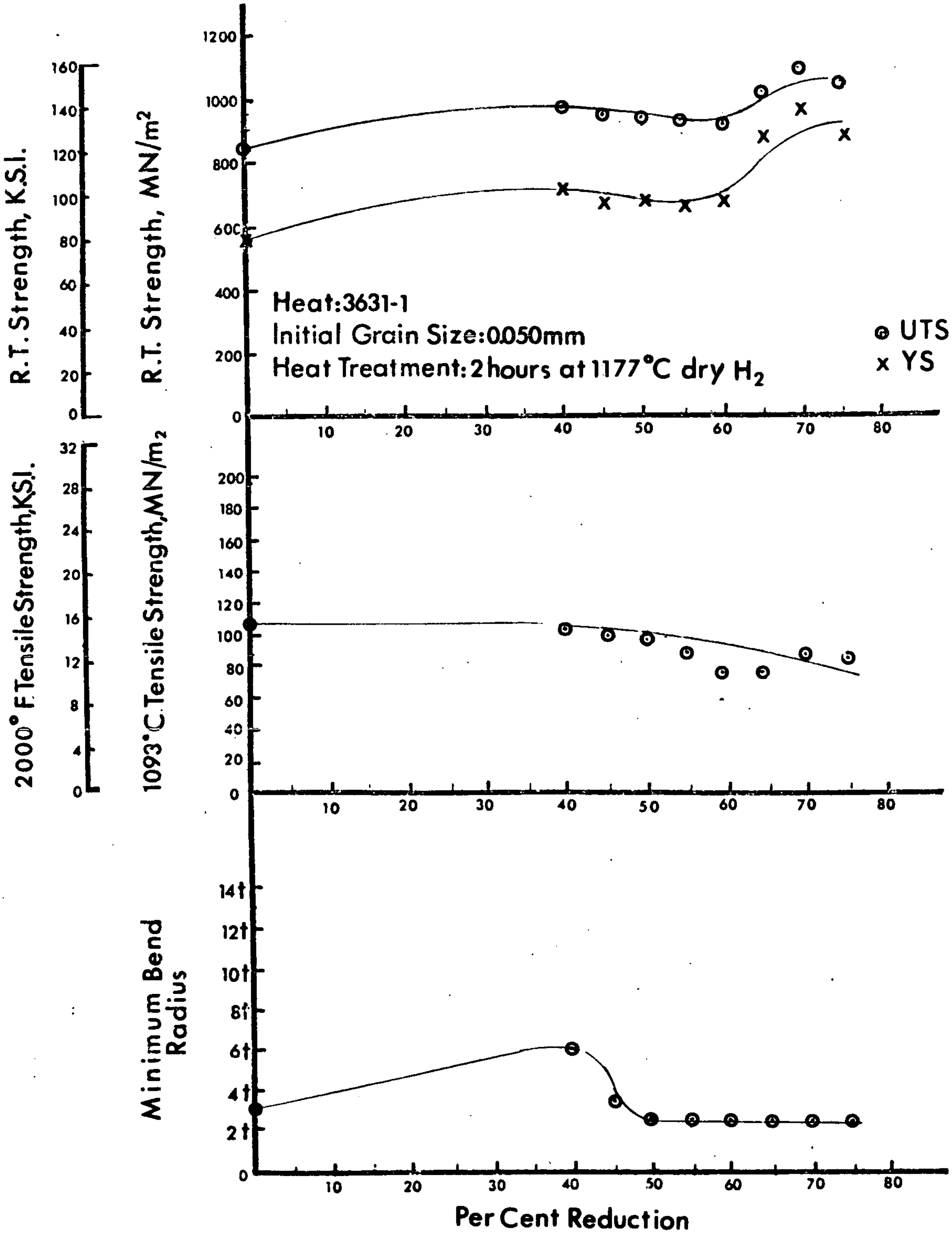


FIGURE 5

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3631

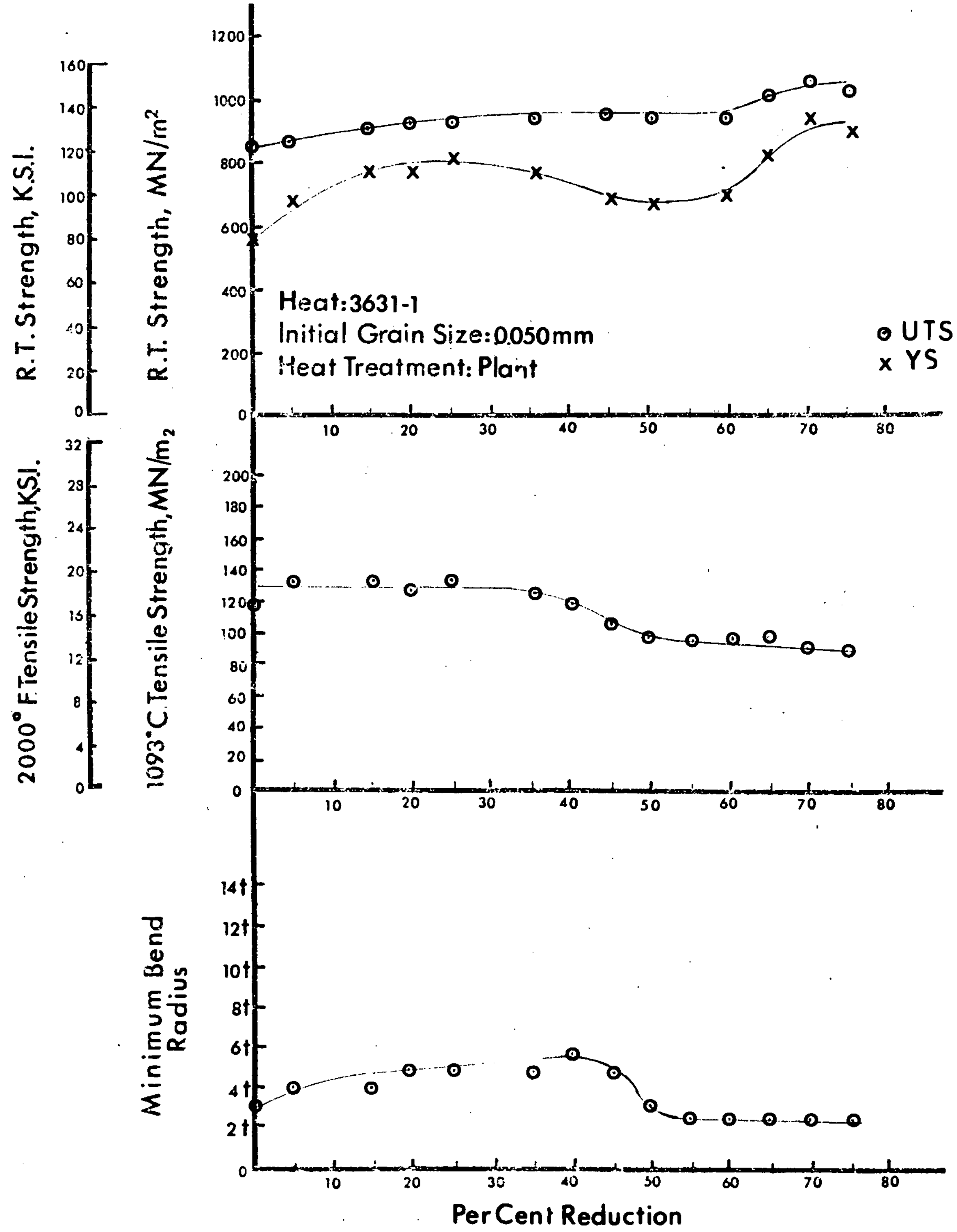


FIGURE 6

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3631

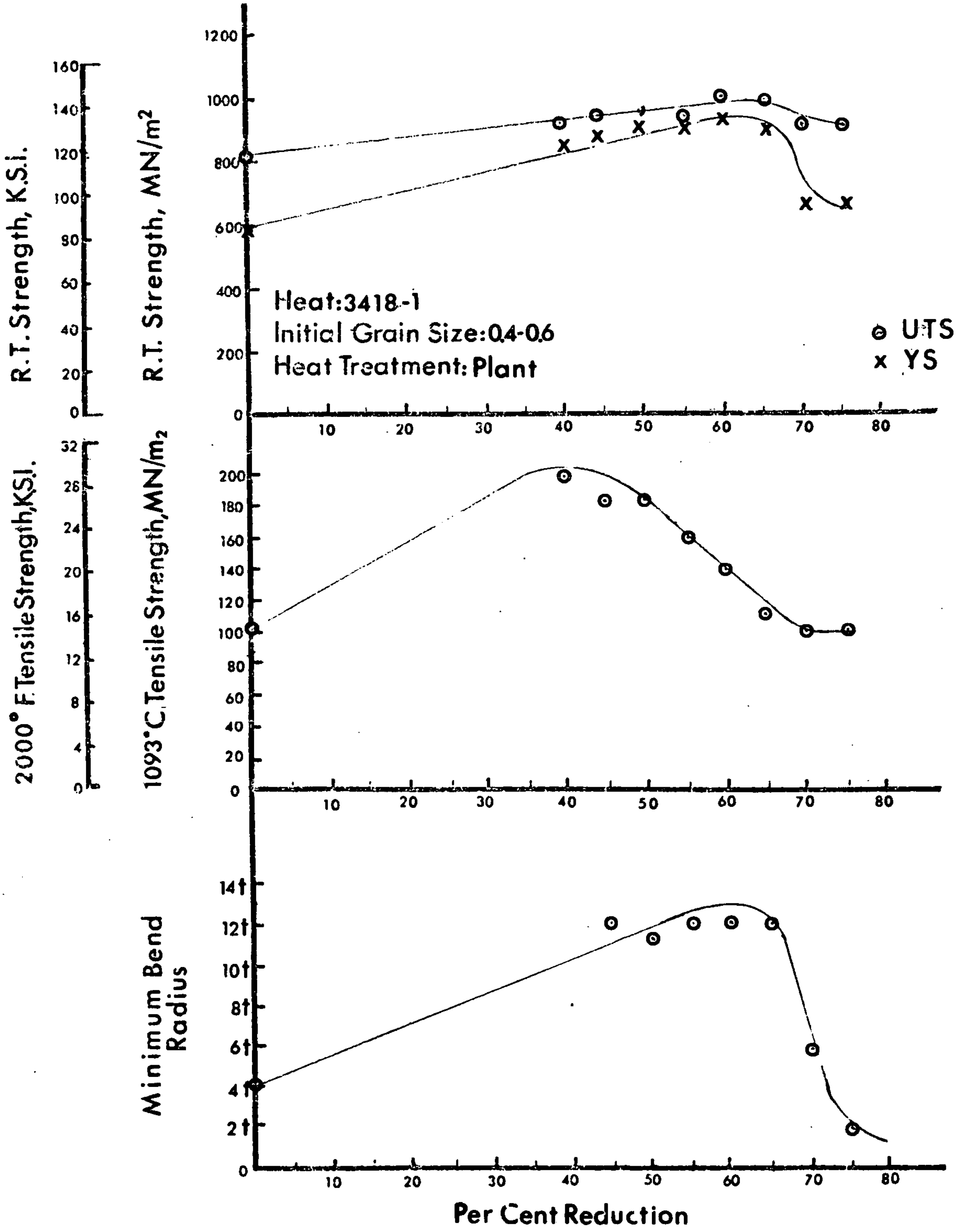


FIGURE 7

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3418

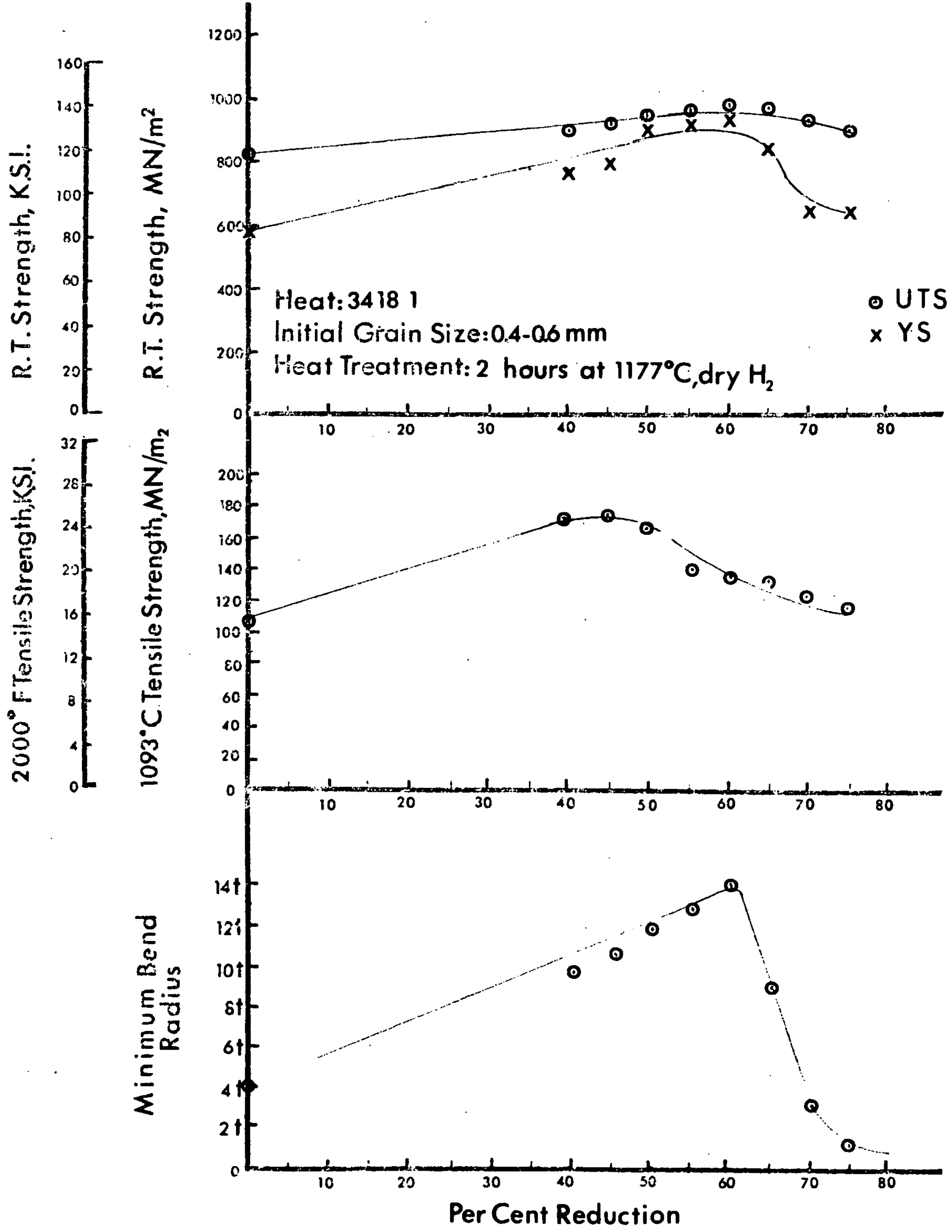


FIGURE 8

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3418

F

encountered. The finer grained Heats 3473 and 3631 with the 2 hour at 1177° C(2150° F) heat treatments fully recrystallized at 40 percent and 50 percent respectively. Heat 3418, however, which had a grain size considerably larger than the other two heats, did not fully recrystallize with this heat treatment until 70 percent reduction was obtained.

Stress rupture tests were conducted at 31.0 MN/m^2 (4.5 ksi) on all of the gauges obtained. These results, along with all other mechanical property test results are included as Appendix A of this report. These data appear to indicate that if the percent cold work is increased by a quantity of greater than 10% beyond the full recrystallization point, stress rupture properties become marginal.

No consistent results were noted for either the room temperature or the 1093° C(2000° F) elongations. The room temperature elongations ranged from 10 percent to 20 percent regardless of the condition of the material tested. The 1093° C(2000° F) elongation remained in the range of 2 to 5 percent.

In conclusion, it appears that for each starting 0.102 cm.(0.040 in.) warm rolled recrystallized grain size, there is an optimum percent cold reduction range that will permit full recrystallization and yield mechanical properties which satisfy specification requirements. The optimum ranges established for Heats 3473, 3631 and 3418 which were fabricated without any intermediate heat treatment are shown in Table 14.

TABLE 14

OPTIMUM COLD REDUCTION RANGE AS A FUNCTION OF 0.102 cm.(0.040 in.) RECRYSTALLIZED SHEET GRAIN SIZES

Heat No.	0.102 cm.(0.040 in.) Grain Size mm	Optimum % Min.	Cold Reduction % Max.
3473	0.035	40	60
3631	0.050	50	>60
3418	0.4 to 0.6	70	>75

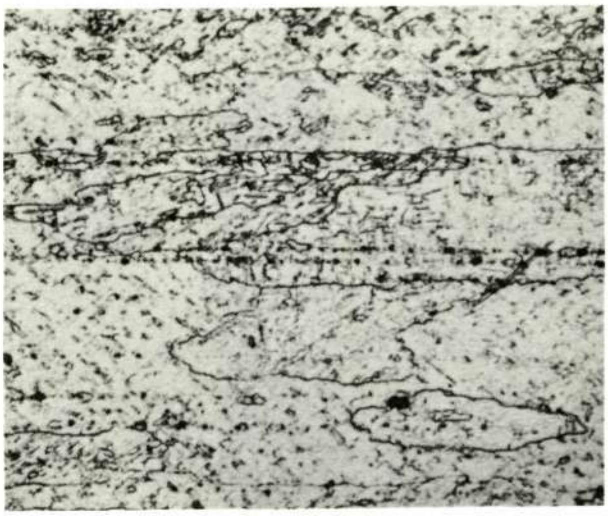
Prior to initiation of metallography, it was found necessary to develop an alternate etching solution to reveal cold worked structures. The procedure utilized is as follows:

1. Solution: 1 gram of oxalic acid dissolved in 100 ml of distilled water.
2. Electrolytically etch with a tantalum sheet cathode at 4 volts D.C. in 4 to 5 second intervals, swab with lactic acid, rinse in warm running water, rinse in methanol, and dry in a hot airstream.

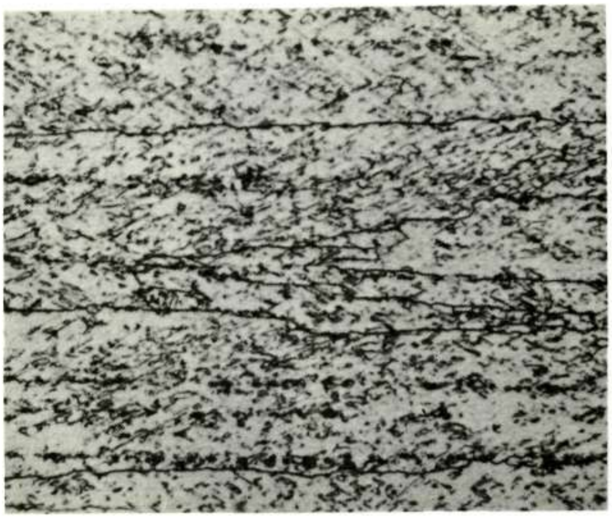
Microstructures were examined at each gauge for each heat. For low percent reductions, the grain thickness was reduced in the longitudinal and transverse directions. In addition, the grain length was increased in the longitudinal direction. Both effects increased the aspect ratio. "Worked" structures still contained easily identifiable grain boundaries, but the angle of intersection of twins was changed by rolling.

Partial recrystallization was observed at approximately 25%, 35% and 50% for Heats 3473, 3631 and 3418 respectively.

Photomicrographs of heat treated samples are shown in Figures 9 and 10 for Heats 3473 and 3418. Since little difference was observed between Heats 3473 and 3631, the latter is not presented. Heat 3473 contains a relatively fine grain size of 0.035 mm at 0.102 cm.(0.040 in.), the



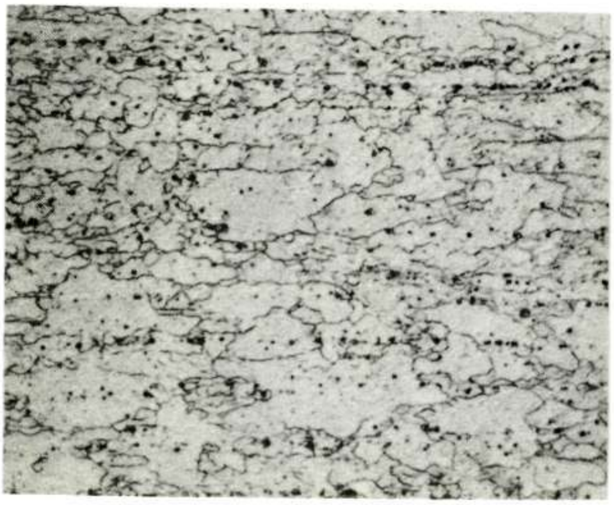
0% Cold Reduction
0.102 cm.(0.040 in.)
500X
Long.
Grain Size = 0.035 mm



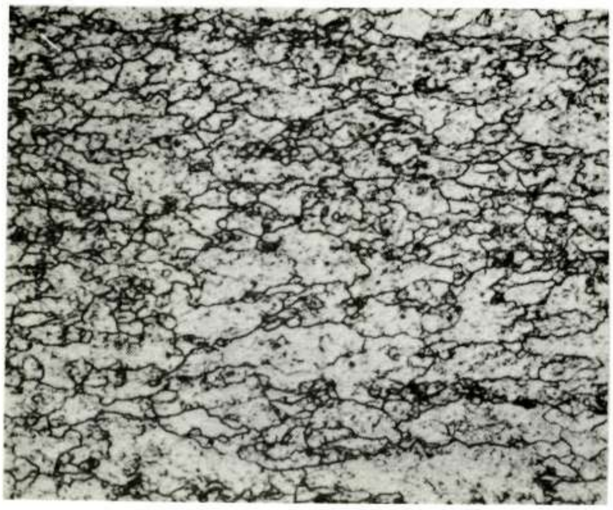
25% Cold Reduction
0.076 cm.(0.030 in.)
500X
Long.
No Recrystallization

FIGURE 9e

MICROSTRUCTURES OF HEAT 3473



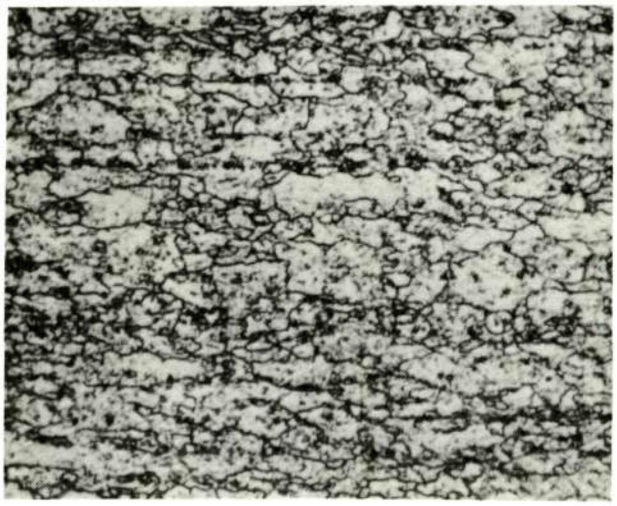
40% Cold Reduction
0.061 cm.(0.024 in.)
500X
Long.
Grain Size = 0.009 mm



45% Cold Reduction
0.056 cm.(0.022 in.)
500X
Long.
Grain Size = 0.0075 mm

FIGURE 9b

MICROSTRUCTURES OF HEAT 3473

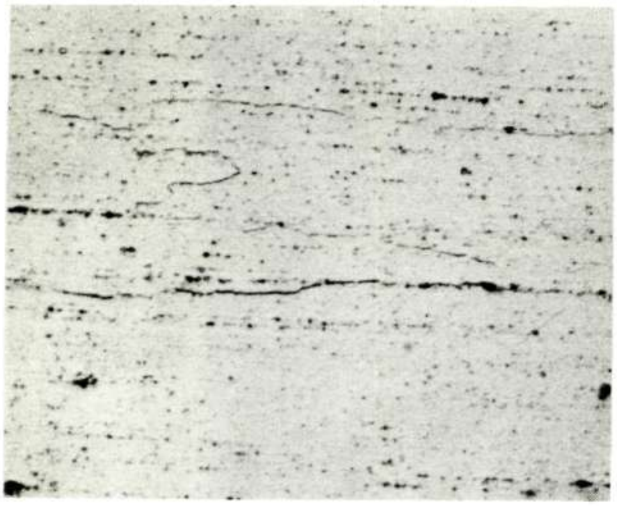


55% Cold Reduction
0.045 cm.(0.018 in.)
500X
Long.
Grain Size = 0.0075 mm

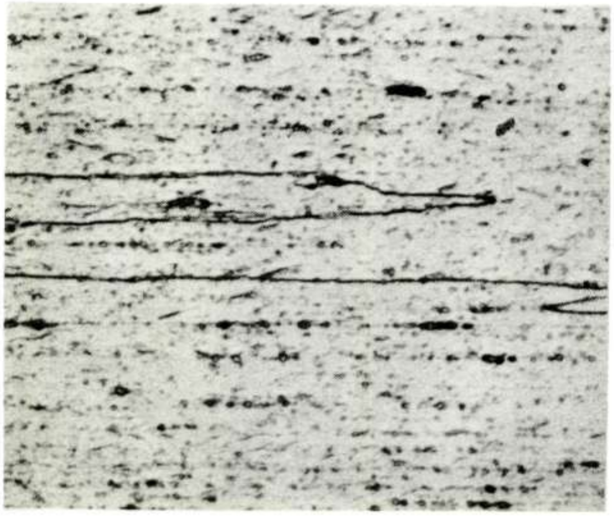
75% Cold Reduction
0.025 cm.(0.010 in.)
500X
Long.
Grain Size = 0.0075 mm

FIGURE 9c

MICROSTRUCTURES OF HEAT 3473



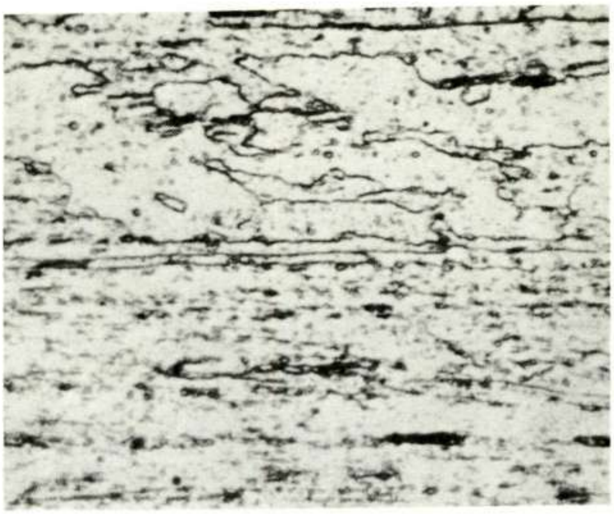
0% Cold Reduction
0.102 cm.(0.040 in.)
500X
Long.
Grain Size=0.4-0.6 mm



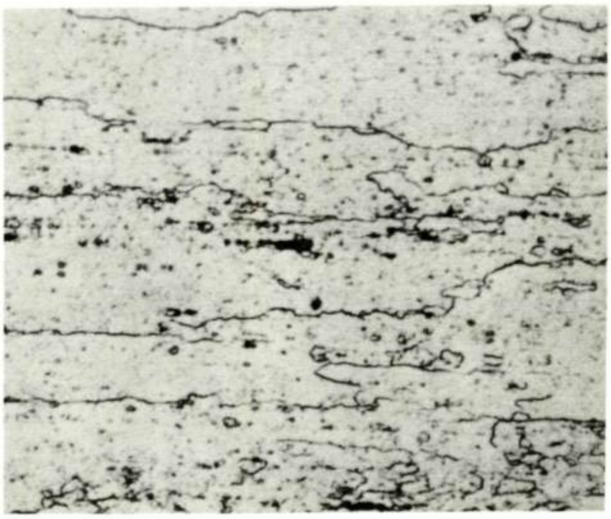
45% Cold Reduction
0.056 cm.(0.022 in.)
500X
Long.

FIGURE 10a

MICROSTRUCTURES OF HEAT 3418



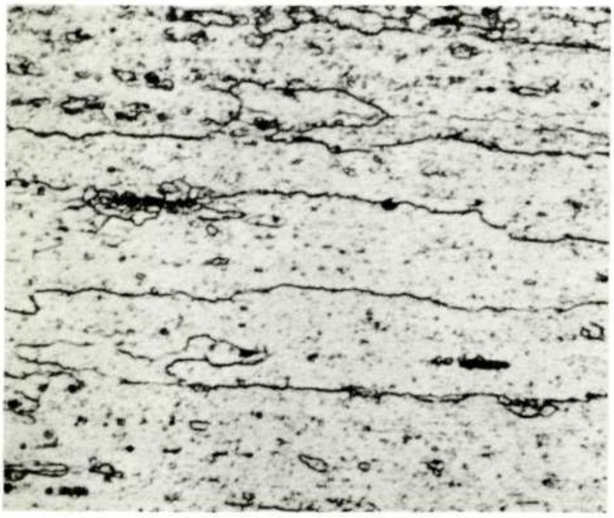
65% Cold Reduction
0.036 cm.(0.014 in.)
500X
Long.



70% Cold Reduction
0.031 cm.(0.012 in.)
500X
Long.
Grain Size = 0.023 mm

FIGURE 10b

MICROSTRUCTURES OF HEAT 3418



75% Cold Reduction
0.025 cm.(0.010 in.)
500X
Long.
Grain Size = 0.023 mm

FIGURE 10c

MICROSTRUCTURES OF HEAT 3418

starting gauge. At 0.076 cm.(0.030 in.), some lengthening of the grains is apparent. Full recrystallization to a grain size of 0.009 mm occurred at 40% reduction. Further rolling produced a grain size of 0.0075 mm for all other gauges. Heat 3418, with an initial grain size of 0.4-0.6 mm, did not fully recrystallize until 70% reduction was reached. The grain size at 70 and 75% reductions was 0.023 mm, several times greater than for Heat 3473. As was shown earlier, this difference was large enough to produce significant differences in mechanical properties.

A duplex grain structure is apparent in most specimens. However, since the dispersion of fine and coarse grains is random and over very small areas, deleterious effects were not expected. If a duplex structure existed with fine grain areas across the thickness of a sheet, this would be of concern, but examination of the photomicrography reveals that this is not the case.

It has been demonstrated that cold reductions in excess of 50% cannot be achieved on the production mill without intermediate heat treatments. Sheet segments, warm rolled to 0.102 cm.(0.040 in.) and recrystallized, from Heats 3473, 3631 and 3418 were cold roll fabricated on the 4-High mill a total reduction of 30%. These were heat treated by standard production process, and cold rolled to various gauges between 40% and 70% or 75%. Samples were obtained at each gauge from each heat and subjected to both a laboratory and production type final heat treatment. The results of mechanical property determination on these samples are included as Appendix A of this report.

Figures 11 through 16 show the trends of mechanical properties of each heat cold rolled utilizing an intermediate heat treatment after the first 30% cold work. Final heat treatments representing laboratory and production processing are presented.

These results indicate that mechanical properties duplicate those obtained in cold rolling directly to gauge. Room temperature strengths remain well above values for standard TDNiCr, and the 1093° C(2000° F) tensile strength exhibits the same slow decline. The point of full recrystallization, however, was increased by an average of 5%. An examination of the bend radius results confirms this fact.

The optimum cold work range within which all properties are met was apparently reduced to 10% on Heat 3473 by utilizing an intermediate heat treatment. It is probable that some recrystallization occurred after the intermediate heat treatment. Fine grains are thought to have re-recrystallized to even finer grains after rolling to final gauge. However, since it was demonstrated that this heat could be rolled easily on the production mill, without an intermediate anneal, this was of no concern. Heat 3631, exhibited little change due to heat treatment. The percent reduction for full recrystallization of Heat 3418 was increased by only 5%.

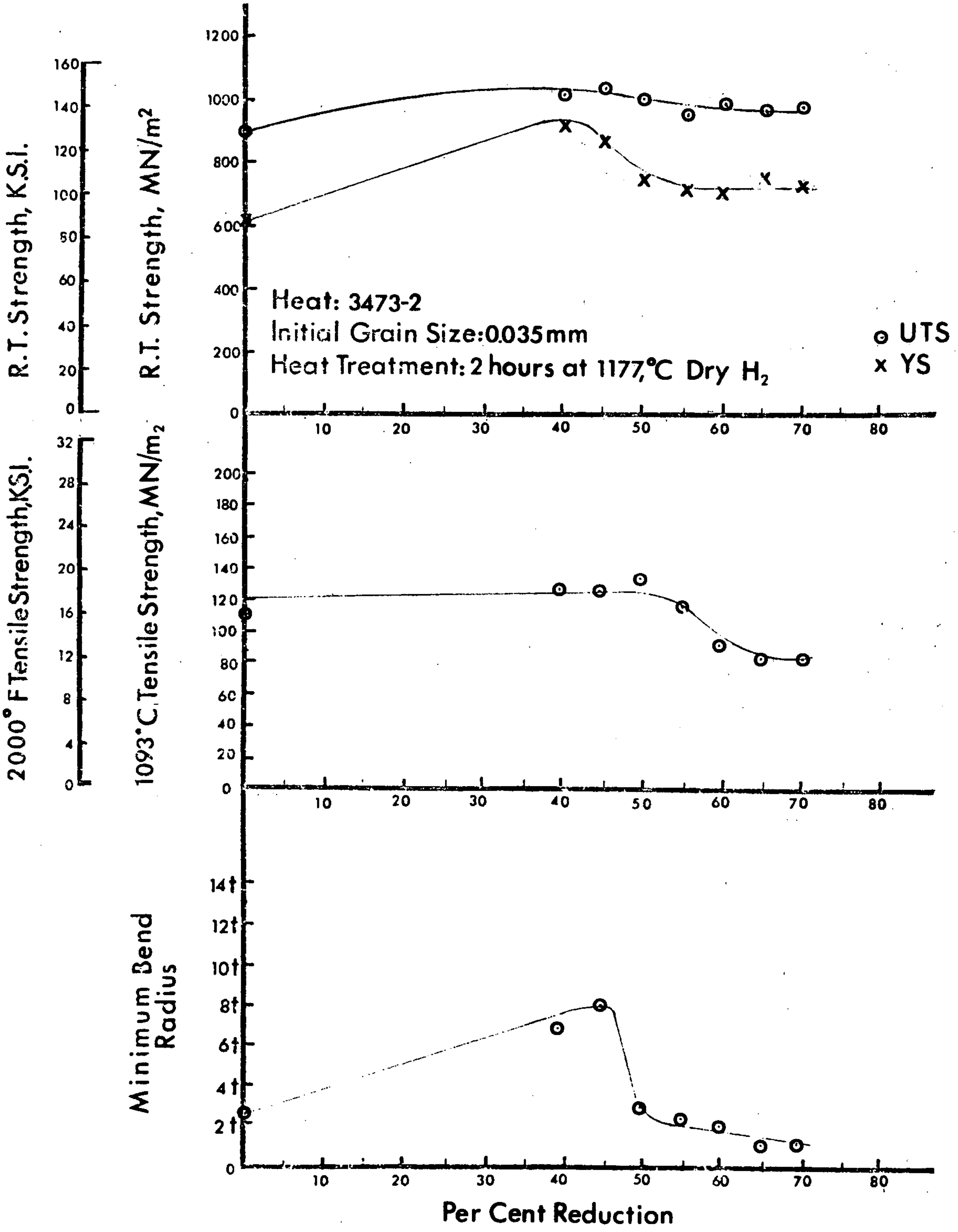


FIGURE 11

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3473

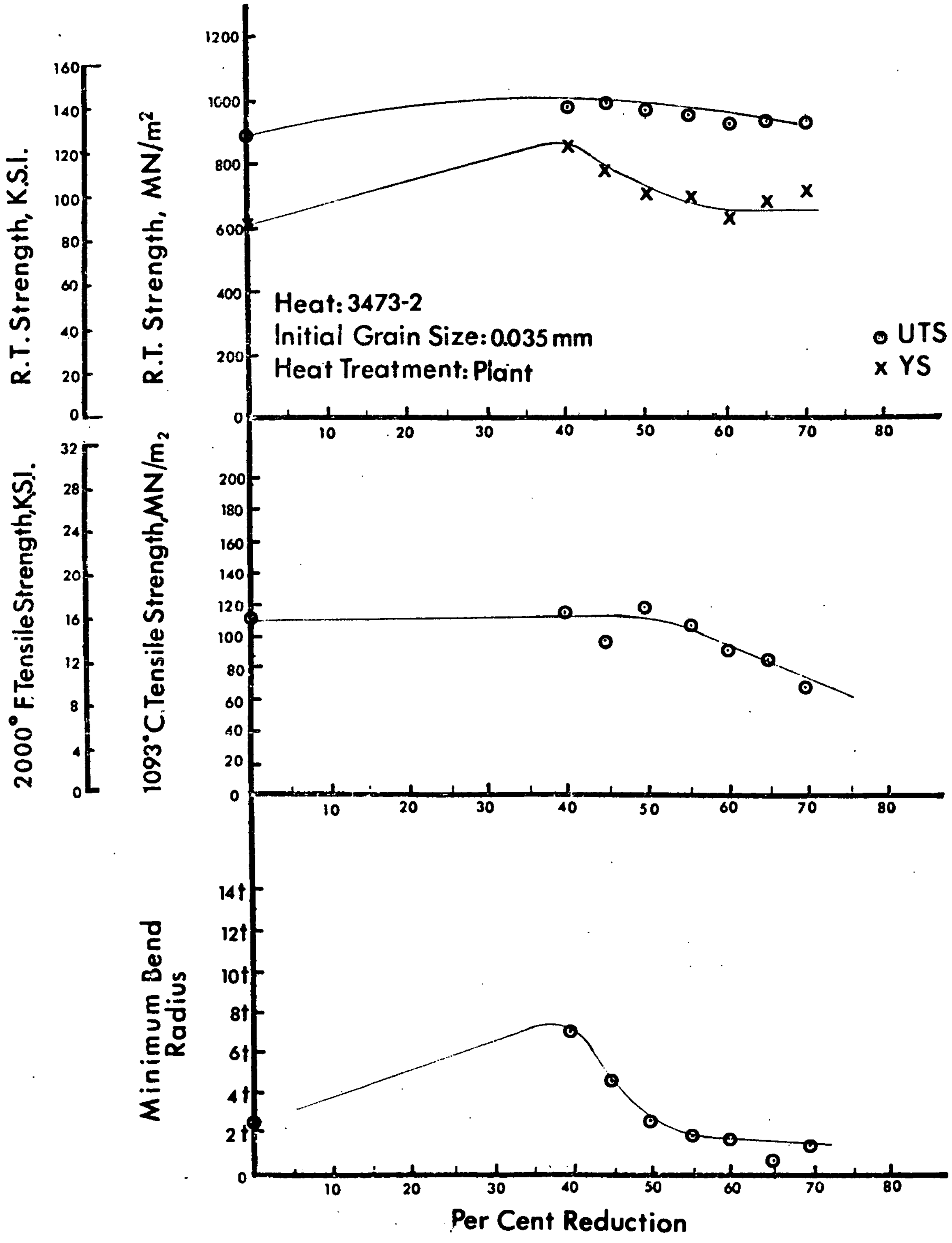


FIGURE 12

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3473

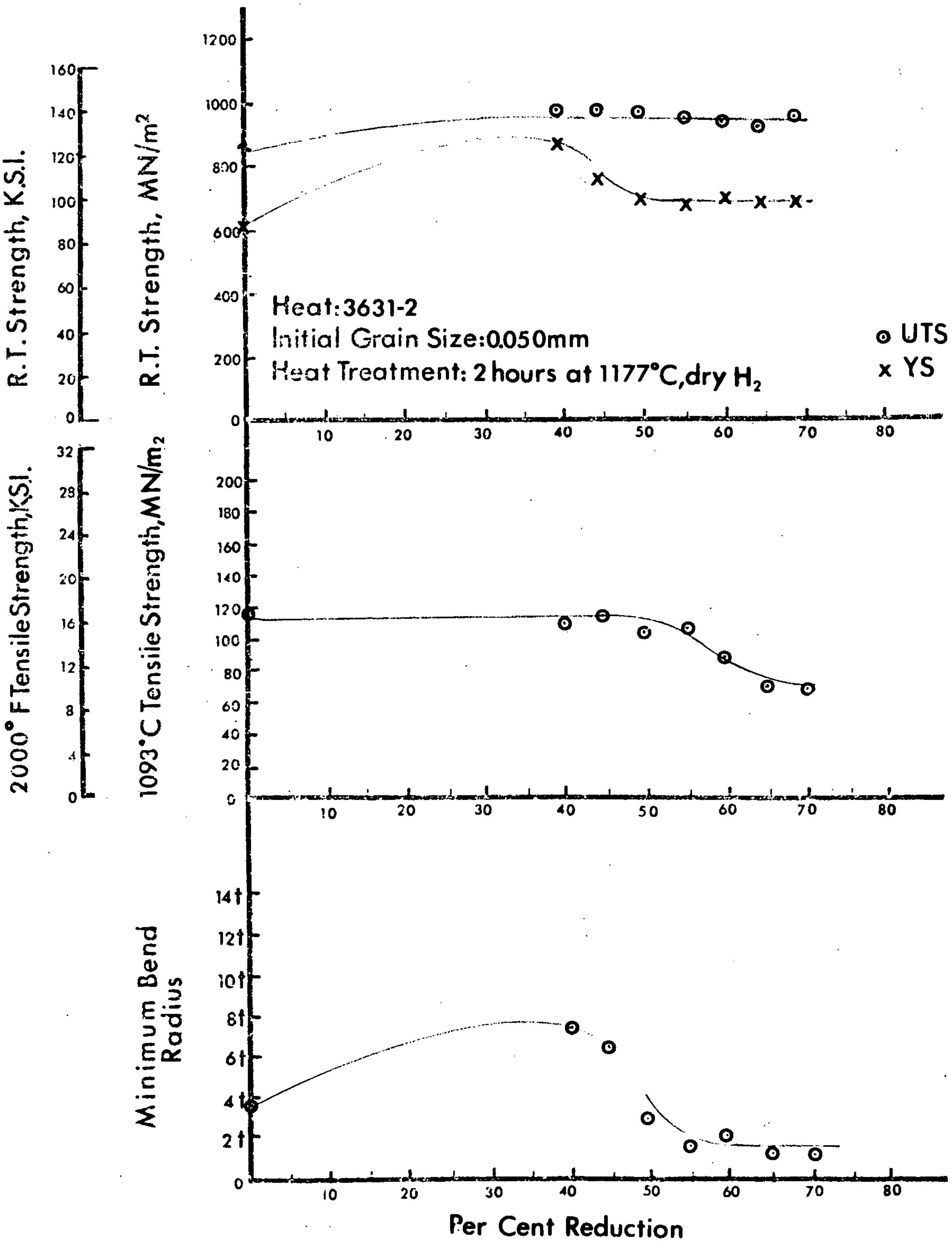


FIGURE 13

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3631

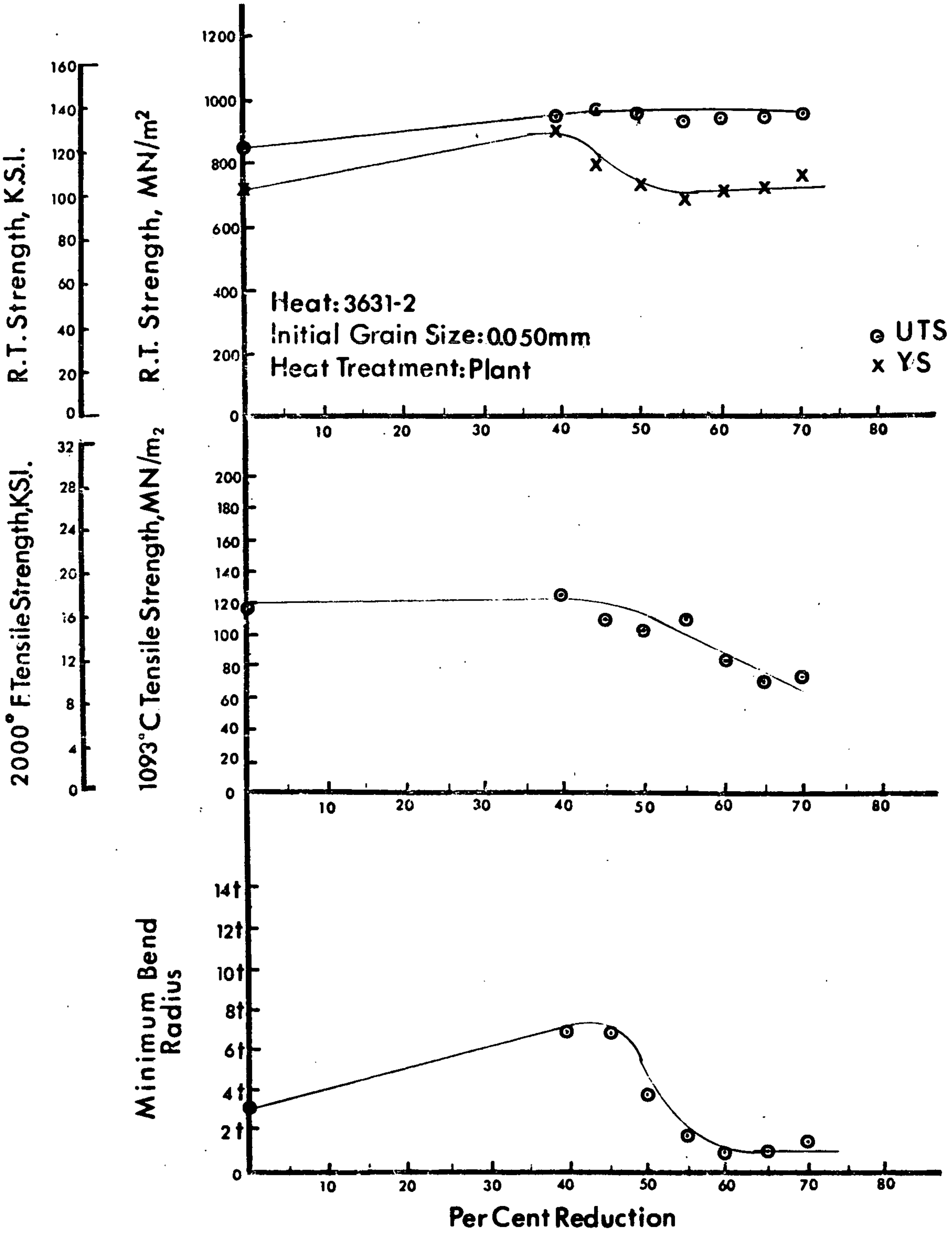


FIGURE 14

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3631

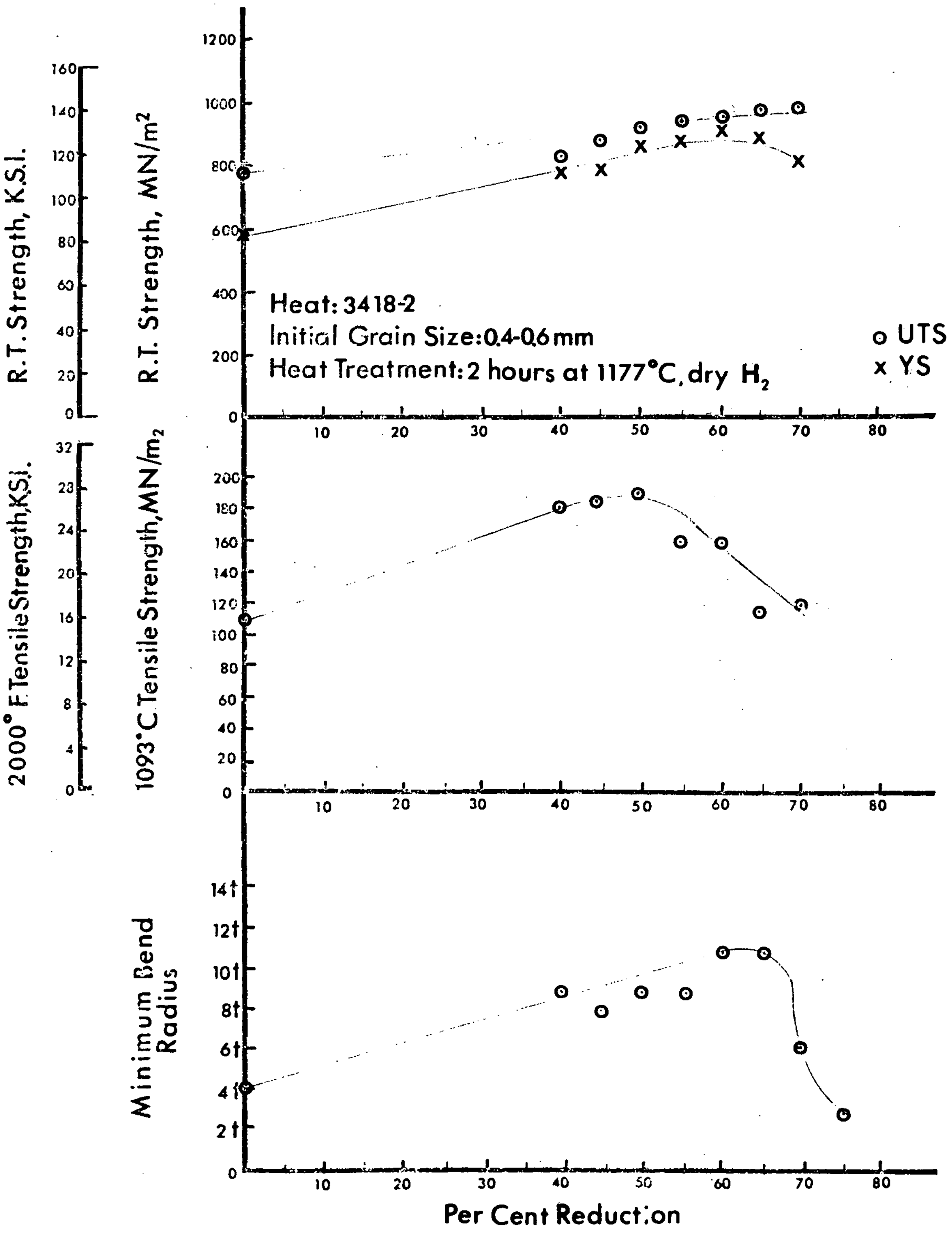


FIGURE 15

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3418

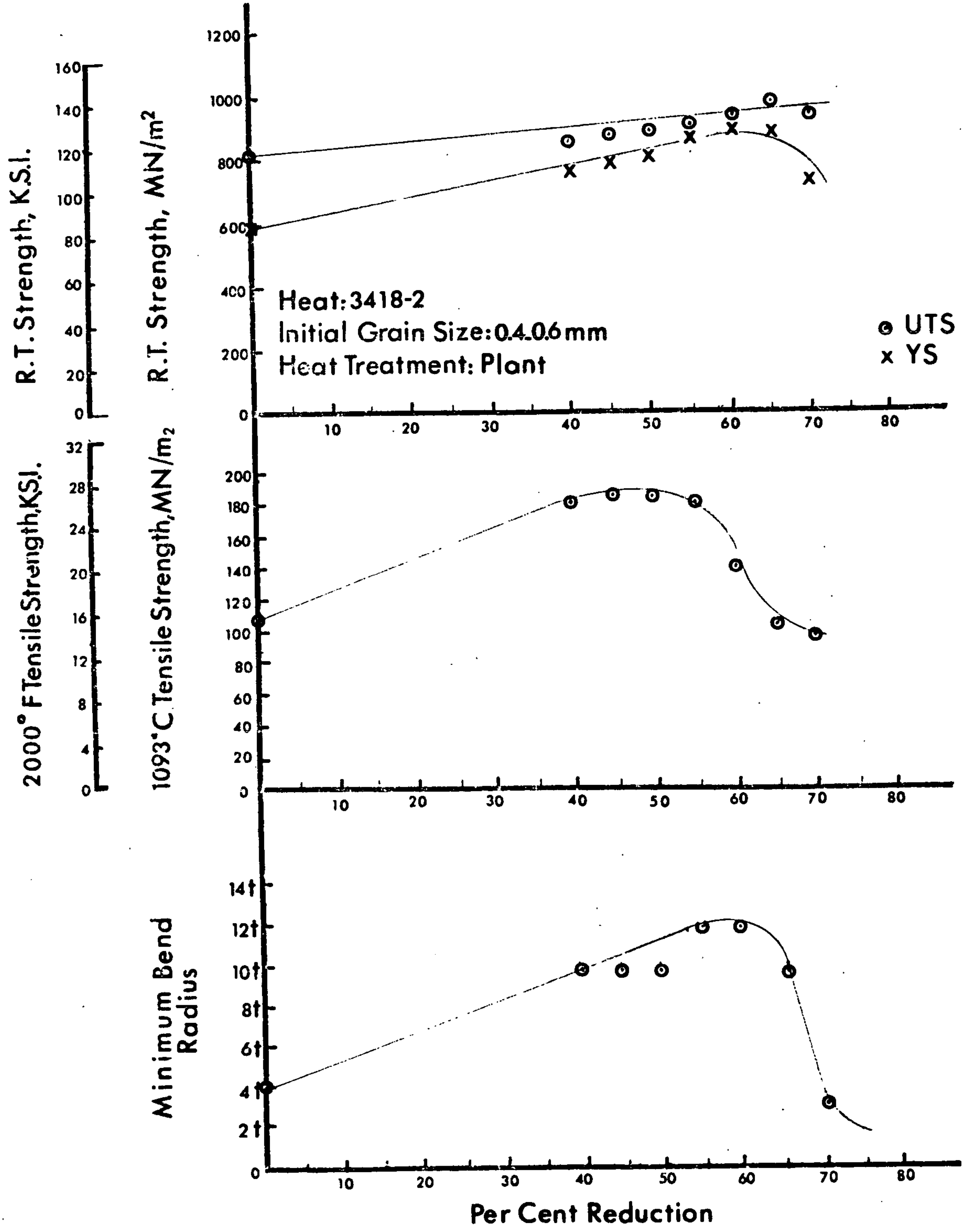


FIGURE 16

MECHANICAL PROPERTIES VERSUS PERCENT REDUCTION FOR HEAT 3418

(2.) Production Trials

Production of full size sheets of 0.025 cm. (0.010 in.) TDNiCr by the alternate process was performed concurrently with the laboratory investigation. Since much of the information from the laboratory investigation was not known when some of the heats were rolled, not all were fabricated by an optimum process. However, rolling on a production scale permitted early identification of rolling parameters, finishing techniques, etc.

Tables B-1 through B-14 in Appendix B contain the processing sequences, grain sizes and mechanical properties for each of the heats rolled on a production scale. In general, the same trends seen in the laboratory investigations were noted for these heats. Alternate process sheet possessed lower elevated temperature strength, but better room temperature strength and formability when finished at 0.025 cm. (0.010 in.). Most material produced met the Fansteel specification for 0.025 cm. (0.010 in.) sheet.

The processing sequence established made use of the grain size-optimum cold reduction relationship partially established in the laboratory investigations. More data points were obtained so that eventually accurate prediction of the optimum amount of cold work needed was possible. If the optimum percent reduction exceeded 50%, the processing sequence was to warm roll to 0.089-0.114 cm. (0.035-0.045 in.) thickness, recrystallize heat treat, grind to optimum gauge based upon grain size, cold tension roll to 0.051 cm. (0.020 in.), intermediate heat treat, and cold tension roll to final gauge followed by a final heat treatment. The rolling parameters were as follows:

- Roll on Schloemann single cluster configuration.
- Nickel or stainless steel leader strips spot welded to TDNiCr with the gauge of the leaders approximately the same as the starting gauge of TDNiCr.
- 15.3 cm.(6 in.) diameter work rolls with a 0.0025-0.0125 cm.(0.001-0.005 in.) crown.
- Strip speeds of 2.54 to 5.09 cm./second (1 to 2 in./second).
- Tension forces of 17.8-122.2 KN(4000-5000 lbs.), which is a maximum of 15% of the yield strength for the sheet widths used.
- Roll separating forces from 0.45 to 1.96 MN (50 to 220 tons).

As was shown in the laboratory investigation, high strength (large grain) warm rolled sheet produced higher strength cold rolled sheet. For this reason, later efforts were directed at cold rolling large grain sheet. Availability of this type of sheet was limited at first. Material with a "medium" grain size was therefore utilized. Later, two units, 3871 and 3872, were specially processed from the slab stage to sheet for the alternate process. Neither received the ThO_2 control normally exercised during sinter-consolidation. In addition, low reductions per pass were used in rolling from plate to sheet.

The use of coarse grain TDNiCr made necessary an alternate method of evaluating grain sizes. Heats such as 3418, which had an average grain diameter of 0.4 to 0.6 mm, contain very few grain boundaries when viewed in a standard micro. Hence, an accurate measurement, which is necessary for determining the optimum amount of cold work, was not possible.

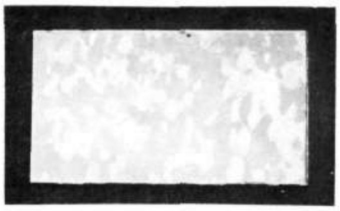
The use of a macro etch of a planar sheet section was found desirable for distinguishing between micro grain sizes greater than 0.10 mm. It is not known whether the variations in surface reflectivity revealed by macro etching are grains or clusters of grains, but it is known that the macro grain size is directly proportional to the micro grain size. Figure 17 shows several samples subjected to a macro etch.

The solution used for macro etching is as follows:

50 gm	$FeCl_3$ dissolved in
500 ml	distilled water
100 ml	HCl
100 ml	H_3PO_4

The specimen is polished through 400 grit paper, heated to approximately 65° C(150° F), dipped in the above solution, swabbed with lactic acid and rinsed in running water.

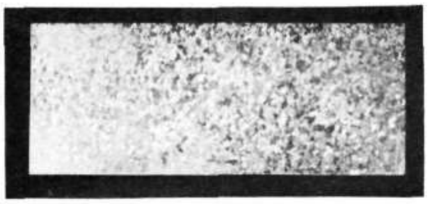
Each heat which was processed by the alternate process was macro etched and the macro grain size is included in the tables in Appendix B.



Heat 3471 at
0.127 cm.(0.050") gauge

Macro grain size
0.25 cm.(0.10")

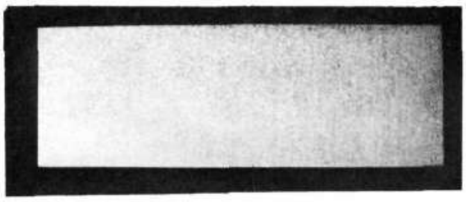
Micro grain size
0.18 mm



Heat 3456 at
0.025 cm.(0.010") gauge

Macro grain size
0.13 cm.(0.05")

Micro grain size
0.094 mm



Heat 3415 at
0.076 cm.(0.030") gauge

Macro grain size
0.03 cm.(0.01")

Micro grain size
0.045 mm

Heat 3418 at
0.102 cm.(0.040") gauge

Macro grain size
0.76 cm.(0.30")

Micro grain size
0.40 mm

1X Magnification

FIGURE 17

MACRO ETCHED SHEET

The principal advantage of a cold rolling process is the improved quality of the sheet product.

Gauge uniformity was obtained on randomly selected sheets throughout the course of this investigation and these results are included in Table 15. The average gauge spread per sheet is 0.0025 cm.(0.001 in.) and in no case does this number exceed 0.0034 cm.(0.0013 in.). It is noted that most of the heats first rolled have an average gauge above the nominal 0.025 cm.(0.010 in.). This accounts for the large deviations from the nominal weights shown in Table 16. However, Heat 3693 was rolled as close as possible to an average gauge of 0.025 cm. (0.010 in.) with the resultant weight deviations listed in Table 16. No deviation exceeds five percent of the theoretical weight. Subsequent heats were rolled equally as close to the nominal 0.025 cm.(0.010 in.) with improved weight deviations also.

Random sheets were also selected for rms finish readings. Results are presented in Table 17. The increase in surface roughness after the Scotch Brite finish of most of the sheets is due to the use of coarse abrasive pads. The use of finer abrasive pads, used on one sheet of Heat 3707, kept the rms finish below 6 microinches. Figure 18 shows the difference between the as-rolled and Scotch Brite finishes. As-rolled surfaces are thermal-etched by the heat treatment and show a macro grain structure. Polishing with Scotch Brite removes the surface patterns.

TABLE 15

GAUGE UNIFORMITY FOR COLD ROLLED 0.025 cm.(0.010 in.) TDNiCr SHEET

Heat No.	Maximum Gauge cm.	Maximum Gauge in.	Minimum Gauge cm.	Minimum Gauge in.	Maximum Gauge Spread cm.	Maximum Gauge Spread in.
3695-2	0.0282	0.0111	0.0262	0.0103	0.0024	0.0008
3690-1	0.0297	0.0117	0.0269	0.0106	0.0028	0.0011
3497	0.0280	0.0110	0.0244	0.0097	0.0033	0.0013
3707	0.0295	0.0116	0.0269	0.0106	0.0025	0.0010
3693-1	0.0280	0.0110	0.0254	0.0100	0.0025	0.0010
3693-2	0.0254	0.0100	0.0239	0.0094	0.0015	0.0006
3693-3	0.0266	0.0105	0.0239	0.0094	0.0028	0.0011

TABLE 16

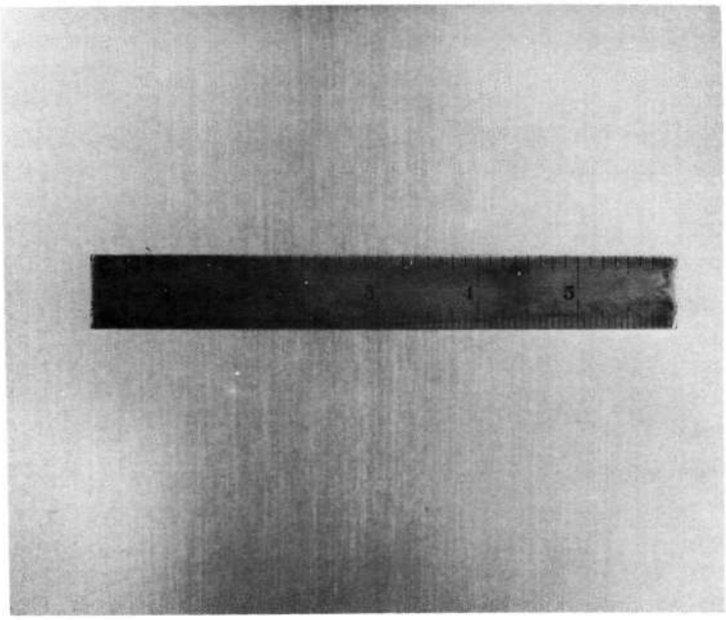
WEIGHT DEVIATIONS FOR COLD ROLLED 0.025 cm.(0.010 in.) TDNiCr SHEET

Heat No.	Actual Weight kg	Actual Weight lbs.	Theoretical Weight kg	Theoretical Weight lbs.	% Deviation
3707-1	3.2	7.0	2.8	6.2	+13.0
3707-D	3.7	8.2	3.4	7.5	+ 9.3
3707-A	1.8	3.9	1.6	3.5	+11.5
3707-B	1.3	2.8	1.1	2.5	+12.0
3497	4.4	9.6	4.1	9.1	+ 5.5
3690-1	2.3	5.1	2.1	4.6	+10.8
3690-2	2.2	4.9	2.1	4.6	+ 6.5
3690-3	2.2	4.9	2.2	4.9	0
3690-4	2.3	5.1	2.1	4.6	+10.8
3690-5	2.0	4.4	1.9	4.1	+ 6.8
3695-1	3.5	7.8	3.1	6.8	+14.8
3695-2	3.3	7.2	2.8	6.2	+16.0
3695-3	3.3	7.3	3.1	6.8	+ 7.7
3693-1	2.5	5.5	2.6	5.7	- 3.5
3693-2	1.9	4.1	2.0	4.3	- 4.7
3693-3	2.7	6.0	2.6	5.8	+ 3.5
3693-4	2.9	6.4	2.9	6.3	+ 1.6
3693-5	3.0	6.7	3.1	6.8	- 1.5
3693-6	2.8	6.1	2.9	6.3	- 3.2

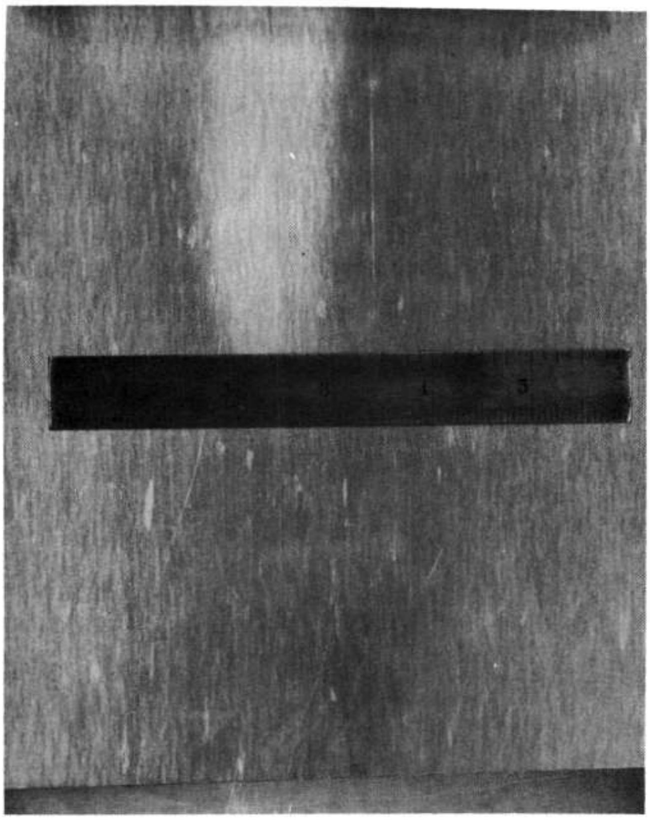
TABLE 17

SURFACE FINISH OF COLD ROLLED 0.025 cm.(0.010 in.) TDNiCr SHEET

Heat No.	Surface Treatment	Surface Finish, rms Long.	Surface Finish, rms Trans.
3707	None	2-1/2 to 3	3 to 3-1/2
3707	Scotch Brite	2-1/2 to 3	5 to 6
3707	Sand Blasted	45 to 50	40 to 45
3707	Sand Blasted and Roller Leveled	38 to 43	43 to 58
3707	Sand Blasted and Scotch Brite	12 to 15	19 to 22
3695-1	Scotch Brite	6 to 8	11 to 13
3695-2	Scotch Brite	4 to 6	11 to 13
3690	Scotch Brite	6 to 7	9 to 11
3690	Scotch Brite	4 to 8	4 to 6



Scotch Brite
Finish



As-rolled
Finish

FIGURE 18

SURFACE FINISHES OF HEAT TREATED TDNiCr
SHEET BEFORE AND AFTER SCOTCH BRITE POLISH

One sheet of Heat 3707 was sandblasted to remove indications of grains or grain clusters which appeared on sheet surfaces after heat treatment. Although a highly uniform matte surface was obtained, the rms finish increased to 38-60 microinches. This technique is therefore considered unacceptable.

One other added benefit of cold rolling is the increased length and widths possible. For warm rolled sheet, the maximum length is furnace limited. Cold rolled sheets were produced in lengths up to 610 cm.(20 ft.) as shown in Figure 19. In addition, edge cracking is rare in cold rolling, which means greater widths can be obtained at 0.025 cm.(0.010 in.). Production of sheet wider than 61.0 cm.(24 in.) was commonplace.

(3.) Creep Tests

Since one of the primary design criteria for TDNiCr sheet in a space shuttle application is the creep strength, several tests were run on alternate process sheet. In addition, residual properties, i.e., the room temperature strength and elongation after creep deformation, were determined on both alternate process sheet and standard TDNiCr. Most early alternate process sheet creep tested at 13.8 MN/m^2 (2.0 ksi) at 1093°C(2000°F) for 100 hours had an average .5% creep. Table 18 shows these results. This compares with approximately 27.6 MN/m^2 (4.0 ksi) for standard TDNiCr. There were, however, some notable exceptions. As previously discussed, large grains could be produced after cold rolling by beginning with large grains and choosing the proper amount

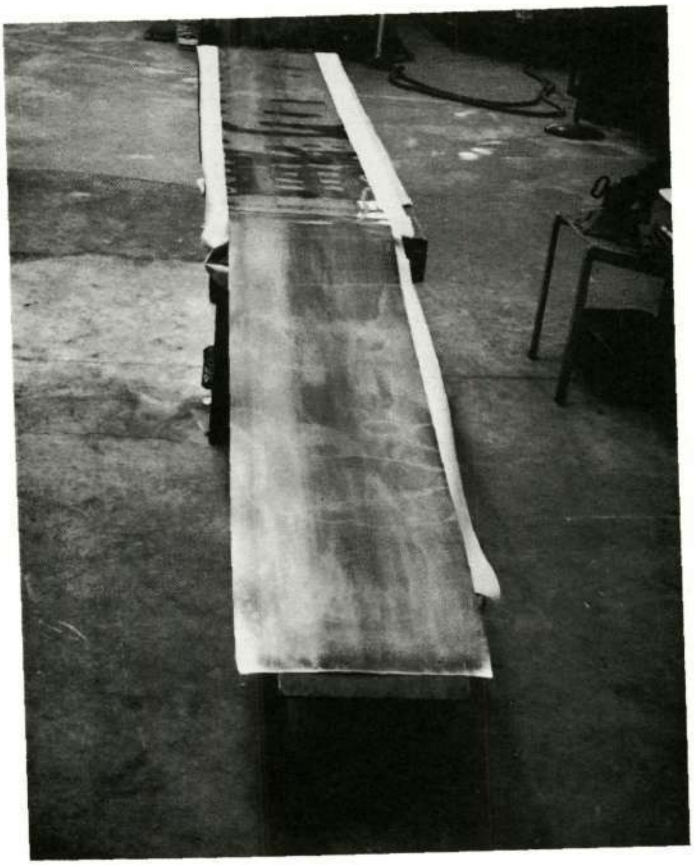


FIGURE 19

0.030 x 61 x 610 cm. (0.012 x 24 in. x 20 ft.)
SHEET OF COLD ROLLED TDNiCr, HEAT 3702

CZ

TABLE 18

1093°C (2000°F) CREEP RESULTS FOR ALTERNATE PROCESS SHEET

Ident.	Gauge cm.	Gauge in.	Creep Stress MN/m^2	Creep Stress ksi	Test Duration Hours	% Creep
3693	0.025	0.010	17.2	2.5	16.0	0.26
3693	0.025	0.010	27.6	4.0	0.25	0.62
3693	0.025	0.010	20.7	3.0	30.0	0.83
3693	0.025	0.010	20.7	3.0	30.0	0.55
3693	0.025	0.010	17.2	2.5	16.0	0.19
3631	0.046	0.018	17.2	2.5	1.0	0.22
3693	0.025	0.010	13.8	2.0	100.0	0.37
3418-1	0.025	0.010	20.7	3.0	100.0	0.06
3830	0.025	0.010	13.8	2.0	100.0	0.52
3830	0.025	0.010	13.8	2.0	100.0	0.34
3830	0.025	0.010	20.7	3.0	100.0	12.1

of reduction. Heat 3418, after cold rolling of 75%, showed only .06% creep at 1093°C(2000°F) 100 hours and 20.6 MN/m^2 (3.0 ksi).

As discussed previously, however, it was not until two units (3871 and 3872) were specially processed that this strength could be approached again. Heat 3872 possessed after cold rolling grains comparable in size to 3418. This fact, coupled with the high elevated temperature tensile strength, 135.7 MN/m^2 (19.7 ksi), indicates that the creep strength of this heat was about the same as 3418. Creep tests could not be run within the time limits of the Part II program.

Room temperature tensile tests were performed on standard and alternate process TDNiCr creep specimens to determine the residual properties. Twenty-two tests were conducted on TDNiCr produced by the standard process. These samples were creep tested as part of the reproducibility evaluation of Part I of this program; the creep deformations ranged from 0.02% to 6.25%.

Some loss of properties was found in all tests on standard TDNiCr, as shown in Table 19. The ultimate tensile strength and elongation are most susceptible to creep deformation. Strength reductions are approximately proportional to the amount of creep for long time tests (100 hours or more). Percent elongations generally fall between 1.0% and 6.0% regardless of the amount of creep over 0.08%.

TABLE 19

RESIDUAL PROPERTIES OF STANDARD TDNiCr AFTER CREEP TESTING

Heat/Sample No. /No.	Gauge		Creep Stress		Test Duration	% Creep	Room Temperature				
	cm.	in.	MN/m^2	ksi	Hours		UTS MN/m^2	ksi	YS MN/m^2	ksi	% Elong.
3415-64	0.051	0.020	20.7	3.0	100	0.02	777.9	112.9	516.1	74.9	18.0
3456-B2-3	0.051	0.020	27.6	4.0	100	0.08	682.1	99.0	565.7	82.1	3.0
3415	0.051	0.020	48.2	7.0	3.4	0.10	540.2	78.4	521.6	75.7	3.0
3456	0.025	0.010	27.6	4.0	200	0.10	646.3	93.8	564.3	81.9	3.0
3456-2	0.076	0.030	34.5	5.0	100	0.11	692.4	100.5	539.5	78.3	9.5
3456-B1-3	0.051	0.020	27.6	4.0	100	0.13	615.3	89.3	543.6	78.9	2.6
3502-1	0.076	0.030	27.6	4.0	100	0.20	456.1	66.2	456.1	66.2	2.5
3415-65	0.051	0.020	41.3	6.0	2.2	0.20	626.3	90.9	587.7	85.3	2.0
3415	0.051	0.020	48.2	7.0	2.0	0.20	731.0	106.1	516.8	75.0	10.0
3415-49	0.051	0.020	55.1	8.0	1.0	0.20	693.1	100.6	476.1	69.1	7.0
3456-1	0.076	0.030	27.6	4.0	100	0.21	680.0	98.7	494.7	71.8	2.5
3456-M2	0.051	0.020	34.5	5.0	180	0.25	513.3	74.5	513.3	74.5	1.0
3415	0.025	0.010	27.6	4.0	100	0.30	735.9	106.8	603.6	87.6	6.0

TABLE 19
(Cont'd)

RESIDUAL PROPERTIES OF STANDARD TDNiCr AFTER CREEP TESTING

Heat/Sample	Gauge		Creep Stress		Test Duration	%	Room Temperature				
							UTS		YS		
No. /No.	cm.	in.	MN/m^2	ksi	Hours	Creep	MN/m^2	ksi	MN/m^2	ksi	% Elong.
3456	0.051	0.020	34.5	5.0	105	0.33	443.7	64.4	443.7	64.4	1.5
3502-1	0.051	0.020	27.6	4.0	100	0.38	556.0	80.7	556.0	80.7	2.0
3456-B1-4	0.051	0.020	34.5	5.0	100	0.42	126.1	18.3	126.1	18.3	2.5
3415-61	0.051	0.020	55.1	8.0	0.5	0.44	461.6	67.0	461.6	67.0	2.5
3456-B2-3	0.051	0.020	34.5	5.0	100	0.45	161.9	23.5	161.9	23.5	2.0
3502-2	0.051	0.020	34.5	5.0	100	0.48	187.4	27.2	187.4	27.2	2.5
3415	0.051	0.020	44.8	6.5	20	0.60	184.6	26.8	184.6	26.8	2.5
3502	0.025	0.010	27.6	4.0	100	0.70	514.7	74.7	514.7	74.7	1.0
3415	0.076	0.030	34.5	5.0	100	6.25	135.6	19.7	135.6	19.7	2.5

The yield strength does not lose a substantial amount of its original strength up to 0.38% creep. For all tests on samples with 0.38% or less creep, the values were above 441 MN/m^2 (64 ksi). The majority of tests were conducted on transverse specimens which had undergone a long time creep test. However, several tests were made at high stresses and short times and results are also in Table 18. No difference in residual properties was found for creep tests of 2.2 hours or more. However, for creep tests less than two hours, significant increases in residual properties over long time test samples with the same percent creep were noted. For example, Heat 3415 with 0.20% creep in 2 hours had tensile, yield, and elongation values of 731.1 MN/m^2 (106.1 ksi), 516.8 MN/m^2 (75.0 ksi) and 10.0%.

Microstructural examination was performed on standard TDNiCr specimens with creep percents ranging from 0.15% to 6.0%. It was found that either grain boundary cracks or voids were present in all samples, and that the amount was greater with increasing percent creep. Frequently, oxidation had occurred in the cracks. It is not known, however, whether the oxidation or cracking occurred first. No evidence of slip lines, subgrain formation, grain boundary migration or fold formation was observed with light microscopy. The creep deformation measurements therefore include grain boundary cracking. The proportions of cracking deformation and true plastic deformation is not known.

Creep and tensile tests were also conducted on TDNiCr produced by the alternate process and these data are presented in Table 20. Cold rolled TDNiCr sheet showed virtually no decreases in ultimate tensile strength, yield strength, or elongation up to at least 0.26% creep (see Table 18). It is believed that texture, grain size and/or creep stress level are responsible for the improvements. Short time tests (less than two hours) again showed improved residual properties. One sample of Heat 3693 was deformed to 0.62% in creep in fifteen minutes with no change in room temperature properties after creep testing. One additional sample with 0.50% creep in six hours also maintained its original strength. Because the stress was increased in steps during the test, the majority of the creep in this specimen was introduced in one hour.

TABLE 20

RESIDUAL PROPERTIES OF ALTERNATE PROCESS TDNiCr AFTER CREEP TESTING

Heat	Gauge cm.	Gauge in.	Creep Stress MN/m^2	Creep Stress ksi	Test Duration Hrs.	% Creep	Residual Room Temperature Properties UTS MN/m^2	UTS ksi	YS MN/m^2	YS ksi	% Elong.
3693	0.025	0.010	17.2	2.5	16	0.26	860.6	124.9	648.3	94.1	18.0
3693	0.025	0.010	27.6	4.0	0.25	0.62	892.3	129.5	638.0	92.6	13.0
3693	0.025	0.010	20.7	3.0	30	0.83	330.0	47.9	330.0	47.9	0.5
3693	0.025	0.010	20.7	3.0	30	0.55	498.8	72.4	496.1	72.0	2.0
3693	0.025	0.010	20.7	3.0	1.5	0.20	855.7	124.2	605.6	87.9	15.0
3693	0.025	0.010	17.2	2.5	16	0.19	853.0	123.8	651.8	94.6	12.0
3631	0.046	0.018	17.2	2.5	1.0	0.22	928.1	134.7	671.8	97.5	15.5
3693	0.025	0.010	13.8	2.0	100	0.37	735.9	106.8	644.9	93.6	4.0
3693	0.025	0.010	20.7-34.5	3.0-5.0	6	0.50	865.4	125.6	620.1	90.0	10.0
3418-1	0.025	0.010	20.7	3.0	100	0.06	837.1	121.5	617.3	89.6	16.5

(4.) Comparison of Standard Process and Alternate Process for 0.025 cm.(0.010 in.) Sheet

Mechanical property trends and quality aspects are compared with standard TDNiCr sheet in Table 21. Both typical and minimum values have been given where applicable.

Sheet produced by the alternate process sacrifices some elevated temperature strength for improved quality, bend ductility, room temperature strength, and residual properties after creep testing. The amount of sacrifice in creep strength cannot be accurately measured. A conservative figure would be a 50% decrease from standard TDNiCr. However, it was shown that by cold rolling large grain material, significant increases in creep strength can result.

Because higher strength material was not produced until the end of this contract, no shipments were made to NASA. Applications should exist where the trade off of strength for quality and formability is desirable.

TABLE 21

COMPARISON OF STANDARD VS. ALTERNATE PROCESS
0.025 cm. SHEET

Characteristic	Standard	Alternate
Room Temp. UTS, MN/m^2	758-827 Typ. 689 Min.	896-999 Typ. 86 Min.
Room Temp. YS, MN/m^2	517-586 Typ. 482 Min.	620-792 Typ. 551 Min.
Room Temp. Elong. %	18% Typ. 10% Min.	18% Typ. 10% Min.
1093° C UTS, MN/m^2	124 Typ. 68.9 Min.	82.7 Typ. 68.9 Min.
1093° C Elong. %	3.0% Typ. 1.0% Min.	3.0% Typ. 1.0% Min.
1093° C Stress Rupture for 20 Hr. Life MN/m^2	41.3 Typ. 31.0 Min.	34.4 Typ. 31.0 Min.
Creep Stress to produce 0.5%, 100 Hrs., 1093° C MN/m^2	27.6 Typ.	13.8 Min. up to 24.1*
Minimum Bend Radius	3.0T Max.	2.0T Max.
Flatness	8%	4%
rms Finish	16 rms Max.	10 rms Max.
Max. Length cm.	274	508
Gauge Uniformity cm.	±0.0038	±0.0025
Max. % Creep without Loss in Residual Properties	0.08	0.27

*By cold rolling large grain sheet.

TABLE 21

COMPARISON OF STANDARD VS. ALTERNATE PROCESS 0.010 in. SHEET

Characteristic	Standard	Alternate
Room Temp. UTS, ksi	110-120 Typ. 100 Min.	130-145 Typ. 125 Min.
Room Temp. YS, ksi	75-85 Typ. 70 Min.	90-115 Typ. 80 Min.
Room Temp. Elong. %	18% Typ. 10% Min.	18% Typ. 10% Min.
2000°F UTS, ksi	18.0 Typ. 10.0 Min.	12.0 Typ. 10.0 Min.
2000°F Elong. %	3.0% Typ. 1.0% Min.	3.0% Typ. 1.0% Min.
2000°F Stress Rupture for 20 Hr. Life, ksi	6.0 Typ. 4.5 Min.	5.0 Typ. 4.5 Min.
Creep Stress to produce 0.5%, 100 Hrs., 2000°F ksi	4.0 Typ.	2.0 Min. up to 3.5*
Minimum Bend Radius	3.0T Max.	2.0T Max.
Flatness	8%	2%
rms Finish	16 rms Max.	10 rms Max.
Max. Length ft.	9	20
Gauge Uniformity in.	±0.0015	±0.0010
Max. % Creep without Loss in Residual Properties	0.08	0.27

*By cold rolling large grain sheet.

c. Finalized Process

Based upon the work carried out and described in the foregoing sections of this report, two sheet fabrication processes were established. These were a standard process for the fabrication of 0.025-0.102 cm.(0.010-0.040 in.) TDNiCr sheet and a tentative alternate process for the fabrication of 0.025 cm.(0.010 in.) sheet having improved quality, bend ductility, and room temperature strength at the expense of a sacrifice in elevated temperature strength.

Exploratory studies which were not incorporated in the Finalized Process are described in Appendix C.

(1.) Standard Process for 0.025-0.102 cm. x 60.96 cm. x 152.4 cm.(0.010-0.040 in. x 24 in. x 60 in.) Sheet

(a.) Powder Manufacture

- $Ni-20Cr-2ThO_2$ - Nominal Composition.
- ThO_2 size determination (Min. 8.5 mμ - Max. 12.0 mμ).

(b.) Hydrostatic Compaction

- Rubber boot - No Mylar liner - Metal mesh container - 414 MN/m^2 (60,000 psi).
- 68.0 kg.(150 lb.) Unit 8.25 x 21.59 x 66.04 cm.(3-1/4 x 8-1/2 x 26 in.) - Nominal size.

(c.) Sinter-Consolidate

- Measure compact.
- Cut edge border stock to fit - mild steel bar 5.08 cm.(2.0 in.) high x 3.81 cm.(1.5 in.) wide x length.

- Miter and weld edge border segments to form picture frame configuration.
- Cut tubing for gas inlet and outlet tubes - weld in position in picture frame edge.
- Cut and brake form 0.315 cm.(0.125 in.) thick mild steel cover plates to fit unit.
- Cut and form longitudinal edge of 15 or 16 gauge Armco aluminized steel, Type I (or equivalent) slip sheets.
- Assemble, clamp and weld seal canned unit and weld attach nose block.
- Leak check.
- Place unit in Pereny furnace (or equivalent) at temperature less than 204° C(400° F).
- Connect gas inlet and outlet tubes.
- Purge with Argon.
- Manual program heat up and introduce hydrogen:

  204° C(400° F) - dewpoint -18° C(0° F)

  316° C(600° F) - dewpoint -29° C(-20° F)

  454° C(850° F) - dewpoint -57° C(-70° F)

  1093° C±28° (2000° F±50°) - Min. 2 hours
  Max. 3 hours

- Pinch off seal gas tubes.

- Remove unit from furnace.
- Roll parallel to 21.59 cm.(8-1/2 in.) dimension at roll gap settings of 5.08 cm.(2.0 in.).
- Turn unit 90° and roll with roll gap setting adjusted to yield length equivalent to finish gauge required sheet width plus 15.24 cm.(6.0 in.).
- Turn unit 90° and roll two passes to a thickness of 4.17 cm.(1.64 in.).
- Turn unit 90° and subject to a flattening pass.
- Air cool.

(d.) Decan, Condition and Sample

- Saw crop all edges and ends as required to remove all cracks.
- Sample edge or end crop; ThO_2 size 17-22 mμ
- Mechanically strip cover plates and slip sheets.
- Mill, shape, face and/or grind all surfaces to remove all cracks, laminations, pits and sharp corners.

(e.) Recan

- Record L x W x T and weight of conditioned slab on route card.
- Cut edge border stock to fit mild steel bar 2.22 to 3.81 cm.(7/8 to 1-1/2 in.) width x thickness of slab ±0.315 cm.(0.125 in.).

B

- Miter and weld edge borders to form picture frame configuration.
- Ludox coat TDNiCr slab all surfaces.
- Ludox coat inside surface of 0.64 cm. (0.25 in.) thick mild steel cover plates.
- Weld assemble unit leaving corners open for venting during subsequent heat up.

(f.) Roll to Intermediate Gauge Plate

- Preheat canned slab to 760°C +0°C -38°C (1400°F +0°F -100°F) for minimum of 1 hr.
- Breakdown roll from 2.54 to 0.315 cm. (1 to 0.125 in.) utilizing reductions of 15 percent per pass.
- Reheat for a minimum of 10 minutes every other pass for a total of eight passes and then reheat after each pass.

(g.) Decan

- Shear trim edge and end borders.
- Strip cover plates.
- Stamp identify.

(h.) Chemical Clean

- Degrease
- Immerse for one to two hours in a solution of:

Water	3.79 mm^3 (1 gal.)
Sodium Hydroxide	1.07 mm^3 (36 oz.)
Potassium Permanganate	0.26 mm^3 (9 oz.)

at 100°C(212°F).

- Water rinse
- Wipe
- Preheat sheets in warm water rinse.
- Immerse for a maximum of one hour in a solution of:

Water	1.26 mm^3 (1/3 gal.)
Hydrochloric Acid	0.12-0.36 mm^3 ($\frac{1}{4}$-3/4 pt.)
Ferric Chloride	0.04 mm^3 (1$\frac{1}{2}$ oz.)

  at Room Temperature.

- Cold water rinse.
- Warm water rinse.
- Immerse for 5 to 60 minutes in a solution of:

Water	3.79 mm^3 (1 gal.)
Nitric Acid	1.20 mm^3 (2$\frac{1}{2}$ pt.)
Hydrofluoric Acid	0.24 mm^3 ($\frac{1}{2}$ pt.)

  Temperature not to exceed 51° C(125° F).

- Cold water rinse.
- Warm water rinse.
- Visually check each surface of each sheet for traces of rolled-in or included steel. Verify this examination by spot checking with a solution of 10% hydrochloric acid and a solution of 10% potassium ferricyanide for iron trace. In the event that traces of iron are present, repeat immersion of material

to the water-hydrochloric acid-ferric chloride solution followed by a cold and warm water rinse.

(i.) Preparation of Intermediate Gauge Plate for Canning

- Layout chemically cleaned plate and chalk mark for edge and/or end trimming to remove all cracks. In addition, chalk mark plate to appropriate sizes to satisfy finish product gauge, width and length.
- Preheat plate at 760° C +0° C -38° C (1400° F +0° F -100° F).
- Warm shear to layout lines. (Sample for chemistry). Reheat as required.

(j.) Can

- Shear a top and bottom cover plate having an approximate 1.27 cm.(0.5 in.) overlap on the TDNiCr piece to be canned. Material shall be either 15 or 16 gauge Armco Aluminized Steel, Type I or equivalent.
- Using a paint roller, apply a uniform coat of U.S.P. Milk of Magnesia to each surface of each TDNiCr plate and one side of each cover plate.
- Place top and bottom cover plate on TDNiCr plate with appropriate 1/2" overlap and clamp in position.

- Resistance spot weld leading and trailing ends of cover plates to secure clamped configuration.

(k.) Rolling to Gauge

- Preheat units at 760° C +0° C -38° C (1400° F +0° F -100° F) for a minimum of 30 minutes.
- Roll to a thickness of:

  0.152 cm.(0.060 in.) for finish sheet
  of 0.076 cm.(0.030 in.)
  0.119 cm.(0.047 in.) for finish sheet
  of 0.102 cm.(0.040 in.)
  or <0.076 cm.(0.030 in.)

  using average reductions per pass of between 5 and 6 percent.
- Reheat for 10 minutes after each double pass. Turn canned assembly end for end prior to each reheat.
- Decan, shear trim edges, ends and sheet lengths as required.
- Recan in 15 or 16 gauge Armco Aluminized Steel, Type I or equivalent, either two pieces of 0.152 cm.(0.060 in.) TDNiCr sheet to finish at 0.076 cm.(0.030 in.) final gauge or two pieces of 0.119 cm. (0.047 in.) TDNiCr sheet to finish at

0.051 to <0.076 cm.(0.020 to <0.030 in.). Utilize average reductions per pass of 5 to 6 percent for all rolling.

- Decan between 0.051 and 0.076 cm. (0.020 and 0.030 in.) dependent on final gauge required. Shear trim edges, ends and sheet lengths as required.
- Recan in 15 or 16 gauge Armco Aluminized Steel, Type I or equivalent, optimally four pieces of 0.051 to 0.076 cm.(0.020 to 0.030 in.) TDNiCr sheet to finish at 0.025 to <0.051 cm.(0.010 to <0.020 in.). In the event four pieces are not available, a minimum of two or a maximum of six pieces may be employed. Utilize average reductions per pass of 5 to 6 percent for all rolling.
- Decan all material to finish at 0.038 to 0.051 cm.(0.015 to 0.020 in.) to the high side of the tolerance plus 0.0025 to 0.0051 cm.(0.001 to 0.002 in.) in thickness.
- Decan all material to finish at 0.025 cm. (0.010 in.) at 0.030 to 0.033 cm.(0.012 to 0.013 in.).

(1.) Chemical Clean

- Decrease
- Immerse for one to two hours in a solution of:

Water	3.79 mm^3 (1 gal.)
Sodium Hydroxide	1.07 mm^3 (36 oz.)
Potassium Permanganate	0.26 mm^3 (9 oz.)

at 100° C (212° F).

- Water rinse
- Wipe
- Preheat sheets in warm water rinse.
- Immerse for a maximum of one hour in a solution of:

Water	1.26 mm^3 (1/3 gal.)
Hydrochloric Acid	0.12-0.36 mm^3 ($\frac{1}{4}$-3/4 pt.)
Ferric Chloride	0.04 mm^3 ($1\frac{1}{2}$ oz.)

at Room Temperature.

- Cold water rinse.
- Warm water rinse.
- Immerse for 5 to 60 minutes in a solution of:

Water	3.79 mm^3 (1 gal.)
Nitric Acid	1.20 mm^3 ($2\frac{1}{2}$ pt.)
Hydrofluoric Acid	0.24 mm^3 ($\frac{1}{2}$ pt.)

Temperature not to exceed 51° C(125° F).

- Cold water rinse.
- Warm water rinse.
- Visually check each surface of each sheet for traces of rolled-in or included steel. Verify this examination by spot checking with a solution of 10% hydrochloric acid and a solution of 10% potassium ferricyanide for iron trace. In the event that traces of iron are present, repeat immersion of material to the water-hydrochloric acid-ferric chloride solution followed by a cold and warm water rinse.

(m.) <u>Recrystallization Heat Treat</u>

- Check to determine that each sheet of material is identified.
- Punch holes for hangers.
- Load furnace rack.
- Place rack in retort.
- Preheat furnace to 538° C(1000° F) and hold for one hour.
- Purge retort with argon for a minimum of 15 minutes prior to loading into furnace.
- Increase furnace temperature to 704° C(1300° F).
- Load retort into furnace.

- Increase furnace temperature 38° C(100° F) every 30 minutes from 704° C(1300° F) to 871° C(1600° F).
- Introduce hydrogen.
- Continue 38° C(100° F) heat up rate every 30 minutes to 1177° C(2150° F). Hold for 2 hours.
- Turn off furnace, introduce argon.
- Remove retort from furnace at approximately 649° C(1200° F).
- Open retort and air cool at 204° C(400° F) to room temperature.

(n.) Finishing
0.038 to 0.102 cm.(0.015 to 0.040 in.) inclusive

- Attach TDNiCr sheet to stainless steel backer plate by resistance spot welding end containing furnace hang holes.
- Wide belt abrasive grind TDNiCr sheet surface using welded end as lead end in direction of belt travel. DO NOT REVERSE. Use Carborundum "Fastcut Waterproof Cloth 3830 - Aloxite Flex 45-100 grit x 965 F backing" (or equivalent). Use a minimum number of passes necessary to clean up surface irregularities - local condition as required.

- Repeat above two steps on reverse side of TDNiCr sheet to attain finish gauge requirements.
- Using worn 100 grit belt, subject each side of each TDNiCr sheet to a sufficient number of passes to obtain a 32 RMS or better finish.
- Vidigage sheet thickness on a 5" grid pattern, record results on layout sheet assigning Q.C. serial number to each sheet.
- Check surface finish in both longitudinal and transverse direction with a profilometer and record results on layout sheet.
- Shear to finish required width and length.
- Sample.

<u>0.025 to <0.038 cm.(0.010 to <0.015 in.)</u>

- Attach TDNiCr sheet to stainless steel backer plate by resistance spot welding end containing furnace hang holes.
- Wide belt abrasive grind TDNiCr sheet surface using welded end as lead end in direction of belt travel. DO NOT REVERSE. Use Carborundum "Fastcut Waterproof Cloth 3830 - Aloxite Flex 45-100 grit x 965 F backing" (or equivalent). Use a minimum number of passes necessary to clean up

surface irregularities - local condition as required.

- Repeat above two steps on reverse side of TDNiCr sheet to attain finish gauge requirements.
- Using worn 100 grit belt, subject each side of each TDNiCr sheet to a sufficient number of passes to obtain a 32 RMS or better finish.
- Shear to square sheet and remove all edge and/or end cracks.
- Condition sheared edges and ends with emory paper.
- Spot condition as required.
- Hand wipe degrease.
- Cold tension roll a minimum of 2 percent to a maximum of 5 percent reduction on single cluster configuration of Schloemann Mill, or equivalent to attain flatness and uniform surface appearance.
- Degrease.
- Heat treat per process step (m.) above.
- Shear to required width and length.
- Sample.

(2.) Alternate Process for 0.025 to <0.038 cm. (0.010 to <0.015 in.) Sheet - Tentative

(a.) Powder Manufacture

Same as Standard Process

(b.) Hydrostatic Compaction

Same as Standard Process

(c.) Sinter-Consolidate

Same as Standard Process except for sinter consolidation temperature: 1010° C±28° (1850° F±50°)

(d.) Decan, Condition and Sample

Same as Standard Process except that a ThO_2 size range need not be specified.

(e.) Recan

Same as Standard Process

(f.) Roll to Intermediate Gauge Plate

Same as Standard Process

(g.) Decan

Same as Standard Process

(h.) Chemical Clean

Same as Standard Process

(i.) Preparation of Intermediate Gauge Plate for Canning

Same as Standard Process

(j.) Can

Same as Standard Process

(k.) Rolling to Recrystallization Gauge

- Preheat units at 760° C +0° C -38° C (1400° F +0° F -100° F) for a minimum of 30 minutes.
- Using average reductions per pass of 2 to 4 percent, roll to a thickness of 0.089 to 0.102 cm.(0.035 to 0.040 in.).

(l.) Chemical Clean

Same as Standard Process

(m.) Recrystallization Heat Trest

Same as Standard Process

(n.) Sample

- Micro and macro grain size determination.

(o.) Wide Belt Abrasive Grind

- Optimum gauge based upon grain size.

(p.) Preparation for, and Cold Tension Rolling

- Shear trim and square.
- Attach nickel or stainless steel leader strips by resistance welding techniques.
- Roll to 0.051 cm.(0.020 in.) on Schloemann single cluster mill using 15.3 cm.(6 in.) diameter work rolls with a crown of 0.0025-0.0125 cm.(0.001-0.005 in.), strip speeds of 2.54 to 5.09 cm./sec.(1 to 2 in./sec.), tension forces of 17.8-122.2 KN(4000-5000 lbs.) and roll separating forces of 0.45 to 1.96 MN(50 to 220 tons).
- Crop off leader strips.

(q.) <u>Intermediate Heat Treat</u>

- Degrease
- Same as Step (m.) above

(r.) <u>Preparation for, and Cold Tension Rolling to Gauge</u>

- Same as Step (p.) above except -

  Roll to 0.025 cm.(0.010 in.)

(s.) <u>Final Heat Treat</u>

- Same as Step (q.) above

2. TDNiCr Foil

An interest developed for space shuttle application of TDNiCr Foil to be used as an insulation packaging material. For this use, the material would be required to be in the 0.0051 to 0.0127 cm.(0.002 to 0.005 in.) thickness range, have a quality surface finish and be readily fabricable. Strength requirements were of little or minor importance in this application.

Initial work carried out during Part I of this program consisted of two primary objectives. These were as follows:

- An interim process was to be developed to produce 45.72 x 121.92 cm.(18 x 48 in.) foil at gauges from 0.025 cm.(0.010 in.) down to the limit of the rolling equipment at Baltimore. At a later date, the width requirement would be scaled to 60.96 cm. (24 in.) either at the Baltimore Plant or at a toll facility.
- An interim process was to be developed to produce 30.48 x 121.92 cm.(12 x 48 in.) foil at gauges of 0.0051-0.0127 cm.(0.002-0.005 in.). Final rolling was planned for the Sendzimir Rolling Mill at the Fansteel North Chicago Plant or at a toll facility.

Two types of rolling were carried out at the Baltimore facility for the rolling of foil. These consisted of warm pack rolling of 0.025 cm.(0.010 in.) unrecrystallized TDNiCr sheet on a 2-High United Mill having rolls 66.04 cm.(26 in.)

C

in diameter by 91.44 cm.(36 in.) wide and cold tension rolling of recrystallized 0.025 cm.(0.010 in.) TDNiCr sheet on the Schloemann Mill employing a single cluster roll configuration.

It was found feasible to warm pack roll foil to 0.0128 cm. (0.0055 in.) thickness having both properties and surface finish comparable to warm rolled TDNiCr sheet at 0.025 cm. (0.010 in.) thickness. This process, however, was not believed to be very practical for subsequent sheet size scale up. Further, sheet surface finishes produced by warm rolling appeared to lack reproducibility.

Cold tension rolling on the Schloemann single cluster configuration yielded foil having a minimum thickness of 0.0127 to 0.0152 cm.(0.005 to 0.006 in.). While mechanical properties of this material appeared to be slightly lower than those obtained on warm rolled material, surface quality and bend ductility was greatly improved.

Sendzimir rolling was carried out on a 30.48 cm.(12 in.) mill at the Fansteel Plant in North Chicago as well as a 60.96 cm.(24 in.) mill at Rodney Metals. Utilizing these mills, it was feasible to produce foil to gauges of 0.0051 cm. (0.002 in.). Again, mechanical properties were found to be lower than those obtained on the warm rolled material, but surface quality and bend ductility were vastly improved.

Work initiated during Part II of the program consisted of additional rolling on the Rodney Metals Sendzimir mill and also on a double cluster Schloemann Mill at Cabot Corporation. This work is described below.

Two sheets from Heat 3702 which were rolled to 0.025 cm. (0.010 in.) as part of the "Alternate Process Investigation", were rolled at Rodney Metals to foil gauges. One sheet had a thickness range of 0.028-0.031 cm.(0.011-0.012 in.) at the starting gauge. An attempt to reduce it to 0.0155 cm. (0.0045 in.) resulted in breakage. The equivalent of 122 cm. (4 ft.) of 0.025 cm.(0.010 in.) material was lost. No problems were encountered with the second sheet.

Figure 20 shows these two sheets at the finish gauge of 0.076 cm.(0.003 in.). One sheet produced was 61 cm.(24 in.) wide and 591 cm.(19 ft. 5 in.) long, a size far in excess of the 61 cm.(24 in.) by 153 cm.(60 in.) goal for foil. The other sheet, while not as wide, was 1188 cm.(39 ft.) long. Quality of the coils was excellent.

Mechanical properties of this material are presented in Table 22.

Sheets of TDNiCr were also sent to Cabot Corporation of Kokomo, Indiana for toll rolling to 0.013 cm.(0.005 in.) foil. A total of 12.5 m^2 (135 sq.ft.) was required by NASA.

The mill utilized was a Schloemann double cluster with a work roll diameter of approximately 5 cm.(2 in.). The rolling conditions were similar to those previously employed both on a Sendzimir mill and the Baltimore Schloemann single cluster. Approximately equal front and back tensions, never exceeding 30% of the yield strength, were utilized. Work rolls were flat, but crown was introduced by a crowning device

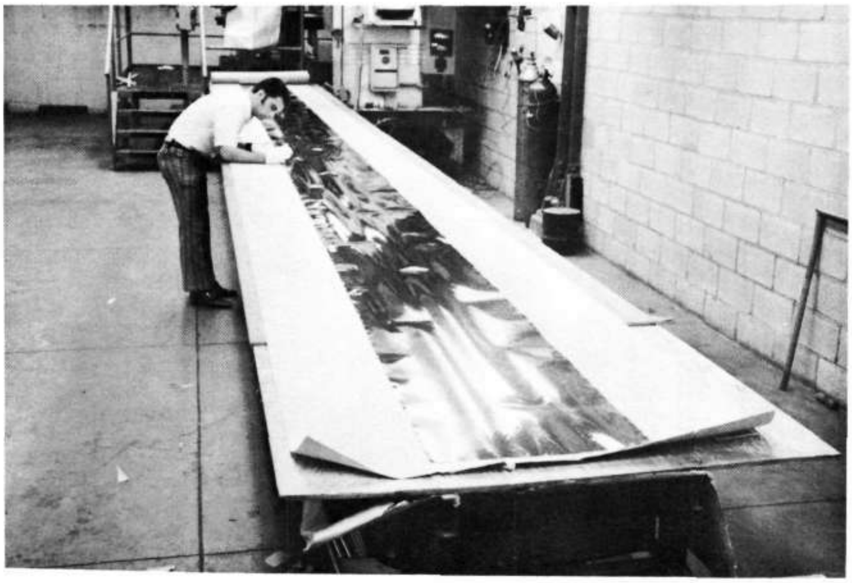
TABLE 22

MECHANICAL PROPERTIES OF TDNiCr FOIL

		Room Temperature			1093°C				1093°C
Heat No.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
3702	0.0076	748.9	675.9	2.5	77.2	68.2	9.2	2.0T	>20

		Room Temperature			2000°F				2000°F
Heat No.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
3702	0.0030	108.7	98.1	2.5	11.2	9.9	9.2	2.0T	>20

Heat 3702 - .0076x61x591 cm.
(.003"x24"x19'5")



Heat 3702 - .0076x49.5x1188 cm.
(.003"x19½"x39')

FIGURE 20

.0076 cm. (.003 in.) TDNiCr Foil

on the mill. Stainless steel leader strips were spot welded to the TDNiCr sheet.

An average of fifteen passes was necessary to achieve 50% reduction. This compares with approximately fifty passes for the single cluster configuration and eight passes for a Sendzimir mill.

A heat treatment of fifteen minutes at 1177° C(2150° F) in hydrogen was also performed at Cabot. Since tension is used on material passing through the bright anneal line, one end of the TDNiCr foil was welded onto "rider" sheets, which carried the foil through furnace. This arrangement prevented damage to the foil during the heat treatment.

A total of seven pieces was finished at 0.013 cm.(0.005 in.) with lengths ranging from 335 cm.(11 ft.) to 764 cm.(25 ft.). All but one sheet were rolled to the high side of the tolerance because starting gauges were slightly greater than 0.025 cm. (0.010 in.). An attempt to reduce one sheet to <0.013 cm. (0.0050 in.) resulted in breakage.

In addition, one sheet was annealed at 0.013 cm.(0.005 in.) and rolled to 0.0089 cm.(0.0035 in.) to demonstrate the feasibility of rolling to thinner gauges.

Mechanical properties of two of the 0.013 cm.(0.005 in.) sheets are presented in Table 23. It is expected that other sheets have similar properties since processing was identical.

The dimensions of all foil shipped to NASA designated locations may be found in Section 3. "Production of TDNiCr Product".

TABLE 23

MECHANICAL PROPERTIES OF 0.013 cm.(0.005 in.) TDNiCr FOIL

Heat No.	Room Temperature UTS MN/m²	ksi	YS MN/m²	ksi	% Elong.	1093°C (2000°F) UTS MN/m²	ksi	YS MN/m²	ksi	% Elong.	Stress Rupture Hrs. at 31.0 MN/m² (4.5 ksi)	Min. Bend Radius
3794	866.8	125.8	777.2	112.8	8.4	90.9	13.2	87.5	12.7	2.0	1.1	1.2T
3795	897.8	130.3	784.1	113.8	6.9	103.4	15.0	103.4	15.0	2.0	35.7	1.2T

Based upon these results, it was concluded that 60.96 cm. (24 in.) wide foil in the 0.0051 to 0.0127 cm.(0.002 to 0.005 in.) thickness range was feasible to produce on either the Sendzimir Mill or the double cluster Schloemann Mill.

In order to optimize mechanical properties, surface finish and bend ductility, it is recommended that the Finalized Alternate Process for TDNiCr 0.025 cm.(0.010 in.) sheet be utilized to prepare starting material. Further, the process should just be extended using the same fabrication techniques (Grain Size - Cold Reduction Relationship) to obtain optimized properties at finish gauge.

3. Production of TDNiCr Flat Product

During the Part II program, a total of 449.2 kg.(989.4 lbs.) of TDNiCr foil, sheet, and plate was shipped to NASA or NASA designated contractors.

Individual piece inventories for these shipments are included in Appendix D of this report.

II - TDNiCrAl SHEET PROCESS DEVELOPMENT

1. Alloy Selection

Alloy studies were conducted on the Part I program which demonstrated that the addition of Al to the TDNiCr matrix alloy provided improved cyclic oxidation protection under severe test conditions at temperatures up to 1260°C(2300°F).

During the Part II program the alloy studies were continued in order to fix the composition. The details of these studies are given in Appendix E, TDNiCrAl Exploratory Studies.

Several manufacturing techniques were explored for the production of the TDNiCrAl powders; these techniques are described in Appendix E.

Two alloy compositions were chosen for further development:

$$\text{Ni-16\%Cr-3.5\%Al-2\%ThO}_2$$

$$\text{Ni-16\%Cr-5.0\%Al-2\%ThO}_2$$

The 3.5% Al alloy appeared to afford the optimum combination of oxidation resistance, fabricability, and strength for space shuttle applications. The alloy containing the 5.0% Al provides additional oxidation resistance for long time service such as may be required in advanced aircraft engine applications.

The powder for both alloys was manufactured by a combination chemical-attrition route. The Ni-ThO_2 was manufactured by the Du Pont-Fansteel coprecipitation chemical route. The

$Ni\text{-}ThO_2$ powder was then wet attritored in a 15S Union Process Company attritor with a master alloy of Cr-Al of the appropriate composition to provide the desired alloy composition. The Cr-Al master alloys were purchased from Reading Alloys, Inc.

Sheet development and extrusion studies were made with both alloys. Standard procedures referred to herein are those used in standard practice for the manufacture of TDNiCr.

2. Sinter Cycle Development

A combined sinter-roll consolidation heating cycle for aluminum containing alloys modified to maximize oxygen and carbon removal and minimize recontamination was evaluated at 22.7 kg.(50 lb.) slab scale.

The heat used for the experimental cycle was Heat 3862, a 22.7 kg.(50 lb.) slab of $Ni\text{-}16Cr\text{-}2Al\text{-}2.2ThO_2$ compacted to the same cross section dimensions as are 45.4 and 68 kg. (100 and 150 lb.) slabs but to a shorter length.

A combined sinter and roll consolidation cycle was chosen so that possible reoxidation due to room temperature exposure during recanning for roll consolidation would be eliminated. Accordingly, the slab was canned in the as-compacted state in a standard roll consolidation can.

The sinter cycle temperature arrests and hydrogen flowrates were chosen to aid deoxidation and decarburization. The effluent gas was monitored by dewpoint and by a gas chromatograph.

Oxygen removal in the form of water vapor occurred primarily at the first arrest temperature 204° C(400° F) at which point a -57°C(-70°F) dewpoint was achieved before proceeding. Lesser amounts of water vapor were evolved up to 427°C(800°F) beyond which none was observed. Carbon removal in the form of methane was first observed at 204°C(400°F). Methane evolution reduced to nil values above 316°C(600°F). At 593°C(1100°F) the hydrogen flow was reduced to approximately 10% of the normal flowrate in order to reduce the chance of oxygen or carbon pickup from trace impurities in the hydrogen. Temperature was raised directly to 1204°C(2200°F) and the slab was roll consolidated by standard procedures.

After decanning at a rolled thickness of 2.54 cm.(1.0 in.) the slab was cut in two so that the center could be sampled. Oxygen and carbon analyses from the edge surface, the mid face surface and the center of the slab as recorded below show relatively high oxygen values at the edge surface but satisfactorily low in the center. The edge surface values are not considered representative of the material. This material is removed in conditioning at this stage.

	C ppm	Total O2 ppm
edge surface	206	12,200
center surface	104	5,420
center mid plane	102	3,400

Microstructural examination of the center of the slab shows approximately equivalent cleanliness to TDNiCrAl processed by separate sinter and roll consolidation cycles.

The experimental cycle was adopted for processing TDNiCrAl slabs in the processing studies.

3. Rolling Development

Studies on a number of heats of TDNiCrAl prepared early in Part II of this program are described in Appendix E. Various compositions were investigated, primarily for determining the oxidation resistance. All of these heats were fabricated similarly. Roll consolidation and hot rolling to gauge were both performed at 1204°C(2200°F). Heavy reductions per pass (at least 25%) were utilized in rolling. The microstructures produced in recrystallized sheet were non-uniform. The 1093°C (2000°F) tensile strength was typically in the 83.6-96.5 MN/m^2 (12-14 ksi) range, while the stress to produce 0.5% creep in 100 hours at 1093°C(2000°F) was 13.8 MN/m^2(2.0 ksi).

The next objective was to optimize the processing of these alloys to yield maximized properties in a reproducible manner. In order to initiate this work, material taken from scale-up Heats 3806-3812, 3816, 3831, 3848 and 3849 at the 2.54, 0.33 and 0.152 cm.(1.0, 0.13 and 0.060 in.) stages of processing were utilized. These studies are described in the following section.

Chemical analysis data for these scale-up heats are summarized in Table 24. As indicated, carbon levels are all below 500 ppm. The total oxygen is shown to vary from a low of 3300 ppm to a high of 7350 ppm over the series of scale-up units. However, the majority of results indicate a range of 4500 to 7000 ppm which is higher than the 3500 to 5500 ppm range experienced for standard TDNiCr material. An increase in oxygen content is to be expected as a result of the aluminum addition.

TABLE 24

CHEMICAL ANALYSIS OF TDNiCrAl SCALE UP ALLOYS

	Nominal Composition				Actual Composition							
Heat No.	% Cr	% Al	% ThO_2	Ni	% Cr	% Al	% ThO_2	% C	% S	Total O_2 ppm	Ni
3806	16.0	2.0	2.0	Bal.	15.11	2.65	2.35	0.014	0.003	3300	Bal.
3807	16.0	3.0	2.0	Bal.	15.45	3.00	2.37	0.022	0.003	4650	Bal.
3808	16.0	3.0	2.0	Bal.	15.32	3.04	2.35	0.017	0.004	6060	Bal.
3809	16.0	3.0	2.0	Bal.	15.51	3.12	2.34	0.026	0.005	6800	Bal.
3810	16.0	3.0	2.0	Bal.	15.48	3.04	2.37	0.014	0.003	6360	Bal.
3811	20.0	2.0	2.0	Bal.	19.30	2.30	2.29	0.010	0.004	6550	Bal.
3812	20.0	3.0	2.0	Bal.	19.09	3.10	2.20	0.013	0.005	6010	Bal.
3816	16.0	3.0	2.0	Bal.	15.64	3.02	2.27	0.022	0.005	7350	Bal.
3831	16.0	4.0	2.0	Bal.	15.25	3.97	2.29	0.043	0.003	5220	Bal.
3848	16.0	3.0	1.5	Bal.	14.53	2.80	1.42	0.044	0.005	4540	Bal.
3849	16.0	4.0	1.5	Bal.	15.38	3.80	1.40	0.047	0.004	4702	Bal.

a. Preliminary Studies

Initial work consisted of determining effect of various roll consolidation and hot rolling temperature upon TDNiCrAl sheet product.

Heats 3807 through 3810 were all of the same chemical composition, therefore, the effect of varying consolidation and rolling temperature to finish gauge was evaluated on these heats.

Heats 3807 and 3810 were both rolled from the consolidation stage to finish gauge at a temperature of 1204° C(2200° F) in a manner identical to Heats 3806, 3811, 3812, 3816, 3831, 3848 and 3849. Heats 3808 and 3809 were fabricated in a similar manner except that temperatures of 1093° C(2000° F) and 1149° C(2100° F) respectively, were employed for all rolling operations to final gauge.

Evaluation throughout processing of Heats 3807, 3808, 3809 and 3810 did not reveal any qualitative difference as a result of varying fabrication temperature from 1093° C(2000° F) to 1204° C (2200° F). The only observed effect due to varying temperatures was an increase in rolling mill load as temperature decreased.

Sheet from each heat at the final hot rolled gauge was wide belt abrasive ground to a gauge of 0.038 cm.(0.015 in.). At this gauge, all sheet was subjected to an 1177° C(2150° F) heat treatment for six hours in a hydrogen atmosphere to form a protective oxide coating prior to a final recrystallization heat treatment at 1316° C(2400° F) for two hours in a predominantly argon atmosphere followed by furnace cooling. Samples were then secured from each sheet of each heat for mechanical property evaluation. These results are summarized in Table 25. No effect

TABLE 25

MECHANICAL PROPERTIES OF TDNiCrAl SCALE UP ALLOYS

Heat No.	Gauge in.	Room Temperature UTS ksi	Room Temperature YS ksi	Room Temperature Elong. %-in.	2000°F UTS ksi	2000°F YS ksi	2000°F Elong. %-in.	Stress Rupture 2000°F Hours at 4500 psi	105° Bend
3806-A	0.015	128.3	83.3	24.5	14.3	13.4	3.0	>20.0	0.5T
B		133.8	79.9	15.0	14.1	12.9	5.0	>20.0	0.5T
C		126.3	73.2	20.0	13.9	12.6	4.5	-	1.0T
3807-A	0.015	134.5	77.7	15.5	13.1	12.2	4.5	>20.0	1.0T
B		138.1	87.0	14.0	13.5	12.6	4.0	>20.0	0.5T
C		140.2	97.4	14.5	13.8	11.9	4.5	-	2.0T
3808-A	0.015	144.6	97.9	13.0	14.0	13.2	3.0	>20.0	1.0T
B		148.1	97.7	13.5	13.5	12.6	5.0	>20.0	1.0T
C		155.4	105.8	13.5	15.0	13.6	4.5	-	2.0T
3809-A	0.015	139.3	97.2	12.0	16.8	15.9	2.5	>20.0	2.0T
B		140.1	96.9	12.5	13.5	11.9	3.5	>20.0	2.3T
C		133.3	95.3	12.5	16.7	14.6	4.0	-	4.0T
3810-A	0.015	133.6	77.9	17.5	14.7	12.9	3.5	>20.0	0.5T
B		132.5	88.1	13.0	13.7	12.0	4.5	>20.0	2.0T
C		135.8	87.7	13.5	15.3	13.1	5.0	-	2.0T
3811-A	0.015	136.0	80.1	20.5	14.0	12.7	3.0	>20.0	0.5T
B		128.0	83.1	14.5	13.6	13.4	4.0	>20.0	0.5T
C		120.1	69.8	15.5	14.9	14.1	2.0	-	0.5T

TABLE 25

MECHANICAL PROPERTIES OF TDNiCrAl SCALE UP ALLOYS

Heat No.	Gauge cm.	Room Temperature			1093°C			Stress Rupture 1093°C	105° Bend
		UTS MN/m^2	YS MN/m^2	Elong. %-cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	Hours at 31.0 MN/m^2	
3806-A	0.038	884.0	573.9	24.5	98.5	92.3	3.0	>20.0	0.5T
B		921.9	550.5	15.0	97.1	88.9	5.0	>20.0	0.5T
C		870.2	504.3	20.0	95.8	86.8	4.5	-	1.0T
3807-A	0.038	926.7	535.3	15.5	90.2	84.1	4.5	>20.0	1.0T
B		951.5	599.4	14.0	93.0	86.8	4.0	>20.0	0.5T
C		966.0	671.1	14.5	95.1	82.0	4.5	-	2.0T
3808-A	0.038	996.3	674.5	13.0	96.5	90.9	3.0	>20.0	1.0T
B		1020.4	673.1	13.5	93.0	86.8	5.0	>20.0	1.0T
C		1070.7	729.0	13.5	103.4	93.7	4.5	-	2.0T
3809-A	0.038	959.8	669.7	12.0	115.7	109.6	2.5	>20.0	2.0T
B		965.3	667.6	12.5	93.0	82.0	3.5	>20.0	2.3T
C		918.4	656.6	12.5	115.1	100.6	4.0	-	4.0T
3810-A	0.038	920.5	536.7	17.5	101.3	88.9	3.5	>20.0	0.5T
B		912.9	607.0	13.0	94.4	82.7	4.5	>20.0	2.0T
C		935.7	604.3	13.5	105.4	90.3	5.0	-	2.0T
3811-A	0.038	937.0	551.9	20.5	96.5	87.5	3.0	>20.0	0.5T
B		881.9	572.6	14.5	93.7	92.3	4.0	>20.0	0.5T
C		827.5	480.9	15.5	102.7	97.1	2.0	-	1.0T

TABLE 25 (CONT'D.)

MECHANICAL PROPERTIES OF TDNiCrAl SCALE UP ALLOYS

		Room Temperature			1093°C			Stress Rupture	
Heat No.	Gauge cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	1093°C Hours at 31.0 MN/m^2	105° Bend
3812-A	0.038	1048.7	717.9	12.0	90.3	84.1	4.5	>20.0	0.5T
B		1071.4	716.6	12.0	88.9	82.7	5.0	>20.0	0.5T
C		1030.1	713.8	13.0	93.0	87.5	3.0	-	1.0T
3816-A	0.038	939.1	610.4	12.0	106.8	106.8	2.0	>20.0	2.0T
D		944.6	640.1	9.0	92.3	86.1	2.5	>20.0	0.5T
C		1004.6	655.9	13.0	84.1	81.3	2.0	-	2.0T
3831-A	0.038	1119.6	723.5	14.5	104.0	97.8	4.5	>20.0	3.0T
B		1029.4	673.8	14.5	110.2	97.1	5.0	>20.0	3.0T
3848-A	0.038	883.3	492.7	15.0	90.9	86.1	2.5	>20.0	0.0T
B		997.4	626.3	17.0	90.9	85.4	4.5	>20.0	0.0T
3849-A	0.038	959.1	713.8	7.5	95.1	90.9	2.0	>20.0	3.0T
B		1019.0	663.5	10.0	106.8	102.0	2.5	>20.0	3.0T

TABLE 25 (CONT'D.)

MECHANICAL PROPERTIES OF TDNiCrAl SCALE UP ALLOYS

Heat No.	Gauge in.	Room Temperature UTS ksi	Room Temperature YS ksi	Room Temperature Elong. %-in.	2000°F UTS ksi	2000°F YS ksi	2000°F Elong. %-in.	Stress Rupture 2000°F Hours at 4500 psi	105° Bend
3812-A	0.015	152.2	104.2	12.0	13.1	12.2	4.5	>20.0	0.5T
B		155.5	104.0	12.0	12.9	12.0	5.0	>20.0	0.5T
C		149.5	103.6	13.0	13.5	12.7	3.0	-	1.0T
3816-A	0.015	136.3	88.6	12.0	15.5	15.5	2.0	>20.0	2.0T
D		137.1	92.9	9.0	13.4	12.5	2.5	>20.0	0.5T
C		145.8	95.2	13.0	12.2	11.8	2.0	-	2.0T
3831-A	0.015	162.5	105.0	14.5	15.1	14.2	4.5	>20.0	3.0T
B		149.4	97.8	14.5	16.0	14.1	5.0	>20.0	3.0T
3848-A	0.015	128.2	71.8	15.0	13.2	12.5	2.5	>20.0	0.0T
B		144.9	90.9	17.0	13.2	12.4	4.5	>20.0	0.0T
3849-A	0.015	139.2	103.6	7.5	13.8	13.2	2.0	>20.0	3.0T
B		147.9	96.3	10.0	15.5	14.8	2.5	>20.0	3.0T

upon properties was evident as a result of varying fabrication temperature. Overall, the properties achieved were generally higher than TDNiCr at room temperature, but lower at 1093°C (2000°F).

Subsequent work was carried out to determine the feasibility of bare hot rolling from the 2.54 cm.(1.0 in.) slab thickness at varied hot rolling temperatures. This work consisted of taking one half segments of Heats 3807, 3808 and 3809 at the 2.54 cm.(1.0 in.) slab thickness, conditioning by shaping and preheating at 1204, 1149 and 1093°C(2200, 2100, and 2000°F) respectively. Each piece was then subjected to a 25 percent reduction on the Schloemann Mill. In every case, surface tears resembling hot shortness appeared transverse to the rolling direction. These imperfections were more pronounced at work piece edges and became deeper as the rolling temperature was decreased as indicated in Figure 21. Since it was believed that the origin of the surface tears might possibly be attributed to machine tool marks from the conditioning operations, a fourth heat, 3810, was rolled at 1204°C(2200°F) in a direction parallel to the surface conditioning machining direction. Some improvement was noted, however, heavy edge tearing still persisted as shown in Figure 22. Two additional heats, 3816 and 3831, were conditioned on all surfaces by surface grinding. They were subsequently preheated at 1204°C(2200°F) and rolled. Again, surface tearing appeared on both surfaces and edges of two pieces.

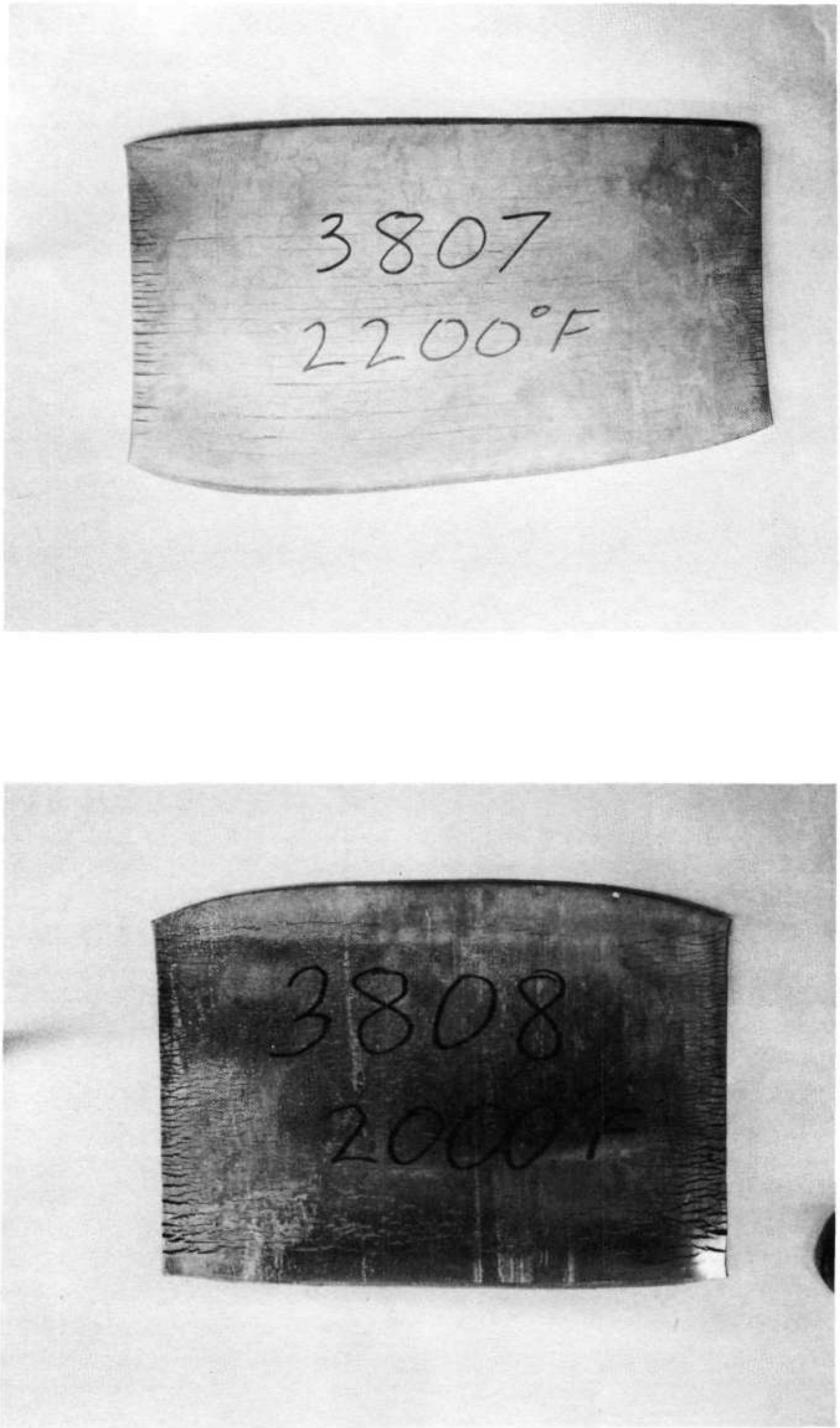


FIGURE 21

HEATS 3807 AND 3808 BARE HOT ROLLED
FROM 2.54 cm.(1.0 in.)

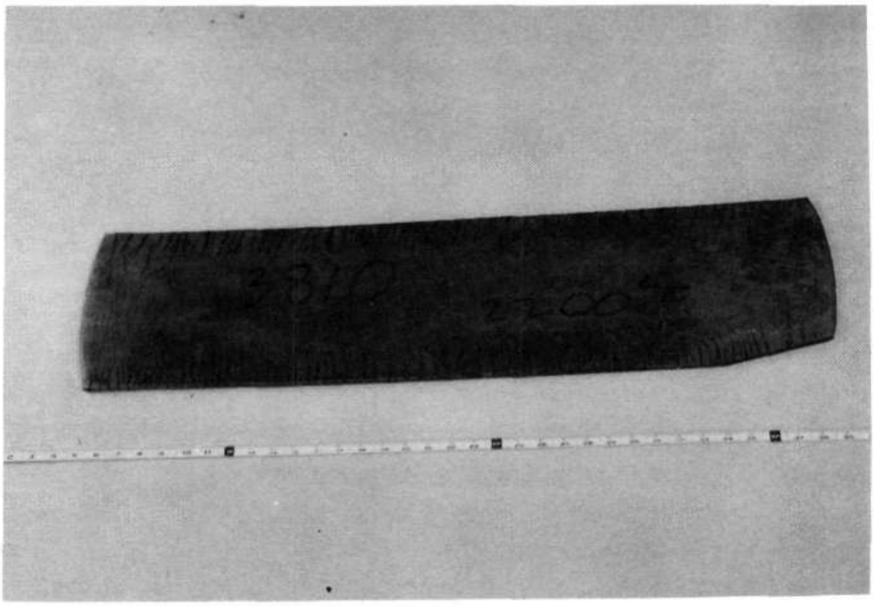


FIGURE 22

HEAT 3810 BARE HOT ROLLED
FROM 2.54 cm.(1.0 in.)

It was therefore concluded that bare rolling of the TDNiCrAl alloy material from the 2.54 cm.(1.0 in.) slab thickness was not feasible.

Piece 3807 which had been hot bare rolled at 1204° C (2200° F) was subjected to conditioning operations for removal of surface tears and edge cracks. It was then halved. The thickness of both sections subsequent to conditioning was approximately 1.09 cm.(0.43 in.). One piece was then preheated to 1204° C(2200° F) and the second piece was preheated to 1093° C(2000° F). Both pieces were then rolled with a reheat after each double pass. The piece rolled at 1204° C(2200° F) attained a thickness of 0.36 cm.(0.14 in.) and then cracked longitudinally from the trailing end as shown in Figure 23. It was believed that at this stage of processing, a reheat after every pass would have eliminated the occurrence of cracking. The second piece rolled at 1093° C(2000° F) failed in the same manner (see Figure 23) at a thickness of 0.48 cm. (0.19 in.) indicating a lower rolling temperature required reheating more frequently at a thicker gauge to prevent cracking. Thus, bare hot rolling the nominal 3 percent Al TDNiCrAl materials appears to be feasible after hot roll-consolidation and further reduction to about 1.25 cm.(0.5 in.) by standard practice.

While the temperature range of 1093° C to 1204° C(2000° F to 2200° F) appears satisfactory for fabricability of the TDNiCrAl materials, its effect upon thoria size and grain size after recrystallization had not been considered. Thus, thoria size data

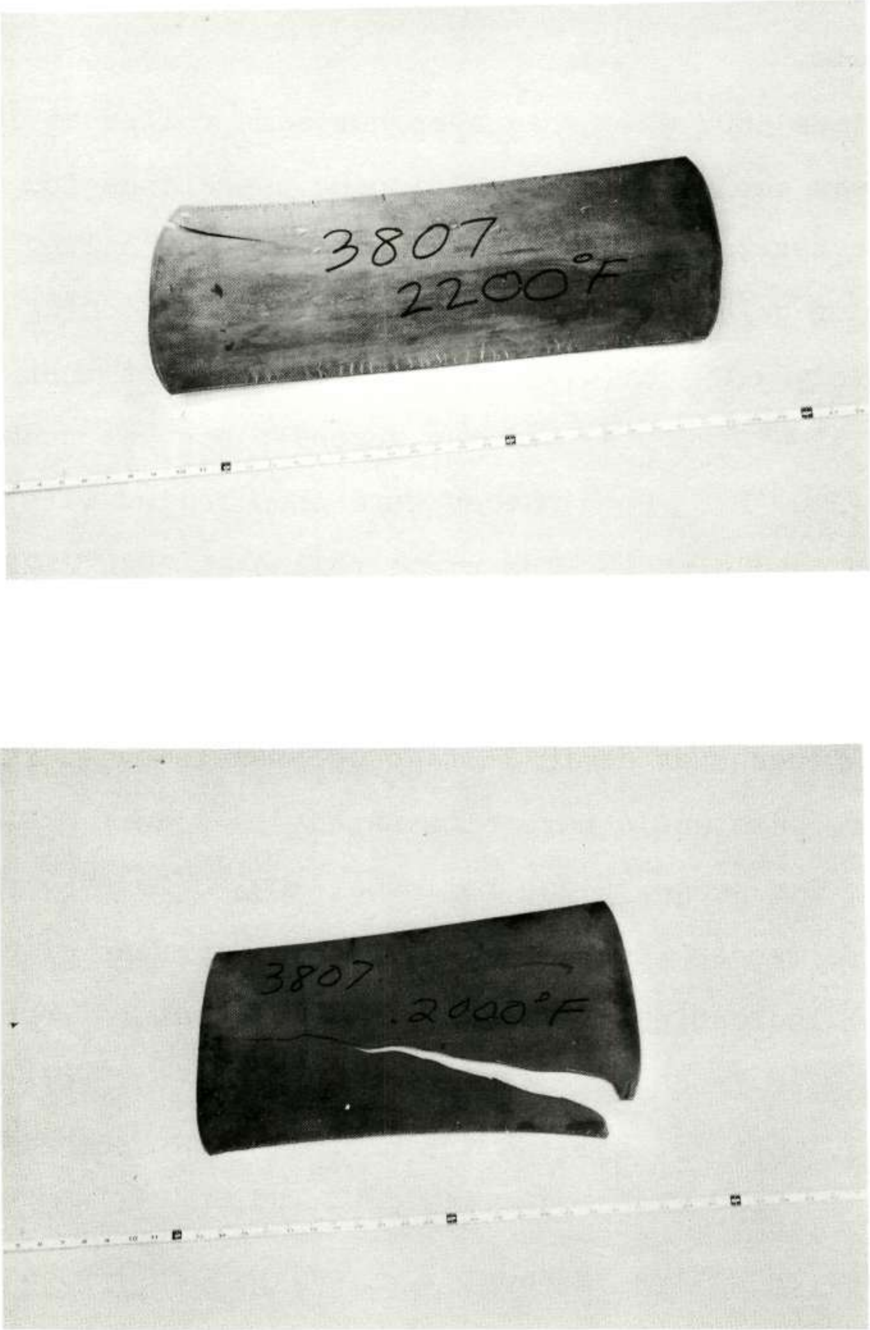


FIGURE 23

EFFECT OF INADEQUATE REHEATING
DURING BARE HOT ROLLING

were obtained as a function of temperature, time and intermediate hot rolled gauge for the scale-up alloys. These data are presented in Table 26A. As indicated, all alloys evaluated had thoria sizes of 19 to 21.5 mμ at the 2.54 cm.(1.0 in.) thick stage of processing regardless of roll consolidation temperature employed. These results are comparable to those attained on the conventional TDNiCr alloy fabricated in the same temperature range. However, upon subsequent fabrication to a thickness of 0.38 cm.(0.15 in.) and an additional time at temperatures of 1-1/2 hours, the thoria size grew to 28 to 29 mμ for all the alloys containing the nominal 2 percent thoria and 23.0 to 23.5 mμ for units containing a lesser thoria content of 1-1/2 percent. In both cases, greater growth was experienced than anticipated based upon time at temperature experiments evaluated during the Part I program for TDNiCr. For this reason, additional investigations were carried out on some of the TDNiCrAl scale-up alloys to determine the effect of time and temperature upon thoria size in 2.54 cm.(1.0 in.) thick slabs subsequent to roll consolidation and hot rolling. The results of this evaluation are shown in Table 26B.

As indicated on Heats 3807 and 3812, a temperature of 1204° C(2200° F) appears to effect a slight increase in thoria size after exposure of the specimen for one hour. Additional time at temperature up to four hours does not appear to cause any further growth, however, exposure for a six hour time period does effect a further 10 to 15 percent growth in thoria

TABLE 26

TDNiCrAl SCALE UP ALLOYS

A. - THORIA SIZE VS. TEMPERATURE, TIME AT TEMPERATURE AND HOT ROLLED GAUGE

			Thoria Size - mμ							
Heat No.	Nominal Composition	°C (°F)	Hrs.@ Temp.	2.54 cm (1.00 in)	Hrs.@ Temp.	0.38 cm (0.15 in)	Hrs.@ Temp.	0.152 cm (0.060 in)	Hrs.@ Temp.	0.076 cm (0.030 in)
3806	Ni-16Cr-2Al-2ThO$_2$	1204 (2200)	1.0	21.0	2.5	28.5	4.0	26.5	5.5	30.5
3807	Ni-16Cr-3Al-2ThO$_2$	1204 (2200)	1.0	21.0	2.5	28.5	4.0	28.0	5.5	30.5
3808	Ni-16Cr-3Al-2ThO$_2$	1093 (2000)	1.0	21.5	2.5	28.0	4.0	25.0	5.5	28.0
3809	Ni-16Cr-3Al-2ThO$_2$	1149 (2100)	1.0	19.0	2.5	28.0	4.0	25.0	5.5	28.0
3810	Ni-16Cr-3Al-2ThO$_2$	1204 (2200)	1.0	21.0	2.5	29.0	4.0	28.0	5.5	30.5
3811	Ni-20Cr-2Al-2ThO$_2$	1204 (2200)	1.0	21.5	2.5	28.0	4.0	28.0	5.5	30.5
3812	Ni-20Cr-3Al-2ThO$_2$	1204 (2200)	1.0	21.5	2.5	28.0	4.0	29.0	5.5	29.0
3816	Ni-16Cr-3Al-2ThO$_2$	1204 (2200)	1.0	21.5	2.5	-	4.0	28.0	5.5	29.0
3831	Ni-16Cr-4Al-2ThO$_2$	1204 (2200)	1.0	20.0	2.5	28.0	4.0	-	5.5	-
3848	Ni-16Cr-3Al-1.5ThO$_2$	1204 (2200)	1.0	20.0	2.5	23.0	4.0	25.0	5.5	30.5
3849	Ni-16Cr-4Al-1.5ThO$_2$	1204 (2200)	1.0	20.0	2.5	23.5	4.0	22.0	5.5	28.0

TABLE 26

TDNiCrAl SCALE UP ALLOYS

B. - THORIA SIZE AT 2.54 cm.(1.0 in.) VS. COMPOSITION, TEMPERATURE AND TIME

Heat No.	Nominal Composition	Temperature °C	Temperature °F	Thoria size - mμ As-rolled	Thoria size - mμ 1 hour	Thoria size - mμ 2 hours	Thoria size - mμ 4 hours	Thoria size - mμ 6 hours
3807	Ni-16Cr-3Al-2ThO_2	1204	2200	21.0	23.0	25.0	23.5	27.0
3808	Ni-16Cr-3Al-2ThO_2	1093	2000	21.5	21.0	20.0	19.0	21.0
3809	Ni-16Cr-3Al-2ThO_2	1149	2100	19.0	21.5	22.0	21.5	23.0
3812	Ni-20Cr-3Al-2ThO_2	1204	2200	21.5	24.0	23.5	24.0	26.5
3848	Ni-16Cr-3Al-1.5ThO_2	1204	2200	20.0	21.5	24.0	24.0	24.0
3849	Ni-16Cr-4Al-1.5ThO_2	1204	2200	20.0	22.0	20.0	22.0	23.5

size. This same trend also appeared for a temperature of 1149° C(2100° F) shown in the case of Heat 3809. On the other hand, a temperature of 1093° C(2000° F) does not appear to alter the as-rolled thoria size for exposure times up to six hours as exhibited by Heat 3808.

Since none of the 2 percent thoria containing alloys at the 2.54 cm.(1.0 in.) thickness which were subjected to temperatures of 1093°, 1149° and 1204° C(2000°, 2100° and 2200° F) for times up to six hours exhibited thoria sizes greater than 27 mμ, it was concluded that hot working and exposure to temperatures in the same range contributed to thoria growth of these same heats evaluated at hot rolled gauges of 0.380, 0.152 and 0.076 cm.(0.150, 0.060 and 0.030 in.) as indicated in Table 26A. Consequently, it is believed that hot rolling temperatures of 1093° C(2000° F) or greater of TDNiCrAl alloys containing 2 percent nominal thoria contents result in larger thoria sizes during fabrication from the 2.54 cm. (1.0 in.) thickness to intermediate gauges of 0.38 cm. (0.150 in.) or less. Larger thoria sizes are believed to contribute to small grained recrystallized structures which then result in lower elevated temperature properties.

During this same period of time, an investigation was initiated to determine temperatures required for recrystallization of TDNiCrAl alloys at various intermediate hot rolled gauges. Samples were obtained from Heats 3807, 3808, 3809 and 3810 in the as-hot-rolled condition at gauges of 2.54, 0.38 and 0.15 cm.(1.0, 0.15 and 0.06 in.). Each gauge of each heat was cut into four segments. One segment was maintained in the as-rolled condition and the three remaining segments were subjected to heat treatments of 1093°, 1204° and 1316° C(2000°, 2200° and 2400° F) for two hours respectively. Subsequently, all four segments were evaluated metallographically to determine degree of recrystallization. Table 27 shows micro-hardness data accumulated during this investigation. On all four heats, the micro-hardness values decrease from the as-rolled condition on the 2.54 and 0.38 cm.(1.0 and 0.15 in.) thick material after a heat treatment of 1093° C(2000° F) indicating recrystallization has taken place. Similar results also occurred as a result of the 1204° and 1314° C(2200° and 2400° F) heat treatments. Metallographic evaluation verified these results. However, it was noted that a much cleaner microstructure was obtained as the temperature increases with the most homogeneous structure occurring at 1316° C(2400° F). Finally, it may also be noted that little if any recrystallization occurs on any of the 0.15 cm.(0.06 in.) material. This is believed to be caused by primary recrystallization taking place during reheating of the 0.38 cm.(0.15 in.) at temperatures of 1093°-1204° C(2000°-2200° F) for subsequent hot rolling to a gauge of 0.15 cm.(0.06 in.) and thus resulting in an insufficient quantity of cold work being imparted to induce recrystallization at this stage of processing. Consequently, rolling must be carried out at temperatures lower than the recrystallization temperature if reheating is required.

TABLE 27.

MICRO-HARDNESS DATA

Heat No.	Gauge cm.	Gauge in.	Micro-hardness DPH - 1000 g.load - 20X As-rolled	1093°C (2000°F)	1204°C (2200°F)	1316°C (2400°F)
3807	2.54	1.00	347	283	240	294
3807	0.38	0.15	379	284	281	275
3807	0.15	0.06	326	339	315	320
3808	2.54	1.00	338	239	284	258
3808	0.38	0.15	382	296	286	282
3808	0.15	0.06	329	321	318	311
3809	2.54	1.00	364	287	274	248
3809	0.38	0.15	379	352	275	274
3809	0.15	0.06	363	358	348	347
3810	2.54	1.00	329	243	250	242
3810	0.38	0.15	365	313	280	273
3810	0.15	0.06	319	332	325	330

E

Next, lower rolling temperatures were evaluated as a means of eliminating the thoria growth and in-process recrystallization problems. Initial work was accomplished utilizing Heat 3806 at the 2.54 cm.(1.0 in.) thickness. One half segment of Heat 3806 had previously been fabricated from the 2.54 cm. (1.0 in.) thickness to a finish gauge utilizing a rolling temperature of 1204° C(2200° F). The remaining half segment was canned in Type 304 stainless steel and rolled at 982° C(1800° F) until can failure occurred at 0.64 cm.(0.25 in.) work piece thickness. Upon decanning, the TDNiCrAl material was found to be completely free of edge cracks and to contain an excellent surface.

At the 0.64 cm.(0.25 in.) thickness, Heat 3806 was halved. One half piece was canned in mild steel and rolled to gauge at 982° C(1800° F) while the second half was canned in aluminized steel and rolled to gauge at 760° C(1400° F).

Evaluation of mechanical properties at final gauge indicated slightly improved elevated temperature strengths on the material rolled to gauge at the lower temperatures. Table 28 summarizes these results.

Metallography evaluation revealed that material rolled at the lower temperatures possessed structures which were more uniform than those rolled at the 1204° C(2200° F) temperature. As indicated in Table 29, the 760° C(1400° F) rolled material had a grain size twice that of the 982° C(1800° F) rolled material and close to that of TDNiCr.

TABLE 28

MECHANICAL PROPERTIES OF TDNiCrAl 3806

		Room Temperature			1093°C			Stress Rupture 1093°C	105°
Heat No.	Gauge cm.	UTS MN/m^2	YS MN/m^2	Elong. %	UTS MN/m^2	YS MN/m^2	Elong. %	Time @ 31.0 MN/m^2	Min. Bend
3806-A*	0.038	884.0	573.9	24.5	98.5	92.3	3.0	>20.0	0.5T
3806-B*	0.038	921.9	550.5	15.0	97.1	88.9	5.0	>20.0	0.5T
3806-C*	0.038	870.2	504.3	20.0	95.8	86.8	4.5	>20.0	1.0T
3806-D**	0.038	815.8	501.2	17.5	115.1	115.1	1.5	>20.0	1.0T
3806-E***	0.038	798.6	579.4	14.5	112.3	106.8	5.0	>20.0	1.0T

*3806-A, B, C - Rolled to gauge at 1204°C.
**3806-D - Rolled to 2.54 cm. at 1204°C. Rolled to gauge at 982°C.
***3806-E - Rolled to 0.635 cm. at 982°C. Rolled to gauge at 760°C.

		Room Temperature			2000°F			Stress Rupture 2000°F	105°
Heat No.	Gauge in.	UTS ksi	YS ksi	Elong. %	UTS ksi	YS ksi	Elong. %	Time @ 4.5 ksi	Min. Bend
3806-A*	0.015	128.3	83.3	24.5	14.3	13.4	3.0	>20.0	0.5T
3806-B*	0.015	133.8	79.9	15.0	14.1	12.9	5.0	>20.0	0.5T
3806-C*	0.015	126.3	73.2	20.0	13.9	12.6	4.5	>20.0	1.0T
3806-D**	0.015	118.4	74.2	17.5	16.7	16.7	1.5	>20.0	1.0T
3806-E***	0.015	115.9	84.1	14.5	16.3	15.5	5.0	>20.0	1.0T

*3806-A, B, C - Rolled to gauge at 2200°F.
**3806-D - Rolled to 1.0 in. at 2200°F. Rolled to gauge at 1800°F.
***3806-E - Rolled to .250 in. at 1800°F. Rolled to gauge at 1400°F.

TABLE 29

GRAIN SIZES AND ASPECT RATIOS FOR HEAT 3806 PROCESSED AT DIFFERENT ROLLING TEMPERATURES

Rolling Temperature °C	Rolling Temperature °F	Orientation	Avg. Grain Dia. mm	Aspect Ratio
982	1800	Long.	0.013	1.9
982	1800	Trans.	0.013	2.5
760	1400	Long.	0.025	1.9
760	1400	Trans.	0.028	2.4

All samples heat treated 6 hours, 1177° C (2150° F) and 2 hours at 1316° C, evaluated at 0.038 cm. (0.015 in.)

As an alternate route to either hot or warm rolling to gauge, the feasibility of cold rolling was also explored. Initial efforts in this direction consisted of obtaining specimens from Heats 3806-3812, 3816, 3848 and 3849 at gauges of 0.38 and 0.15 cm.(0.15 and 0.06 in.). Specimens at both gauges from Heats 3806, 3810, 3811, 3812, 3816, 3848 and 3849 were subjected to a recrystallization-homogenization heat treatment of two hours at 1316° C(2400° F), while specimens from Heats 3807, 3808 and 3809 were maintained in the hot rolled condition. Specimens from all heats at the 0.38 and 0.15 cm. (0.15 and 0.06 in.) thicknesses were then subjected to cold rolling. All the 0.15 cm.(0.06 in.) material was cold rolled on the Schloemann single cluster configuration approximately 50 percent without any intermediate heat treatments. At this stage, material from all the heats appeared excellent. No indications of edge cracking were evident. The pieces were halved. One half was rolled still further to yield a total cold reduction of 70 percent. The remaining half sheet from each heat was subjected to six hours at 1149° C(2150° F) and two hours at 1316° C(2400° F) for homogenization and recrystallization. Samples were then secured for metallographic evaluation as well as mechanical property determination.

Table 30 summarizes the results of mechanical property testing of the cold rolled TDNiCrAl alloys at 0.064 cm. (0.025 in.). It may be noted that little if any effect is indicated upon properties as a result of the intermediate anneal introduced at the 0.15 cm.(0.06 in.) thickness on all heats except 3807, 3808 and 3809. Further, no difference in structure as a result of the intermediate anneal could be detected metallographically. Finally, metallographic evaluation indicated that all alloys subjected to approximately 50 percent cold reduction to a gauge of 0.064 cm.(0.025 in.) had extremely fine grained structures which most likely contributed to the poor elevated temperature properties.

A similar effort to cold roll the 0.38 cm.(0.15 in.) specimens was carried out on the United 2-High mill. This mill was found incapable of reducing the material. The 2-High Schloemann was also tried. Pronounced cracking of the work piece was experienced on this mill.

The results of these studies indicated that warm rolling at temperatures of at least 760° C(1400° F) after consolidation is preferred over cold rolling in terms of achieving high strength, large grain size TDNiCrAl.

b. Process Development Studies

The results of the preliminary investigations formed the basis for process development of the 3.5 percent Al and 5.0 percent Al alloys. For the process development studies, four 45.36 kg.(100 lb.) units of each TDNiCrAl alloy were prepared. The heat numbers were 3902 through 3905 for the 3.5 percent Al alloy and 3926 through 3929 for the 5.0 percent Al alloy. In addition, two sheet bar segments from the extrusion development program were made available for rolling to sheet gauges.

TABLE 30

MECHANICAL PROPERTIES OF COLD ROLLED TDNiCrAl SHEET

		Room Temperature			1093° C			Stress Rupture 1093° C	
Heat No.	Gauge cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	UTS MN/m^2	YS MN/m^2	Elong. %-cm.	Hrs. @ 27.6 MN/m^2	105° Bend
3806	0.064	905.3	568.4	15.5	66.1	59.9	20.0	0.3	0.6T
3807*	0.064	979.8	686.9	15.5	67.5	57.9	18.0	1.4	0.6T
3808*	0.064	1038.3	750.3	17.5	65.5	61.3	17.5	2.2	1.2T
3809*	0.064	1072.1	785.5	17.0	62.0	52.4	22.0	0.7	0.6T
3810	0.064	918.4	621.5	23.0	68.9	67.5	14.5	0.5	0.3T
3811	0.064	912.2	613.9	14.5	60.6	60.6	18.0	0.2	0.3T
3812	0.064	1070.7	797.9	15.5	33.8	31.7	37.5	0.1	0.3T
3816	0.064	876.4	557.4	12.0	67.5	67.5	4.5	0.5	1.2T
3848	0.064	1005.2	668.3	23.0	55.8	51.0	18.5	0.5	0.3T
3849	0.064	1156.1	870.9	15.0	37.2	35.1	25.5	0.1	0.3T

*not heat treated at 0.152 cm.

TABLE 30

MECHANICAL PROPERTIES OF COLD ROLLED TDNiCrAl SHEET

		Room Temperature			2000° F			Stress Rupture 2000° F	
Heat No.	Gauge in.	UTS ksi	YS ksi	Elong. %-in.	UTS ksi	YS ksi	Elong. %-in.	Hrs. @ 4000 psi	105° Bend
3806	0.025	131.4	82.5	15.5	9.6	8.7	20.0	0.3	0.6T
3807*	0.025	142.2	99.7	15.5	9.8	8.4	18.0	1.4	0.6T
3808*	0.025	150.7	108.9	17.5	9.5	8.9	17.5	2.2	1.2T
3809*	0.025	155.6	114.0	17.0	9.0	7.6	22.0	0.7	0.6T
3810	0.025	133.3	90.2	23.0	10.0	9.8	14.5	0.5	0.3T
3811	0.025	132.4	89.1	14.5	8.8	8.8	18.0	0.2	0.3T
3812	0.025	155.4	115.8	15.5	4.9	4.6	37.5	0.1	0.3T
3816	0.025	127.2	80.9	12.0	9.8	9.8	4.5	0.5	1.2T
3848	0.025	145.9	97.0	23.0	8.1	7.4	18.5	0.5	0.3T
3849	0.025	167.8	126.4	15.0	5.4	5.1	25.5	0.1	0.3T

*not heat treated at 0.060 in.

Different approaches were used in the process development of each alloy. For the 3.5% Al composition, a low sinter-consolidation temperature was employed to minimize ThO_2 growth, and rolling temperatures less than 1204° C(2200° F) were explored. It is known that rolling at this temperature causes recrystallization during rolling and a non-uniform microstructure in final gauge sheet. Finally, low reductions per pass were utilized in rolling from plate gauges to final gauges. Two finish gauges 0.126 cm. and 0.038 cm.(0.050 in. and 0.015 in.) were examined.

The 5.0% Al alloy required a different approach since sheet cracking precludes working at lower temperatures. A sinter-consolidation temperature of 1204° C(2200° F) was chosen; however, lower temperatures were investigated in slab rolling. Low percent reductions were also investigated for this alloy. One gauge, 0.127 cm.(0.050 in.) was produced. The defects of these studies are summarized in the following sections.

(1.) <u>3.5% Al</u>

Heat 3902 with 3.5% Al was used to explore a lower sinter-consolidation temperature to inhibit ThO_2 growth. All previous roll consolidations of TDNiCrAl had been performed at 1204° C(2200° F). A maximum temperature of 1010° C(1850° F) was chosen for Heat 3902. It was thought that temperatures less than 1010° C(1850° F) would not be practical from a yield standpoint. Examination of the consolidated 2.54 cm.(1 in.) slab showed edge cracks larger than those previously found. In addition, small tears were present on both surfaces of the slab. The percent yield after removal of edge and surface cracks was 67%. In general, the yield and appearance of this slab was similar to TDNiCr consolidated at the same temperature.

Since material yield was an acceptable level, the three remaining units containing 3.5% Al were sinter-consolidated at the same temperature. Table 31 contains the yields for these heats, which are similar to Heat 3902. Table 31 also contains ThO_2 sizes at this stage. The lower temperature did prevent the growth normally observed by exposure at 1204° C(2200° F). These values are, however, larger than those of TDNiCr consolidated at the same temperature, which are in the range of 11.0 - 14.0 mμ.

Heat 3902 was then halved to give two pieces 40.64 cm. (16 in.) wide. Both pieces were canned in mild steel for breakdown rolling. One piece, identified as 3902-A, was pre-heated for one hour at 760° C(1400° F) and rolled on the Schloemann Mill to 0.318 cm.(0.125 in.). Cracking, however, was evident after the second pass. Rolling was continued to 0.152 cm.(0.060 in.) in order to obtain samples for microstructural characterization. It was noted that several light reductions (3-6%) were used when cracking occurred. To preclude the possibility of these low reductions causing cracking, the second piece of Heat 3902-B, was also rolled at 760° C(1400° F) with reductions of 15% per pass. Severe cracking, however, again occurred during the first several passes and processing was therefore discontinued at a thickness of 1.91 cm.(0.75 in.).

TABLE 31

SLAB YIELDS AND ThO_2 SIZES FOR 3.5% ALUMINUM ALLOY CONSOLIDATED AT 1010° C (1850° F)

Heat	% Yield	ThO_2 Size mμ
3902	66.6%	17.0
3903	61.5%	17.5
3904	61.0%	17.0
3905	57.0%	18.5

The 0.152 cm.(0.060 in.) sheet samples from 3902-A were heat treated 2 hours at 1316° C(2400° F) to examine grain size. Although the structure was uniform, indicating that recrystallization had not occurred during rolling, the average grain diameter was only 0.004 mm. Mechanical properties were therefore not obtained.

Heat 3903 was used to investigate rolling temperatures above 760° C(1400° F). Figure 24 is a flow chart depicting the processing of this slab. The unit was halved at 2.54 cm. (1.0 in.) and each half canned in stainless steel edge and end borders. Piece A was rolled at 982° C(1800° F) from 2.54 cm. (1 in.) to 0.51 cm.(0.20 in.) with reductions of 15% per pass. Recanning was performed at this point and rolling continued to 0.32 cm.(0.125 in.). The condition of the plate was excellent, with very few edge cracks. At this point, the plate was halved and one piece, identified as 3903-14A, was bare rolled at 760° C (1400° F) to 0.152 cm.(0.060 in.) and 0.076 cm.(0.030 in.). Previous bare rolling studies of TDNiCrAl had shown that fabricability increased as gauge was reduced in rolling. However, severe cracking again resulted during rolling of this sheet, and the grain sizes at both gauges were only 0.004 mm.

The remaining "A" piece at 0.32 cm.(0.125 in.) was halved and canned; one piece rolled to 0.152 cm.(0.060 in.) and 0.076 cm.(0.030 in.) at 982° C(1800° F), and another to the same gauges at 871° C(1600° F). Samples were obtained at 0.152 cm. (0.060 in.). Average reductions per pass were 4% for both

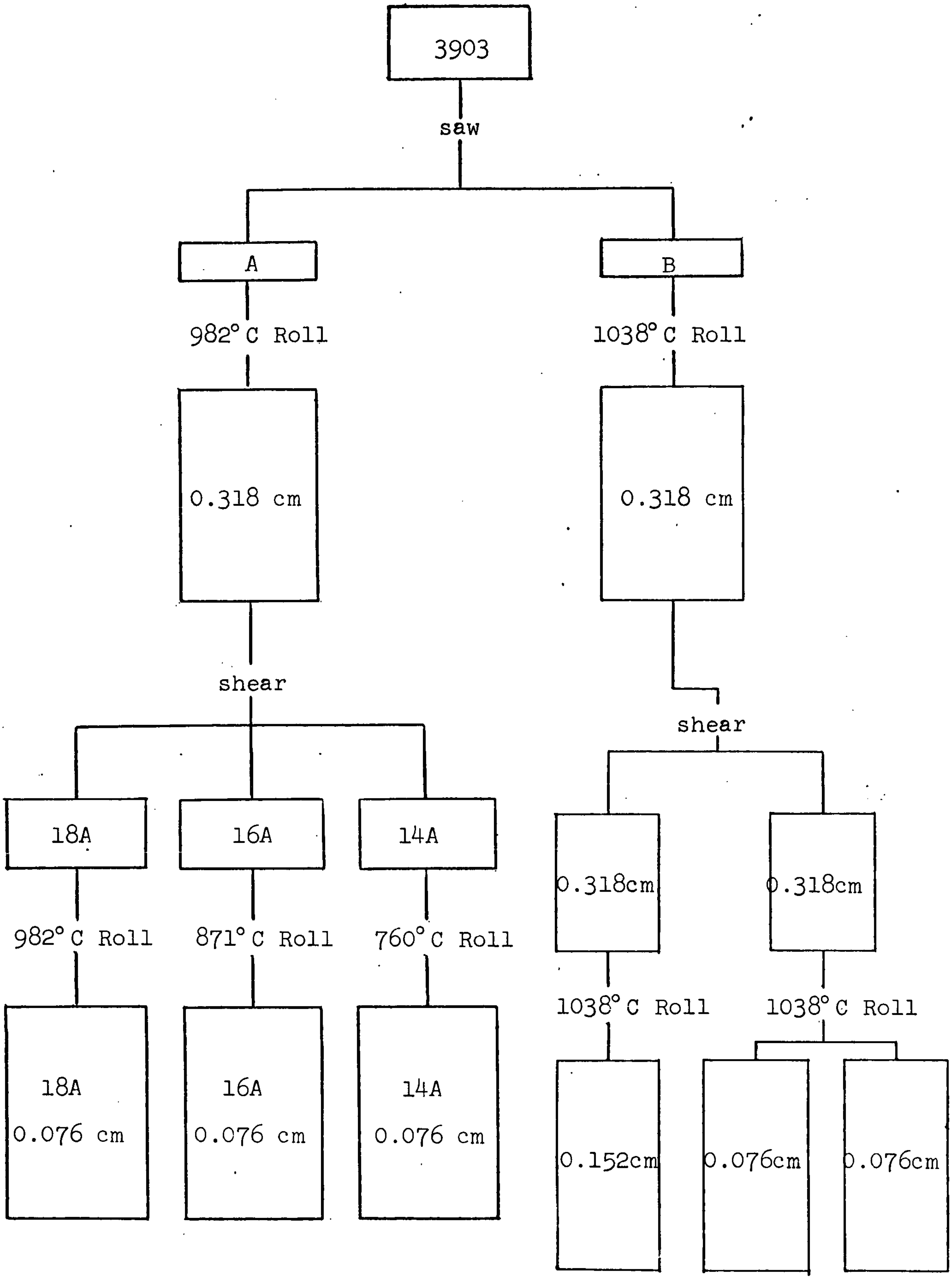


FIGURE 24. PROCESSING FLOW CHART FOR HEAT 3903

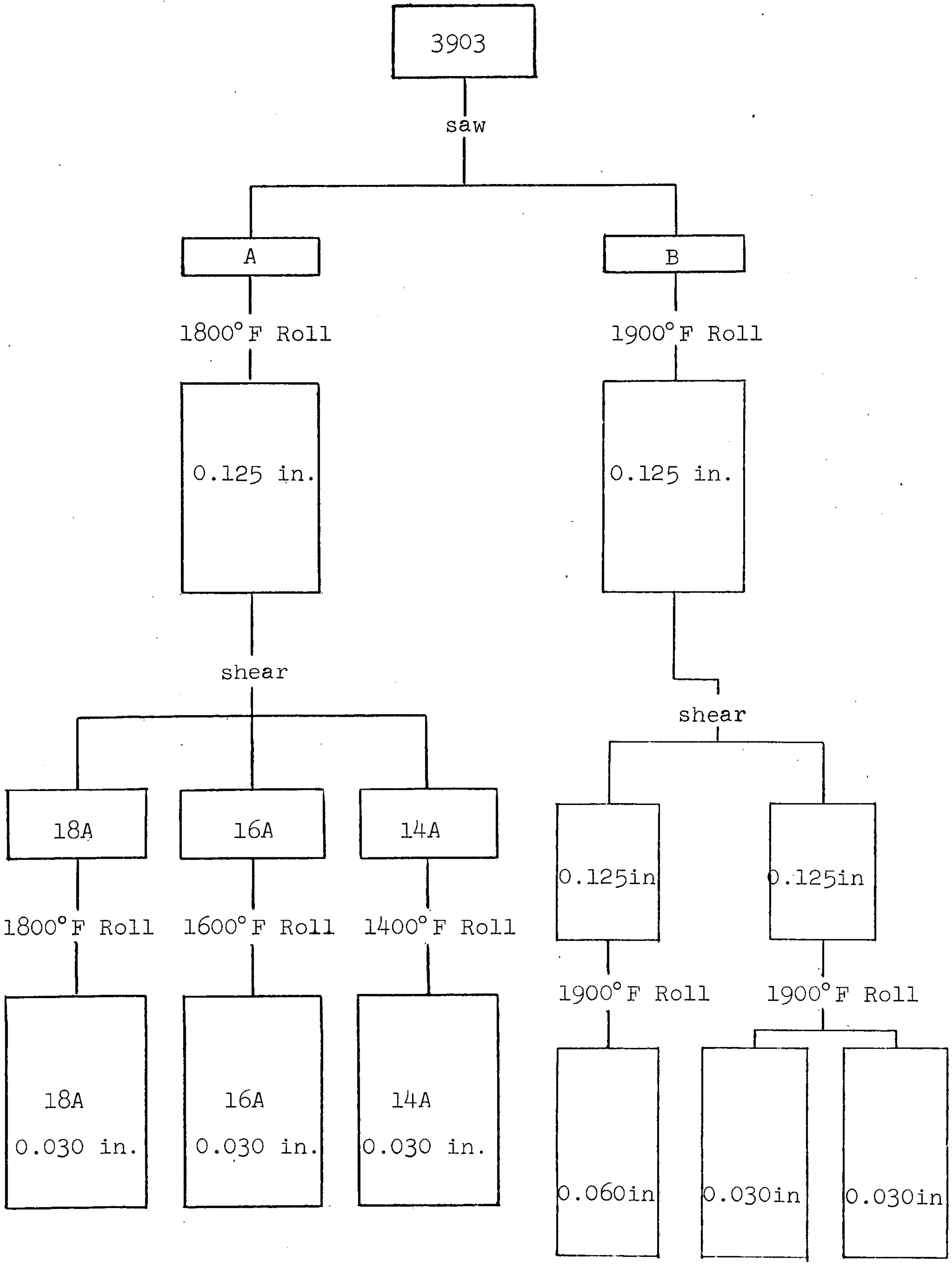


FIGURE 24. PROCESSING FLOW CHART FOR HEAT 3903

gauges, although individual passes did vary from piece to piece. Both were canned in 0.32 cm.(0.125 in.) stainless steel cover plates with edge and end borders for rolling to 0.152 cm.(0.060 in.) and 0.076 cm.(0.030 in.). Edge cracking was minimal for both rolling temperatures.

Samples at both gauges were then heat treated 2 hours at 1177° C(2150° F) and examined for microstructure. Table 32 presents the grain size data. All were found to be uniform. A trend of increasing grain size with increasing temperature and rolling reduction was observed. The most favorable condition, the sample rolled at 928° C(1800° F) to 0.076 cm.(0.030 in.) possessed an average grain diameter of 0.080 mm, a size which is equivalent to that produced in TDNiCr. The grain aspect ratio in a transverse micro was 2.0 vs. 3.0 for the TDNiCr.

Mechanical properties for these sheets are listed in Table 33. In addition to samples mentioned above, test results are given for finish gauge sheet. The operations which follow rolling are sanding to gauge, heat treating in dry hydrogen with a slow heat-up to 1177° C(2150° F) with a six hour hold, plus a 2 hour/1316° C(2400° F) heat treatment in an air/argon mixture. Samples were tested after the 1177° C(2150° F) heat treatment and after the 1316° C(2400° F) heat treatment to determine if any differences could be noted. In both cases, cooling from the peak temperature was slow. Samples heat treated in the laboratory were rapidly cooled in air.

F

TABLE 32

GRAIN SIZES FOR HEAT 3903

Sheet Identity	Rolling Temp. °C	Rolling Temp. °F	Gauge cm.	Gauge in.	Micro Grain Size mm
14A	760	1400	0.152	0.060	0.004
16A	871	1600	0.152	0.060	0.009
16A	871	1600	0.076	0.030	0.012
18A	982	1800	0.152	0.060	0.014
18A	982	1800	0.076	0.030	0.080

TABLE 33

MECHANICAL PROPERTIES OF TDNiCrAl HEAT 3903

			Room Temperature			1093° C			1093° C Stress Rupture				
Heat No.*	Gauge cm.	Rolling Temp. °C	UTS MN/m²	YS MN/m²	% Elong.	UTS MN/m²	YS MN/m²	% Elong.	Hrs.@ 31.0 MN/m²	Step Load MN/m²	Total Hours	% Elong.	Min. Bend Radius
3903-16A	0.076	871°	1103.1	770.3	19.9	119.9	119.2	3.0	4.8	-	4.8	36.5	2.0T
3903-16AN	0.038	871°	1154.8	896.4	14.5	141.2	133.0	5.0	42.5	65.5	47.6	20.0	2.8T
3903-16AS	0.038	871°	1151.3	782.0	16.0	148.1	137.1	4.0	42.5	65.5	47.7	33.0	2.4T
3903-16A	0.152	871°	1098.3	762.7	22.5	88.2	80.6	3.0	1.9	-	1.9	5.0	2.4T
3903-18A	0.076	982°	877.1	595.3	20.0	128.8	119.9	4.5	18.8**	-	18.8	4.0	3.0T
3903-18AN	0.038	982°	1059.0	813.0	8.0	138.5	-	5.0	42.5	72.3	48.8	2.5	2.2T
3903-18AS	0.038	982°	931.5	655.2	10.5	157.8	136.4	5.5	42.5	72.3	59.9	5.0	2.7T
3903-18A	0.152	982°	1065.2	774.4	17.4	108.2	92.3	6.5	2.5	-	2.5	4.0	3.0T

*Heats	-A	Heat Treat 2 hours/1316° C, RAQ
	-AN	Heat Treat 6 hours/1177° C, Slow Cool
	-AS	Heat Treat 6 hours/1177° C, Slow Cool, plus 2 hours/1316° C, Slow Cool

**Test discontinued at 18.8 hours. No failure.

TABLE 33

MECHANICAL PROPERTIES OF TDNiCrAl HEAT 3903

			Room Temperature			2000°F			2000°F Stress Rupture				
Heat No.*	Gauge in.	Rolling Temp. °F	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Hrs. @ 4.5 ksi	Step Load ksi	Total Hours	% Elong.	Min. Bend Radius
3903-16A	0.030	1600°	160.1	111.8	19.9	17.4	17.3	3.0	4.8	-	4.8	36.5	2.0T
3903-16AN	0.015	1600°	167.6	130.1	14.5	20.5	19.3	5.0	42.5	9.5	47.6	20.0	2.8T
3903-16AS	0.015	1600°	167.1	113.5	16.0	21.5	19.9	4.0	42.5	9.5	47.7	33.0	2.4T
3903-16A	0.060	1600°	159.4	110.7	22.5	12.8	11.7	3.0	1.9	-	1.9	5.0	2.4T
3903-18A	0.030	1800°	127.3	86.4	20.0	18.7	17.4	4.5	18.8**	-	18.8	4.0	3.0T
3903-18AN	0.015	1800°	153.7	118.0	8.0	20.1	-	5.0	42.5	10.5	48.8	2.5	2.2T
3903-18AS	0.015	1800°	135.2	95.1	10.5	22.9	19.8	5.5	42.5	10.5	59.9	5.0	2.7T
3903-18A	0.060	1800°	154.6	112.4	17.4	15.7	13.4	6.5	2.5	-	2.5	4.0	3.0T

*Heats -A Heat Treat 2 hours/2400°F, RAQ
-AN Heat Treat 6 hours/2150°F, Slow Cool
-AS Heat Treat 6 hours/2150°F, Slow Cool, plus 2 hours/2400°F, Slow Cool

**Test discontinued at 18.8 hours. No failure.

Several mechanical property differences were noted. First, rapid air quenching increases the room temperature elongation by a factor of two, with decrease in the room temperature strengths. Elevated temperature strengths are in fair agreement with the grain sizes reported in Table 32. The 1093° C(2000° F) properties are higher for material subjected to the multiple plant heat treatment. Inspection of the microstructures revealed coarser grains for this material; it is believed that this accounts for the added strengthening.

The 0.152 cm.(0.060 in.) sheet did not meet the 31 MN/m^2 (4.5 ksi) for 20 hours stress rupture requirement. However, if specimens had been subjected to the plant heat treatments, an increase in grain size and strength may have resulted.

Elevated temperature properties of the 0.038 cm. (0.015 in.) sheet equal or exceed those of TDNiCr. All 1093° C (2000° F) tensile strengths are above 138.7 MN/m^2 (20.0 ksi). Of particular interest is the stress rupture behavior of these specimens. Both of the samples produced by rolling at 871° C (1600° F) exceeded 20 hours at 31.01 MN/m^2 (4.5 ksi) and were step loaded to 65.5 MN/m^2 (9.5 ksi). However, percent elongations at fracture were 20.0 and 33.0%, indicating that this material is only marginally better in creep than previous TDNiCrAl. The material rolled at 982° C(1800° F), however, exhibited 2.5% and 5.0% elongation at fracture and were step loaded to 72.3 MN/m^2 (10.5 ksi).

There was an indication that as rolling temperature was increased up to 982°C(1800°F), recrystallized grain size increased on the 3.5% Al alloy. Since this trend did exist, one half of the 2.54 cm.(1.0 in.) slab of Heat 3903 was rolled to gauge at 1038°C(1900°F). Figure 24 presents the processing flow chart. Nominal percent reductions were the same as for sheet of 3903 rolled at 982°C(1800°F). The slab identification was 3903-B. Samples were obtained both at 0.152 cm.(0.060 in.) and 0.076 cm.(0.030 in.) and subjected to a recrystallization heat treatment of 1 hour at 1316°C(2400°F). The micro grain sizes were 0.031 mm for the 0.152 cm.(0.060 in.) material and 0.040 mm for the 0.076 cm.(0.030 in.) material. These grain diameters are not as large as the material rolled at 982°C(1800°F), and for this reason, mechanical testing was not deemed necessary. It is not clear whether this reduced grain size was a result of the increased temperature or slightly different reductions per pass. The last three passes on this sheet were nearly twice as large as those used on large grain 3903-18A.

An effort was made to reproduce the results of sheet 3903-18A on Heat 3904. In addition, the effects of heavy and light reductions were examined with this heat. Figure 25 is the processing flow chart. The 2.54 cm.(1.0 in.) slab was canned in stainless steel, heated to 982°C(1800°F), and rolled on the Schloemann Mill to 0.318 cm.(1/8 in.) using 15% reductions per pass. Since a can failure occurred at 1.27 cm.(0.5 in.) creating a small crack, the slab was saw cut in half at this point.

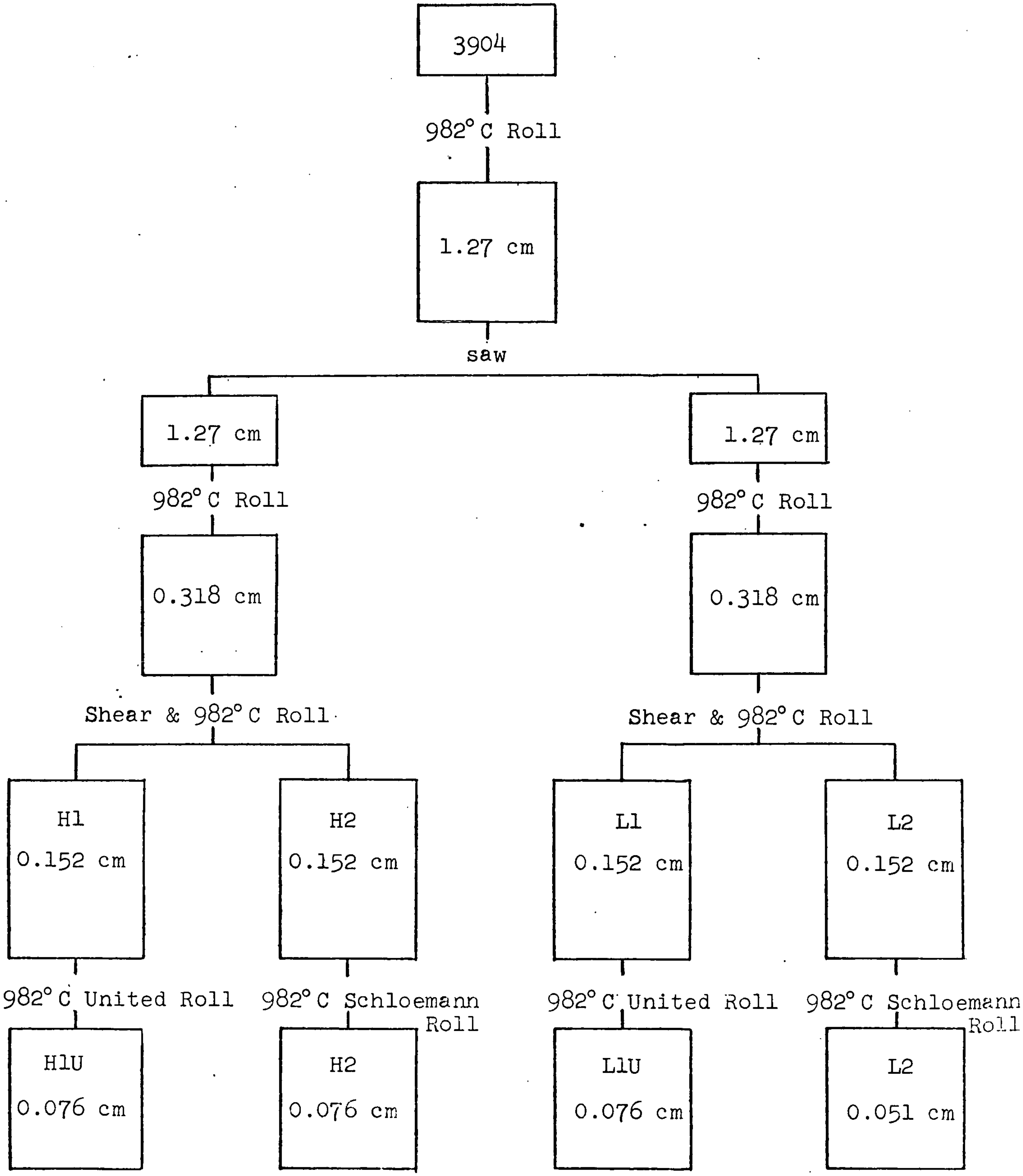


FIGURE 25. PROCESSING FLOW CHART FOR HEAT 3904

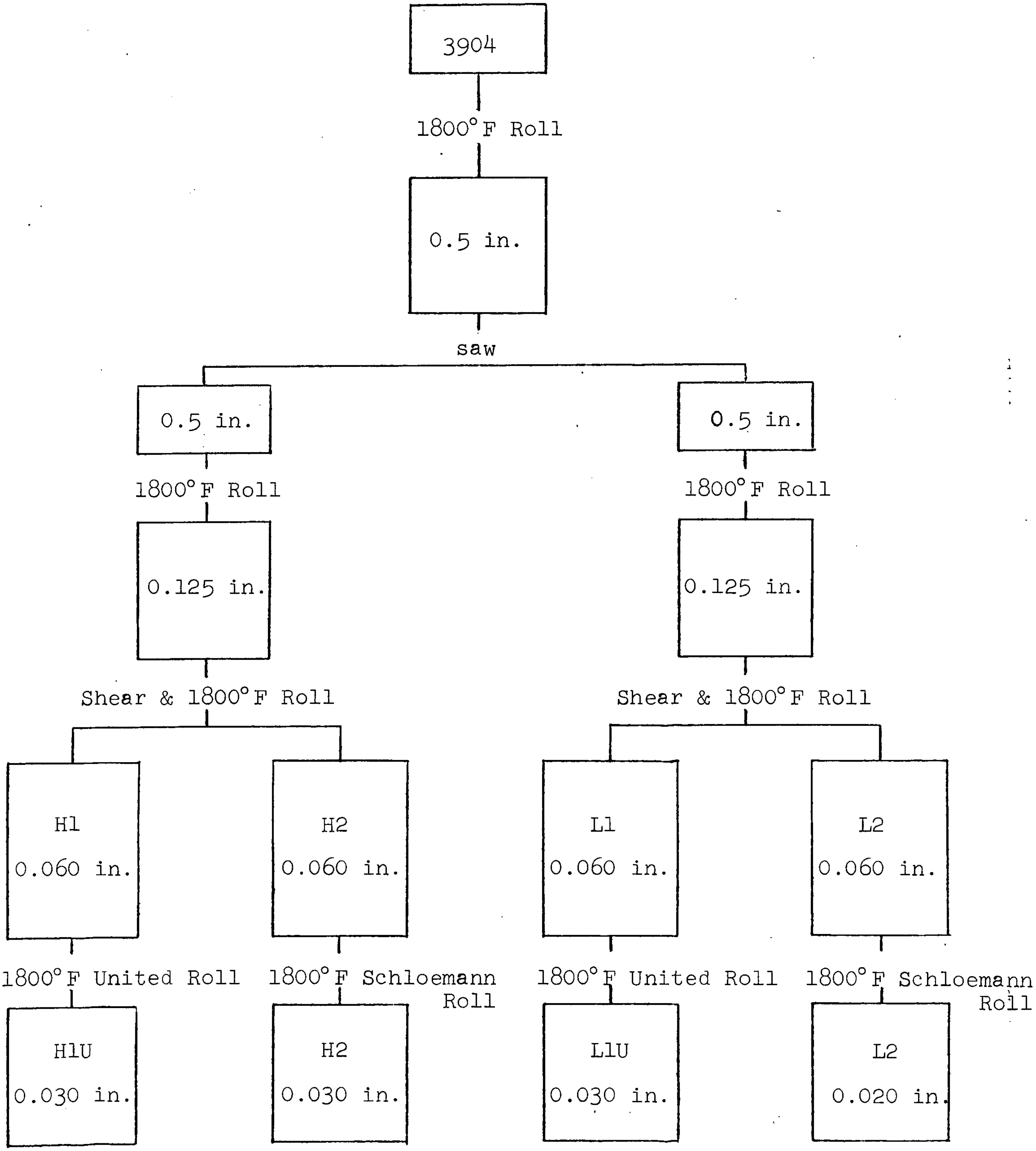


FIGURE 25. PROCESSING FLOW CHART FOR HEAT 3904

Four pieces were rolled from 0.318 cm.(1/8 in.) to 0.152 cm. (0.060 in.), two with heavy reductions and two with light reductions. The respective identifications were "-H" and "-L". One sheet of each reduction was then canned in stainless steel and rolled with either heavy or light reductions to a thinner gauge. The same roll gap settings were used for the light reductions as for Heat 3903-18A, but the Schloemann Mill was used instead of the United. This change, along with different sheet widths, prevented achievement of the exact same reductions.

Table 34 is a summary of the grain size and percent reductions obtained. The thin gauge sheet with light reductions was rolled to 0.051 cm.(0.020 in.) for finishing to 0.025 cm.(0.010 in.).

No significant differences are noted in the micro grain sizes, even though a large range in reductions was obtained. However, a comparison of these reductions with those of Heat 3903-18A showed that the latter received not only a lower average reduction, but also several very light (1-2%) passes at the end of rolling.

For this reason, sheets of both heavy and light reductions at 0.152 cm.(0.060 in.) were rolled on the United Mill to 0.076 cm.(0.030 in.). Roll gaps, although not the same as Heat 3903-18A, were adjusted to give percent reductions similar to 3903-18A. The results are also included in Table 34. The sheet rolled with nominal light reductions from

TABLE 34

GRAIN SIZES AND PERCENT REDUCTIONS FOR HEAT 3904

Sheet Identification	Gauge cm.	Gauge in.	Average % Reduction*	Grain Size mm
H1	0.152	0.060	10.0%	0.013
H2	0.152	0.060	9.0%	0.014
L1	0.152	0.060	8.0%	0.012
L2	0.152	0.060	8.0%	0.012
H2	0.076	0.030	3.5%	0.014
L2	0.051	0.020	3.6%	0.013
H1U**	0.076	0.030	3.6%	0.015
L1U**	0.076	0.030	3.5%	0.050

*For 0.152 cm.(0.060 in.) sheet, from 0.318 cm.(1/8 in.) to 0.152 cm.(0.060 in.).

For 0.076 cm.(0.030 in.) sheet, from 0.152 cm.(0.060 in.) to 0.076 cm.(0.030 in.).

**Rolled on United mill. All others rolled on Schloemann mill.

0.318 cm.(1/8 in.) to 0.076 cm.(0.030 in.) exhibits a grain size close to 3903-18A. Although the average reductions are nearly the same as for the sheets rolled on the Schloemann Mill, processing was adjusted so that the last four passes on these sheets were extremely light (1-2%).

The available mechanical property data are listed in Table 35. No data were obtained for the fine grained 0.076 cm.(0.030 in.) sheet since properties were not expected to equal those of 3903-18A. The 0.152 cm.(0.060 in.) sheet with a grain size of approximately 0.013 mm, although having high elevated temperature tensile strength, does not perform well in stress rupture testing. As expected, 3904-L1U (light reduction) at 0.076 cm.(0.030 in.) is nearly as strong as 3903-18A in stress rupture. An even greater strength probably would have been achieved on sanded, plant heat treated material. Significant increases were noted on 3903-18A after finishing and multiple plant heat treatments.

The remaining heat of the 3.5% Al alloy, 3905, was used to supply 2.61 meters^2 (28 ft.^{2}) of 0.038 cm. (0.015 in.) material to NASA. Based on the processing experience of the previous heats, a 982°C(1800°F) rolling temperature with low reductions per pass from 0.318 cm. (1/8 in.) to 0.076 cm.(0.030 in.) was chosen. A total of eight sheets were produced, four in the recrystallized condition, and four in the unrecrystallized condition. An oxide free surface was requested, which necessitated elimination

TABLE 35

MECHANICAL PROPERTIES OF HEAT 3904

Sheet Ident.	Gauge cm.	Room Temperature UTS MN/m²	Room Temperature YS MN/m²	Room Temperature % Elong.	1093° C UTS MN/m²	1093° C YS MN/m²	1093° C % Elong.	1093° C Stress Rupture Hours at 31.0 MN/m²	% Elong.	Min. Bend Radius
3904-H	0.152	1156.1	885.4	12.0	140.6	134.4	4.5	3.6	3.0	3.5T
3904-L	0.152	1163.7	871.6	15.5	110.9	104.7	5.0	1.1	3.0	3.5T
3904-H1U	0.076	982.5	658.0	17.0	128.8	122.6	5.5	4.9	2.5	3.0T
3904-L1U	0.076	954.3	720.0	13.0	133.7	120.6	4.5	>66.7	8.0	3.0T

- -

Sheet Ident.	Gauge in.	Room Temperature UTS ksi	Room Temperature YS ksi	Room Temperature % Elong.	2000° F UTS ksi	2000° F YS ksi	2000° F % Elong.	2000° F Stress Rupture Hours at 4.5 ksi	% Elong.	Min. Bend Radius
3904-H	0.060	167.8	128.5	12.0	20.4	19.5	4.5	3.6	3.0	3.5T
3904-L	0.060	168.9	126.5	15.5	16.1	15.2	5.0	1.1	3.0	3.5T
3904-H1U	0.030	142.6	95.5	17.0	18.7	17.8	5.5	4.9	2.5	3.0T
3904-L1U	0.030	138.5	104.5	13.0	19.4	17.5	4.5	>66.7	8.0	3.0T

of the 6 hour/1177° C(2150° F) hydrogen preheat treatment to form an aluminum oxide film. Instead, a single 2 hour/1304° C (2400° F) recrystallization heat treatment was performed, which aided in grinding to 0.038 cm.(0.015 in.). Mechanical properties were obtained on two sheets with the largest grain size, and are included in Table 36. Again, the excellent results achieved earlier on 3903 were reproduced.

One segment of an extrusion (Heat 3915) was made available for rolling to sheet. The processing sequence for this extrusion was sinter at 1177° C(2150° F), upset at 1093° C (2000° F) and extrude at 1040° C(1900° F), followed by decanning and conditioning. Subsequent to these operations, a 3.97 cm. x 10.16 cm. x 45.72 cm.(1-9/16 in. x 4 in. x 18 in.) slab was cut, canned in stainless steel cover plates and rolled at 982° C(1800° F) with 15% reduction per pass to 0.32 cm. (0.125 in.). At this point, it was decanned, recanned in stainless steel, and rolled at the same temperature with 4% reductions per pass to 0.152 cm.(0.060 in.) and 0.076 cm. (0.030 in.). Samples were obtained at 0.152 cm.(0.060 in.)

Mechanical properties and grain sizes are presented in Table 37 for sheet sanded to gauge and subjected to the standard heat treatments.

Grain sizes are not as large as those produced on the 0.038 cm.(0.015 in.) sheet of Heat 3903; consequently, mechanical properties are not equivalent. Processing of this extrusion, however, did differ from Heat 3903. The consolidation technique was different, the peak temperature was

TABLE 36

MECHANICAL PROPERTIES OF HEAT 3905 AT 0.038 cm. (0.015 in.)

	Room Temperature			1093° C			1093° C Stress Rupture				
Ident.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Hours at 31.0 MN/m^2	Step Load MN/m^2	Total Hours	% Elong.	Min. Bend Radius
3905-A1	807.4	626.3	13.5	115.0	109.6	2.0	24.0	51.7	27.0	6.0	3.0T
3905-4	1087.9	726.9	10.0	115.8	108.2	2.0	24.0	58.6	28.3	10.0	3.0T

	Room Temperature			2000° F			2000° F Stress Rupture				
Ident.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Hours at 4.5 ksi	Step Load ksi	Total Hours	% Elong.	Min. Bend Radius
3905-A1	128.8	90.9	13.5	16.7	15.9	2.0	24.0	7.5	27.0	6.0	3.0T
3905-4	157.9	105.5	10.0	16.8	15.7	2.0	24.0	8.5	28.3	10.0	3.0T

TABLE 37

GRAIN SIZES AND MECHANICAL PROPERTIES FOR HEAT 3915

	Room Temperature			1093° C			Stress Rupture			
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Hours at 31.0 MN/m^2	% Elong.	Min. Bend Radius	Grain Size mm
0.038	1223.0	870.9	10.5	115.8	113.0	5.0	7.5	12.1	2.0	.013
0.127	1214.0	822.0	15.0	121.3	105.4	8.0	1.9	4.0	3.0	.008

- -

	Room Temperature			2000° F			Stress Rupture			
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Hours at 4.5 ksi	% Elong.	Min. Bend Radius	Grain Size mm
0.015	177.5	126.4	10.5	16.8	16.4	5.0	7.5	12.1	2.0T	.013
0.050	176.2	119.3	15.0	17.6	15.3	8.0	1.9	4.0	3.0T	.008

1177° C(2150° F) which caused the average ThO_2 particle size to increase to 25 mμ, and the initial thickness of the slab was 23.81 cm.(1.5 in.) vs. 2.54 cm.(1.0 in.) for 3903.

The chemical analyses of these heats are presented in Table 38. Heat 3902 was not analyzed since no useable material was made. With the exception of two chromium analyses, all are close to the nominal values. Carbon and sulfur impurity levels are about the same as TDNiCr.

(2.) 5.0% Al

A different approach was taken toward processing of the Ni-16%Cr-5.0%Al-2%ThO_2 alloy. Experience has shown that higher working temperatures are necessary to avoid cracking.

Processing was initiated on Heat 3926. A sinter-consolidation temperature of 1204° C(2200° F) was utilized, causing the ThO_2 to grow to 24-25 mμ. Decanning of the slab at the 2.54 cm.(1.0 in.) stage revealed less surface and edge cracks than found on the 3.5% Al alloy heats. The percent yield after conditioning, however, was only 61.4%. Small surface cracks appeared during milling. An adjustment in the method of clamping, however, eliminated the cracking. The slab was then halved across the length and canned in stainless steel. One half was rolled on the Schloemann Mill at 982° C(1800° F) using 15% reductions per pass. A can failure occurred on the second pass, causing a large crack to form in the slab. The slab was conditioned, recanned, and rolled to 0.64 cm.(0.25 in.).

TABLE 38

CHEMICAL ANALYSES OF 3.5% Al ALLOYS

Nominal Composition Ni-16.0%Cr-3.5%Al-2.0%ThO_2
All are weight percents

Heat	% Cr	% Al	% ThO_2	C ppm	S ppm
3903	17.75	3.99	2.00	392	38
3904	15.68	3.32	1.98	378	37
3905	17.58	3.68	2.12	418	43

The remaining half of Heat 3926 was also rolled at 982° C(1800° F) using the same procedures as for the first piece. Decanning was performed at 0.318 cm.(0.125 in.). The plate contained only small edge cracks. It was then recanned and rolled at 982° C(1800° F). Major cracks, however, occurred at 0.216 cm. (0.085 in.). A small piece was sheared at this gauge and rolled to 0.153 cm.(0.060 in.) at 4% per pass for evaluation.

Samples of Heat 3926 were subjected to a 2 hour/ 1316° C(2400° F) heat treatment and evaluated for mechanical properties and microstructures. Although the grain size was uniform, the average diameter was only 0.013 mm. The mechanical properties listed in Table 39, do not equal the best obtained on the 3.5% alloy. However, creep rupture strength is good, with only 2% elongation at fracture in 66.2 hours at 31.0 MN/m^2 (4.5 ksi).

The remaining three units were also sinter-consolidated at 1204° C(2200° F), giving the yields shown in Table 40. As is evident, after the proper conditioning practice was found, the 2.54 cm.(1 in.) yields improved.

Since rolling difficulties were experienced on material processed at 982° C(1800° F), a 1093° C(2000° F) temperature was chosen for Heat 3927. Processing was as close to 3926 as possible, with the exception of temperature. However, again a micro grain size of only 0.013 mm was achieved.

The low reduction, low temperature approach, having shown no promise for the 5.0% alloy, was replaced with a higher

TABLE 39

MECHANICAL PROPERTIES OF TDNiCrAl HEAT 3926, 0.152 cm. (0.060 in.)

Room Temperature					1093° C (2000° F)					1093° C (2000° F) Stress Rupture		
UTS		YS		%	UTS		YS		%	Hours at	%	Min. Bend
MN/m^2	ksi	MN/m^2	ksi	Elong.	MN/m^2	ksi	MN/m^2	ksi	Elong.	31.0 MN/m^2 (4.5 ksi)	Elong.	Radius
1023.2	198.5	804.8	116.8	8.0	91.6	13.3	83.4	12.1	7.0	66.2	2.0	3.6T

TABLE 40

SLAB YIELDS FOR Ni-16Cr-5Al-2ThO$_2$

Heat	2.54 cm. (1.0 in.) Yield %
3926	61.4
3927	78.6
3928	78.5
3929	79.5

temperature route for the remaining units. Rolling temperatures of 1148°C(2100°F) and 1204°C(2200°F) were chosen for Heats 3928 and 3929 respectively. It was thought that the increased Al content of this alloy might retard recrystallization during rolling. Previously, recrystallization during hot working was only observed on alloys of 4.0% Al and less. Heavy reductions of an average of 25% were utilized since the material is readily worked at these temperatures. The material was successfully rolled to 0.152 cm.(0.060 in.).

Table 41 presents the grain sizes obtained on these heats, and both are similar to Heat 3926.

One portion of extrusion 3925 (Ni-16Cr-5Al-2ThO_2) was made available for rolling to sheet. It was canned in stainless steel, rolled at 1204°C(2200°F) with 25% reductions to 0.152 cm.(0.060 in.). The initial thickness of the slab was 3.80 cm.(1.50 in.) versus only 2.54 cm.(1.0 in.) for the roll consolidated units. Evaluation of sheet at 0.152 cm. (0.060 in.) showed that recrystallization apparently occurred during rolling. Surface bands 0.030 cm.(0.012 in.) thick contained fine grains 0.013 mm while the center grain size was .100 mm.

Mechanical properties were obtained on the additional 5.0% Al alloy heats and these results are presented in Table 42. Neither has a 20 hour life at 31.0 MN/m^2 (4.5 ksi) at 1093°C(2000°F).

TABLE 41

GRAIN SIZES FOR HEATS 3928 AND 3929, 5.0% Al ALLOYS

Heat	Gauge		Micro Grain Size
	cm.	in.	
3928	0.152	0.060	0.019 mm
3929	0.152	0.060	0.015 mm

TABLE 42

MECHANICAL PROPERTIES OF TDNiCrAl HEATS 3928 AND 3925 AT 0.152 cm.(0.060 in.)

	Room Temperature					1093° C (2000° F)					1093° C (2000° F) Stress Rupture	
	UTS		YS		%	UTS		YS		%	Hours at	%
Heat	MN/m²	ksi	MN/m²	ksi	Elong.	MN/m²	ksi	MN/m²	ksi	Elong.	31.0 MN/m² (4.5 ksi)	Elong.
3928	1128.6	163.8	787.5	114.3	13.0	92.3	13.4	81.3	11.8	7.0	5.3	1.0
3925	1063.8	154.4	751.7	109.1	8.0	89.6	13.0	84.7	12.3	5.5	3.0	3.5

Chemical analyses are shown in Table 43. One chromium was high and one aluminum level was low. Other levels, however, were within limits.

Although strength levels for the 5.0% alloy were not as high as desired, this does not mean that high strength is unattainable. It was shown that a 3.5% Al addition does not reduce strength below TDNiCr if a large grain structure is produced. There is no reason to suspect that the addition of 1.5% more aluminum would drastically reduce strength.

The primary effect of aluminum additions is probably on thermomechanical processing. Certain alterations, it was shown, must be made to successfully fabricate the material, and these can cause the reduction in grain size and strength. Compensating measures, such as reducing ThO_2 size or reductions per pass, must be made. It is the pinpointing of these measures that will improve the strength of the alloy under study. With only four units for development of a process, the key parameters were not found for the 5.0% alloy.

4. Creep Tests

Creep tests were run on three samples of Heat 3903 with 3.5% Al and these results are shown in Table 44. As expected, the creep strength is at least equal to standard TDNiCr and double previous TDNiCrAl tests. The stress to produce .5% creep in 100 hours at 1093° C(2000° F) was between 27.6 MN/m^2 (4.0 ksi) and 34.4 MN/m^2 (5.0 ksi).

TABLE 43

CHEMICAL ANALYSES OF 5.0% Al ALLOY

Nominal Composition Ni-16.0%Cr-5.0%Al-2.0%ThO_2
All are weight percents

Heat	% Cr	% Al	% ThO_2	C ppm	S ppm
3926	15.97	5.01	2.01	426	27
3927	17.37	4.55	2.11	340	25
3928	15.37	4.43	2.21	158	37
3929	15.64	4.75	2.20	356	33

TABLE 44

CREEP TEST RESULTS FOR 3.5% Al ALLOY - 0.038 cm.(0.015 in.) GAUGE

All tests at 1093°C(2000°F)

Ident.	Stress MN/m^2	Stress ksi	Hours	% Creep
3903-18AS	34.5	5.0	86.0	.49
3903-18AS	34.5	5.0	114.0	.44
3903-18AS	27.6	4.0	110.8	.55

Individual sheets in Heats 3904 and 3905 probably have equivalent creep strengths since their grain sizes were similar to the above sheet.

No creep testing was performed on the 5.0% alloy since no improvement over previous material was expected. As previously mentioned, the stress to produce .5% creep in 100 hours at 1093° C(2000° F) is estimated to be 13.8 MN/m^2 (2.0 ksi).

5. Production of TDNiCrAl Flat Product

During the Part II program a total of 136.47 kg.(300.6 lbs.) of TDNiCrAl sheet and plate was shipped to NASA or NASA designated contractors.

Individual piece inventories for these shipments are included in Appendix F of this report.

III - TDNiCrAl EXTRUSION DEVELOPMENT

Two types of extruded product were studied as part of the development during Part II. The initial Part II program called for development of fasteners of both TDNiCr and TDNiCrAl alloys. Exploratory extrusions were made on a series of alloy variations of both TDNiCr and TDNiCrAl. These are reported here-in and the product of these extrusions was shipped to NASA-Lewis. No fastener studies were made.

The modified Part II program called for the preparation of a series of billets of the two selected alloys, Ni-16Cr-3.5Al-2ThO_2 and Ni-16Cr-5.0Al-2ThO_2. A number of these were extruded to rectangular bar product for evaluation as potential gas turbine engine components. The goal was to develop sufficient processing technology to demonstrate the potential of achieving structure and properties comparable to commercial TDNiCr in the TDNiCrAl alloys. The product of these extrusions and five densified billets, ready for extrusion, were shipped to NASA-Lewis for continuation of the development.

1. Processing and Properties

a. Fastener Stock

Four 36.30 kg.(80 lb.) billets were initially prepared of TDNiCr powder of varying thoria size and content to provide bar stock for fastener development.

The billets were sintered at 1177° C(2150° F) and were upset consolidated at Armco, Advanced Materials Division. They were then decanned and machined to provide bare billets for extrusion to bar.

The billets were induction heated to a temperature of 1093° C(2000° F) and extruded using glass lubrication through a 6 port multihole die with three 0.76 cm.(0.30 in.) diameter and three 1.14 cm.(0.45 in.) diameter holes.

The extrusions were satisfactory with no surface tearing and a minimum of longitudinal striations. There was no die wash.

Thoria size measurements were made on the billets in the consolidated condition and on the bars both as-extruded and after 1343° C(2450° F) heat treatment:

Heat	Nominal	Thoria Size mμ			Heat Treated
No.	% ThO_2	Powder	Consolidated	As-Extruded	Extrusion
3818	1.6	18.5	18.0	22.0	27.0
3819	1.2	10.5	17.0	20.0	21.0
3820	2.2	10.0	17.0	20.0	24.0
3821	1.8	16.0	16.5	20.0	24.0

Metallographic and mechanical property evaluation showed only Heats 3820 and 3821 satisfactory in recrystallization response to warrant their use in fastener manufacture. These two heats were extruded at a lower strain rate, and fully recrystallized in a 1343° C(2450° F) heat treatment. The other two heats would only partially recrystallize. It is thought that the higher strain rate results in a higher actual extrusion temperature and thereby less residual stored energy to effect recrystallization in subsequent heat treating. Mechanical property data obtained on the extrusions is summarized in Table 45. From the tensile properties and microstructural observations, the product from Heats 3820 and 3821 were judged to be comparable to commercial TDNiCr material.

C3

Four additional billets of TDNiCr were processed to bar by bare extrusion of upset billet stock. Extrusion of three of the four were successful. Heat 3833, which was extruded through a three hole 2.1 cm.(0.825 in.) die exhibited considerable surface cracking. This behavior was unusual for TDNiCr and may have been caused by a can failure during heatup of the canned billet for the prior upset-consolidation step. Micro-examination of the torn areas showed considerable oxide contamination.

The nominal billet compositions and extrusion conditions are given in Table 46. Mechanical properties of the three successful extrusions are listed in Table 47. These properties were measured after heat treatment at 1340° C(2450° F) for 1 hour. Overall, the 1093° C(2000° F) properties appear comparable to commercial TDNiCr. At room temperature the yield strength for most of the extruded rod was lower than that of TDNiCr. Sufficient data were not obtained to identify the effects, if any, of the variations in ThO_2 content.

Six billets of several TDNiCrAl compositions were extruded to bar using a practice similar to that used for TDNiCr. The same 6 port multihole die used for TDNiCr billets 3844 and 3845 was used for all the extrusions. No surface tearing was observed. The extrusion temperatures and diameters are listed in Table 48. On the first extrusion, Heat 3863, the material flowed only through the three larger die ports, the smaller ports probably having been choked off with extrusion glass. This occasionally occurs with multiple ports of different sizes.

TABLE 45

MECHANICAL PROPERTIES OF EXTRUDED TDNiCr BAR

Heat No.	Extruded Dia. cm.	Condition	Test Temp. °C	UTS MN/m^2	YS MN/m^2	Elong. %	RA %
3818	0.76	As Extr.	RT	829.4	656.4	23.8	60.0
	1.14	As Extr.	RT	862.5	669.5	21.0	52.9
	0.76	HT	RT	750.1	548.8	25.0	48.8
	1.14	HT	RT	832.2	604.0	22.5	56.7
	0.76	HT	1093	95.1	94.5	21.3	24.6
	1.14	HT	1093	68.9	66.2	22.0	19.8
3819	0.76	As Extr.	RT	831.5	641.2	27.5	72.2
	1.14	As Extr.	RT	873.6	703.3	20.0	46.3
	0.76	HT	RT	777.7	664.0	28.8	57.4
	1.14	HT	RT	728.1	432.3	23.0	37.2
	0.76	HT	1093	79.3	77.9	20.0	21.4
	1.14	HT	1093	98.6	88.9	14.5	27.1
3820	0.76	As Extr.	RT	939.1	826.0	11.2	26.1
	1.14	As Extr.	RT	958.4	835.6	16.5	32.1
	0.76	HT	RT	819.8	525.4	23.8	43.9
	1.14	HT	RT	806.0	437.1	23.0	30.5
	0.76	HT	1093	115.8	115.8	22.5	29.7
	1.14	HT	1093	117.9	117.2	12.0	13.9
3821	0.76	As Extr.	RT	912.9	725.3	21.3	47.0
	1.14	As Extr.	RT	916.3	752.9	18.5	43.3
	0.76	HT	RT	806.7	557.1	30.0	51.5
	1.14	HT	RT	832.2	428.9	25.0	45.8
	0.76	HT	1093	111.7	103.4	13.8	18.9
	1.14	HT	1093	113.8	104.1	10.0	25.4

TABLE 45

MECHANICAL PROPERTIES OF EXTRUDED TDNiCr BAR

Heat No.	Extruded Dia. in.	Condition	Test Temp. °F	UTS ksi	YS ksi	Elong. %	RA %
3818	0.30	As Extr.	RT	120.3	95.2	23.8	60.0
	0.45	As Extr.	RT	125.1	97.1	21.0	52.9
	0.30	HT	RT	108.8	79.6	25.0	48.8
	0.45	HT	RT	120.7	87.6	22.5	56.7
	0.30	HT	2000	13.8	13.7	21.3	24.6
	0.45	HT	2000	10.0	9.6	22.0	19.8
3819	0.30	As Extr.	RT	120.6	93.0	27.5	72.2
	0.45	As Extr.	RT	126.7	102.0	20.0	46.3
	0.30	HT	RT	112.8	96.3	28.8	57.4
	0.45	HT	RT	105.6	62.7	23.0	37.2
	0.30	HT	2000	11.5	11.3	20.0	21.4
	0.45	HT	2000	14.3	12.9	14.5	27.1
3820	0.30	As Extr.	RT	136.2	119.8	11.2	26.1
	0.45	As Extr.	RT	139.0	121.2	16.5	32.1
	0.30	HT	RT	118.9	76.2	23.8	43.9
	0.45	HT	RT	116.9	63.4	23.0	30.5
	0.30	HT	2000	16.8	16.8	22.5	29.7
	0.45	HT	2000	17.1	17.0	12.0	13.9
3821	0.30	As Extr.	RT	132.4	105.2	21.3	47.0
	0.45	As Extr.	RT	132.9	109.2	18.5	43.3
	0.30	HT	RT	117.0	80.8	30.0	51.5
	0.45	HT	RT	120.7	62.2	25.0	45.8
	0.30	HT	2000	16.2	15.0	13.8	18.9
	0.45	HT	2000	16.5	15.1	10.0	25.4

TABLE 46

TDNiCr BAR EXTRUSIONS

Heat No.	Nominal Composition	Extrusion Temp. °C	°F	Extrusion Die	Extruded Diameters cm.	in.
3833	Ni-20Cr-1.8ThO_2	1038	1900	3 hole	1.98	0.78
3834	Ni-20Cr-1.8ThO_2	1038	1900	1 hole	3.05	1.20
3844	Ni-20Cr-1.8ThO_2	1093	2000	6 hole	1.32	0.52
					0.79	0.31
3845	Ni-20Cr-1.2ThO_2	1093	2000	6 hole	1.32	0.52
					0.79	0.31

TABLE 47

TDNiCr BAR EXTRUSIONS - MECHANICAL PROPERTIES

Heat No.	Nom. Composition	Extruded dia. cm.	Test Temp. °C	Tensile				Stress Rupture		
				UTS MN/m²	YS MN/m²	Elong. %	Redn.Area %	Initial MN/m²	Initial Hrs.	Final MN/m²
3834	Ni-20Cr-1.8ThO$_2$	3.05	RT	869.5	463.0	26.0	22.2			
			1093	104.7	102.0	5.0	2.5	37.9	22.5	44.8
3844	Ni-20Cr-1.8ThO$_2$	1.32	RT	768.9	401.7	21.7	32.8			
			1093	104.0	97.1	13.8	6.5	37.9	22.5	79.2
		0.79	RT	813.0	552.6	34.5	44.9			
			1093	106.1	101.3	8.8	10.1	48.2	115.1	68.9
3845	Ni-20Cr-1.2ThO$_2$	1.32	RT	786.1	418.9	22.5	32.6			
			1093	102.0	87.0	9.2	6.4	48.2	23.4	68.9
		0.79	RT	788.2	531.2	31.3	36.2			
			1093	127.5	121.3	8.5	13.6	48.2	115.1	68.9

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Heat No.	Nom. Composition	Extruded dia. in.	Test Temp. °F	Tensile				Stress Rupture		
				UTS ksi	YS ksi	Elong. %	Redn.Area %	Initial ksi	Initial Hrs.	Final ksi
3834	Ni-20Cr-1.8ThO$_2$	1.20	RT	125.9	67.2	26.0	22.2			
			2000	15.2	14.8	5.0	2.5	5.5	22.5	6.5
3844	Ni-20Cr-1.8ThO$_2$	0.52	RT	111.6	58.3	21.7	32.8			
			2000	15.1	14.1	13.8	6.5	5.5	22.5	11.5
		0.31	RT	118.0	80.2	34.5	44.9			
			2000	15.4	14.7	8.8	10.1	7.0	115.1	10.0
3845	Ni-20Cr-1.2ThO$_2$	0.52	RT	114.1	60.8	22.5	32.6			
			2000	14.8	12.6	9.2	6.4	7.0	23.4	10.0
		0.31	RT	114.4	77.1	31.3	36.2			
			2000	18.5	17.6	8.5	13.6	7.0	115.1	10.0

TABLE 48

TDNiCrAl BAR EXTRUSIONS

Heat No.	Nominal Composition	Extrusion Temp. °C	Extrusion Temp. °F	Extruded Diameters cm.	Extruded Diameters in.
3863	Ni-16Cr-3.2Al-2.2ThO_2	1150	2100	1.32	0.52
3865	Ni-16Cr-3.2Al-2.2ThO_2	1090	2000	1.32 0.79	0.52 0.31
3868	Ni-16Cr-3.2Al-2.2ThO_2	1200	2200	1.32 0.81	0.52 0.32
3864	Ni-16Cr-3.2Al-1.5ThO_2	1090	2000	1.27 0.74	0.50 0.29
3866	Ni-16Cr-3.2Al-1.0ThO_2	1090	2000	1.27 0.74	0.50 0.29
3867	Ni-16Cr-4Al-2.2ThO_2	1090	2000	1.27 0.74	0.50 0.29

An extrusion temperature of 1093° C(2000° F) was chosen for Heats 3864, 3866 and 3867 because more complete recrystallization in heat treatment was achieved with Heat 3863 extruded at 1150° C (2100° F) than with 3868 extruded at 1200° C(2200° F).

Heat treatment studies of the six extrusions showed the following:

Heat No.	Degree of Recrystallization in 1340° C(2450° F) Heat Treatment (1 Hour)
3863	90%
3865	80%
3868	60%
3864	100%
3866	100%
3867	50%

Mechanical properties listed in Table 49 for bar heat treated at 1340° C(2450° F) show elevated temperature strengths for the Ni-16Cr-3.2Al-1.5ThO_2 alloy (Heat 3864) very close to TDNiCr and for the 1.0% ThO_2 alloy (Heat 3866) only slightly less. The lower strengths shown for the 2.2% ThO_2 Heats 3863, 3865 and 3868 are reflective of the incomplete recrystallization obtained in these bars.

Subsequent long time (~20 hours) heat treatments of the bars showing incomplete recrystallization in standard heating times indicated completion of the recrystallization but to a finer grain size. Mechanical property tests are shown in Table 50.

TABLE 49

TDNiCrAl BAR EXTRUSIONS - MECHANICAL PROPERTIES

Heat No.	Nom. Composition	Extruded dia. cm.	% Rx	Test Temp. °C	Tensile UTS MN/m^2	Tensile YS MN/m^2	Tensile Elong. %	Tensile Redn.Area %	Stress Rupture Initial MN/m^2	Stress Rupture Initial Hrs.	Stress Rupture Final MN/m^2
3863	Ni-16Cr-3.2Al-2.2ThO$_2$	1.32	90	RT	1005.3	658.0	22.5	32.8			
				1093°	57.9	53.7	9.5	9.6			
3865	Ni-16Cr-3.2Al-2.2ThO$_2$	1.32	80	RT	1033.5	693.8	25.0	31.8			
				1093°	53.1	44.8	44.0	29.3	48.2	0.1	
		0.79	80	RT	870.8	715.2	18.8	22.4			
				1093°	74.4	71.0	2.3	30.8	48.2	0.2	
3868	Ni-16Cr-3.2Al-2.2ThO$_2$	1.32	60	RT	-	-	-	-			
				1093°	32.4	29.6	-	-			
		0.81	60	RT	-	-	-	-			
				1093°	64.8	59.9	2.7	12.5	48.2	<0.1	
3864	Ni-16Cr-3.2Al-1.5ThO$_2$	1.27	100	RT	840.5	502.5	22.4	32.2			
				1093°	84.9	76.4	2.0	15.2	48.2	23.8	
		0.74	100	RT	765.8	505.0	33.0	36.7			
				1093°	98.5	95.8	2.1	30.5	48.2	103.8	
3866	Ni-16Cr-3.2Al-1.0ThO$_2$	1.27	100	RT	673.2	364.7	32.5	34.6			
				1093°	86.3	82.0	4.9	14.5	48.2	1.7	
		0.74	100	RT	629.1	391.4	31.8	52.0			
				1093°	95.8	95.8	1.7	18.6	48.2	5.7	

TABLE 49

TDNiCrAl BAR EXTRUSIONS - MECHANICAL PROPERTIES

Heat No.	Nom. Composition	Extruded dia. in.	% Rx	Test Temp. °F	Tensile UTS ksi	Tensile YS ksi	Tensile Elong. %	Tensile Redn. Area %	Stress Rupture Initial ksi	Stress Rupture Initial Hrs.	Stress Rupture Final ksi
3863	Ni-16Cr-3.2Al-2.2ThO$_2$	0.52	90	RT	145.9	95.5	22.5	32.8			
				2000°	8.4	7.8	9.5	9.6			
3865	Ni-16Cr-3.2Al-2.2ThO$_2$	0.52	80	RT	150.0	100.7	25.0	31.8			
				2000°	7.7	6.5	44.0	29.3	7.0	0.1	
		0.31	80	RT	126.1	103.8	18.8	22.4			
				2000°	10.8	10.3	2.3	30.8	7.0	0.2	
3868	Ni-16Cr-3.2Al-2.2ThO$_2$	0.52	60	RT	-	-	-	-			
				2000°	4.7	4.3	-	-			
		0.32	60	RT	-	-	-	-			
				2000°	9.4	8.7	2.7	12.5	7.0	<0.1	
3864	Ni-16Cr-3.2Al-1.5ThO$_2$	0.50	100	RT	121.7	72.9	22.4	32.2			
				2000°	12.3	11.1	2.0	15.2	7.0	23.8	
		0.29	100	RT	115.5	73.3	33.0	36.7			
				2000°	14.3	13.9	2.1	30.5	7.0	103.8	
3866	Ni-16Cr-3.2Al-1.0ThO$_2$	0.50	100	RT	97.7	52.9	32.5	34.6			
				2000°	12.5	11.9	4.9	14.5	7.0	1.7	
		0.29	100	RT	91.3	56.8	31.8	52.0			
				2000°	13.9	13.9	1.7	18.6	7.0	5.7	

TABLE 50

MECHANICAL PROPERTIES - TDNiCrAl BAR EXTRUSIONS

Tensile Tests 1093° C (2000° F)

Heat	Bar Dia.		UTS		YS		%	%
No.	cm.	(in.)	MN/m^2	(ksi)	MN/m^2	(ksi)	Elong.	RA
3865	.76	(0.3)	88	(12.7)	88	(12.7)	3.3	26.5
3865	1.27	(0.5)	85	(12.3)	85	(12.3)	11.0	12.8
3863	1.27	(0.5)	79	(11.5)	76	(11.1)	12.0	19.8
3867	.76	(0.3)	78	(11.3)	76	(11.1)	4.1	30.9
3867	1.27	(0.5)	77	(11.2)	70	(10.1)	23.0	24.7
3868	.76	(0.3)	85	(12.3)	85	(12.3)	2.3	21.6
3868	1.27	(0.5)	82	(11.9)	79	(11.5)	15.0	13.0

Stress Rupture 1093° C (2000° F)

Heat	Bar Dia.		Stress Rupture Life		
No.	cm.	(in.)	MN/m^2	(ksi)	Hours
3865	.76	(0.3)	38	(5.5)	116
3865	1.27	(0.5)	38	(5.5)	0
3863	1.27	(0.5)	38	(5.5)	0.3
3867	.76	(0.3)	38	(5.5)	0.3
3867	1.27	(0.5)	38	(5.5)	0.1
3868	.76	(0.3)	38	(5.5)	0.8
3868	1.27	(0.5)	38	(5.5)	1.3

These preliminary studies indicate the feasibility of manufacturing TDNiCrAl rod stock by direct extrusion. Mechanical properties close to that of TDNiCr were achieved. A lower ThO_2 content than commercial TDNiCr appears desirable to aid in achieving a fully recrystallized structure.

b. Rectangular Bar Extrusions

The rectangular bar extrusion program for the two alloys, Ni-16Cr-3.5Al-$2ThO_2$ and Ni-16Cr-5.0Al-$2ThO_2$ was divided into two parts. In the first part two Ni-16Cr-3.5Al-$2ThO_2$ and four Ni-16Cr-5Al-$2ThO_2$ 90.8 kg.(200 lb.) billets were prepared for extrusion to rectangular cross section bar. The primary purpose of these was to determine the structures and properties obtainable in extruded parts. Portions of one extrusion of each alloy were rolled to sheet to study extrusion as an alternate process to roll consolidate in the manufacture of TDNiCrAl sheet.

In the second part of this program hot upset consolidated billet stock was prepared in both alloys, two billets of Ni-16Cr-3.5Al-$2ThO_2$ and six of Ni-16Cr-5Al-$2ThO_2$. Three billets were selected for extrusion experiments. The remaining five billets were prepared for NASA to be used in the production of extrusions for further studies.

In the initial extrusion campaign, two extrusions were made of each of the two alloys; Ni-16Cr-3.5Al-$2ThO_2$, Heats 3915 and 3916; Ni-16Cr-5.0Al-$2ThO_2$, Heats 3924 and 3925. These were followed by two more extrusions of the 5.0% Al alloy, Heats 3937 and 3940.

All of the billets were hydrostatically compacted, starting with 200 pounds of alloy powder, sintered in H_2 and upset-consolidated in the 20.3 cm.(8 in.) diameter extrusion container at the Armco, Advanced Materials Division. They were then decanned, machined and recanned in mild steel for extrusion in the same 20.3 cm.(8 in.) diameter container.

The sinter temperature, upset-consolidation temperature and extrusion conditions are given in Table 51. Preparation and extrusion data for three additional extrusions of the 5.0% Al alloy are also given in Table 51.

Two types of extrusion dies were used, a radiused shear die and a cone insert die. The radiused shear die had been used successfully for previous TDNi and TDNiCr extruded products. The cone insert die was used to attempt to gain more uniform deformation in a shaped or rectangular extrusion.

The billet from Heat 3925 stalled on the original extrusion attempt at 1010° C(1850° F). The billet was remachined, recanned and successfully extruded at 1093° C(2000° F).

Sections of the extruded rectangular bars from Heats 3915 and 3925 were utilized in the Rolling Development study reported in Section II-3 of this report.

Evaluation of the extrusions from the 3.5% Al alloy showed that the structures developed by recrystallization at 1345° C(2450° F) were non-uniform. The structures for Heats 3915 and 3916 were similar, despite the differences in extrusion temperature and die configuration. The structures

TABLE 51

TDNiCrAl RECTANGULAR BAR EXTRUSIONS

Heat No.	Alloy	Sinter Temperature		Upset Temperature		Extrusion Temperature		Die Shape	Extruded Bar Size	
		°C	°F	°C	°F	°C	°F		cm.	in.
3915	3.5% Al	1180	2150	1093	2000	1040	1900	Radiused Shear	4.70 x 11.05	1.85 x 4.35
3916	3.5% Al	1180	2150	1093	2000	1093	2000	Cone Insert	3.81 x 15.24	1.5 x 6
3924	5.0% Al	955	1750	1010	1800	1150	2100	Radiused Shear	4.70 x 11.05	1.85 x 4.35
3925	5.0% Al	955	1750	1010	1800	1093	2000*	Radiused Shear	4.70 x 11.05	1.85 x 4.35
3937	5.0% Al	955	1750	1010	1800	1150	2100	Cone Insert	3.81 x 15.24	1.5 x 6
3940	5.0% Al	955	1750	1010	1800	1093	2000	Cone Insert	3.81 x 15.24	1.5 x 6
3934	5.0% Al	955	1750	1010	1800	1093	2000	Cone Insert	3.81 x 15.24	1.5 x 6
3938	5.0% Al	955	1750	1010	1800	1205	2200	Cone Insert	3.81 x 15.24	1.5 x 6
3936	5.0% Al	955	1750	1010	1800	1093	2000	Cone Insert	3.30 x 11.94	1.3 x 4.7

*Stalled on first extrusion trial at 1010° C(1850° F).

were similar to those which have been sometimes experienced with the extrusion of TDNiCr rectangular extrusions. The structures are illustrated schematically in Figure 26. The structure of the central zone, which receives the minimum amount of deformation, has a medium grain size, the area surrounding it which has a greater amount of deformation has a larger grain size. The outer area is subjected to additional shear deformation due to its proximity to the die surface and has the smallest grain structure.

The microstructures from these three zones are shown in Figure 27 for the extrusion from Heat 3916.

A few tests were made to determine the elevated temperature properties of these microstructures and they are reported in Table 52. The properties for the grain structures in the center and mid areas appear to be similar but both lower than for extruded and recrystallized TDNiCr sheet bar.

Evaluation of the two 5.0% Al alloy extrusions showed that the extrusion from Heat 3924 could not be recrystallized at 1345° C(2450° F) (the highest practical temperature to avoid melting). The extrusion from Heat 3925 which was extruded at the lower temperature of 1093° C(2000° F) did recrystallize at 1345° C(2450° F). The microstructure was non-uniform across the section and was similar to that shown previously for Heat 3915.

Additional working by extrusion was limited because the force requirements at higher reductions or lower extrusion temperatures exceeded the capacity of the tools and the extrusion press. An attempt to introduce additional working by

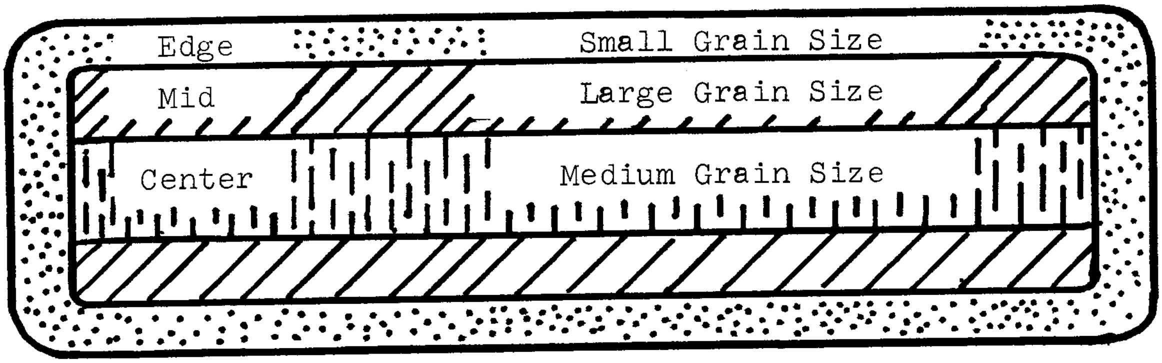
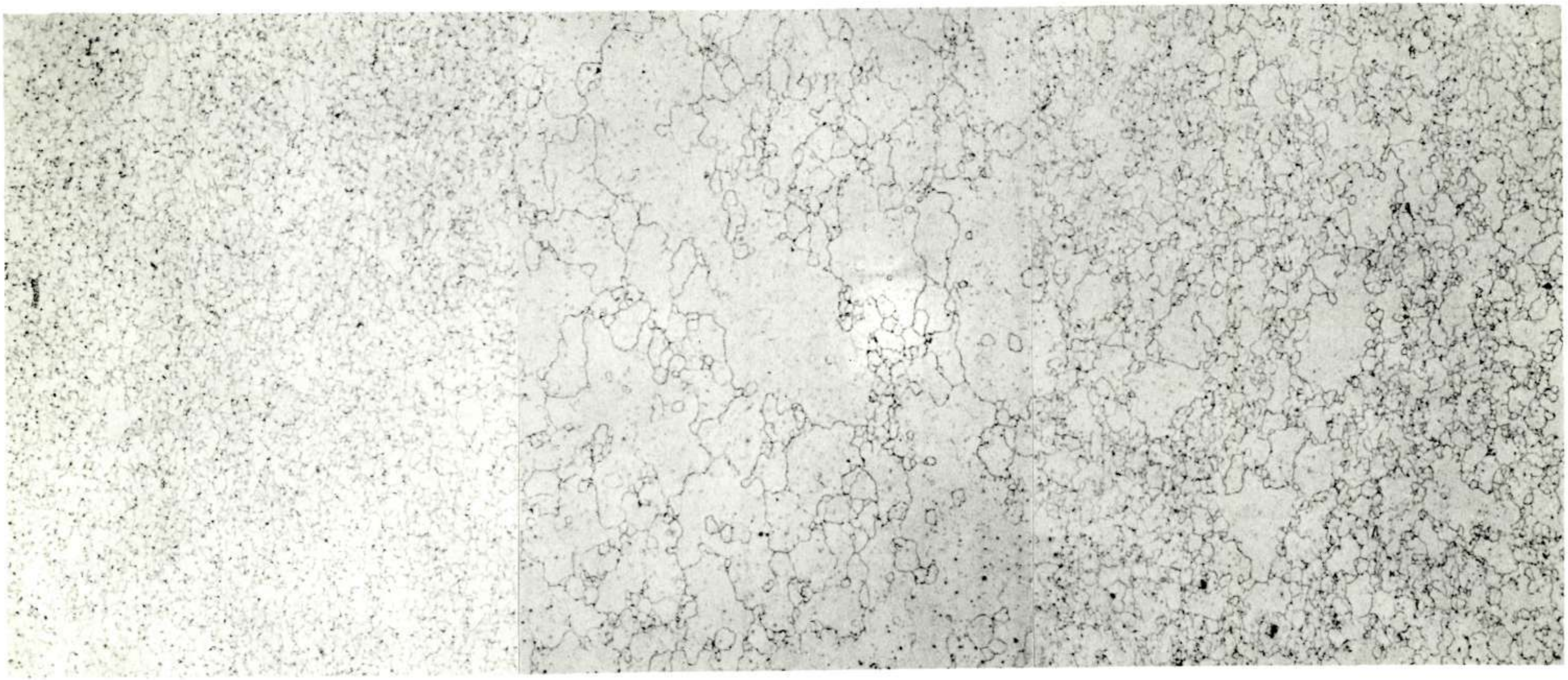


FIGURE 26

SCHEMATIC GRAIN STRUCTURES IN
RECRYSTALLIZED EXTRUDED RECTANGULAR BAR



Edge Mid Center

1-3/8" x 6" Extruded Bar Heat Treated 1345° C (2450° F) 1-1/2 Hours - 250X

FIGURE 27

MICROSTRUCTURES IN RECRYSTALLIZED EXTRUDED BAR - HEAT 3916

TABLE 52

1093° C (2000° F) TENSILE AND STRESS RUPTURE DATA - HEATS 3915 AND 3916

Heat	Sample	UTS		YS		Elong.	RA	Stress Rupture		
		MN/m²	ksi	MN/m²	ksi	%	%	MN/m²	ksi	Hours
3915	Longitudinal Mid Location	103	15.0	89	13.0	15.7	13.2			
3915	Longitudinal Center Location	100	14.5	88	12.8	14.6	14.5	48 38	7.0 5.5	0.2 1.1
3915	Transverse Center Location	89	13.0	83	12.0	7.6	9.3	38 31	5.5 4.5	0.7 1.0
3916	Longitudinal Center Location	-	-	-	-	-	-	48	7.0	0.1
3916	Transverse Center Location	-	-	-	-	-	-	38	5.5	0.8

hot rolling was made on a section of the extruded bar. An as-extruded section of Heat 3925 was canned in mild steel, heated to 1093° C(2000° F) and rolled, 15% in one pass, in the same direction as it had been previously extruded. This did result in some improvement in the microstructure after heat treatment at 1345° C(2450° F).

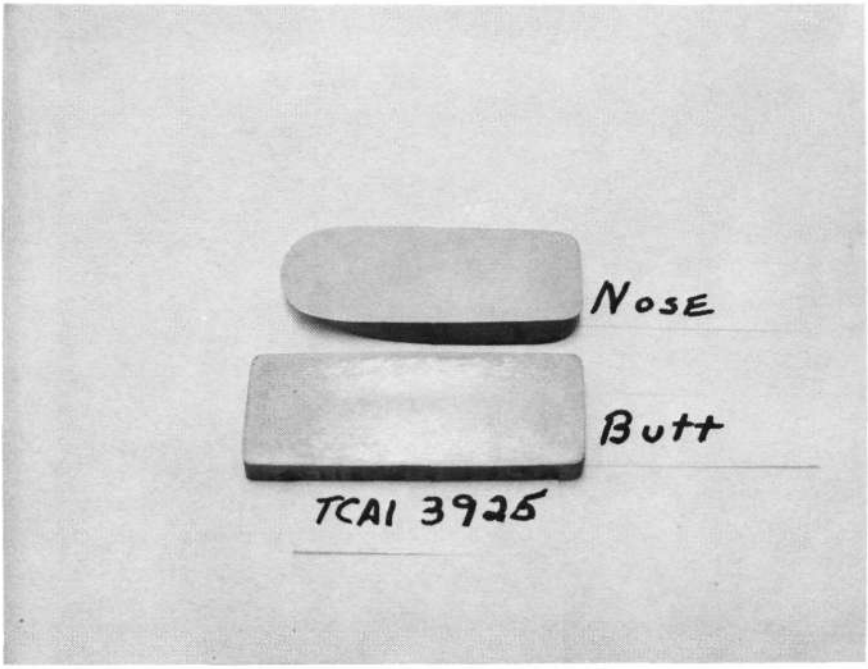
Macro sections are shown in Figure 28 and microstructures in Figures 29 and 30.

No recrystallization occurs in the nose section of the extrusion, Figure 28, because of the lack of working at the front of the extrusion. The pattern of the microstructure appears changed after the 15% rolling.

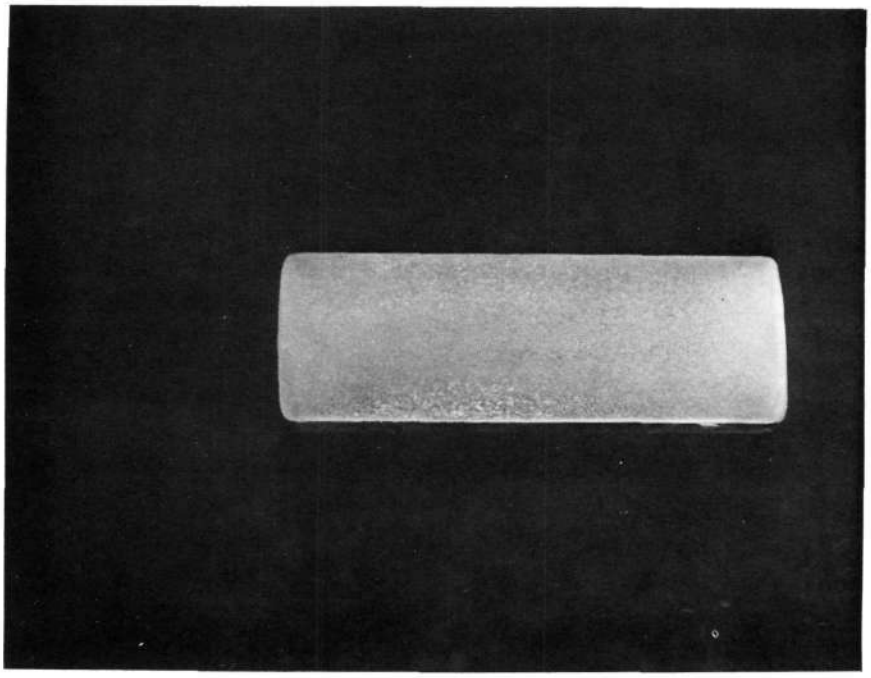
The microstructures, Figures 29 and 30, show that the grain sizes were larger in each of the areas than had been the case for Heat 3915. Improvement in uniformity can be noted due to the rolling deformation.

Tensile and stress rupture tests at 1093° C(2000° F), Table 53, did not appear to show any difference in test values. Microstructural examination of the failed test samples showed the presence of smaller grains in all the microstructures which appear to be responsible for the lower values, particularly in stress rupture.

Two billets of the 5% Al alloy were extruded through cone insert dies, Heat 3937 at 1149° C(2100° F) and Heat 3940 at 1093° C(2000° F); the preparation and extrusion data are listed in Table 51. The extrusion from Heat 3937 cracked and was not



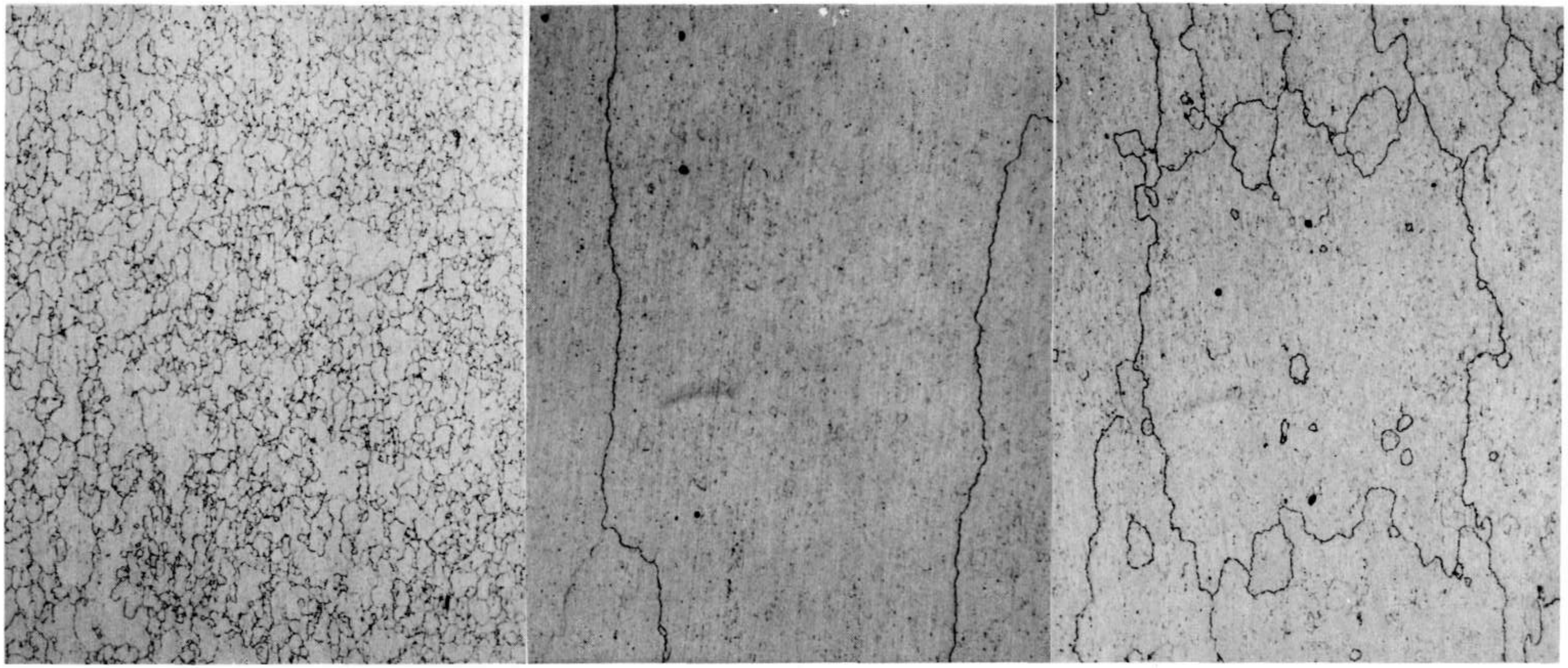
Extruded - Heat Treated 1345° C(2450° F)/1-1/2 Hours



Extruded and Rolled 15% - Heat Treated 1345° C(2450° F)/1-1/2 Hours

FIGURE 28

MACROSTRUCTURES - HEAT 3925

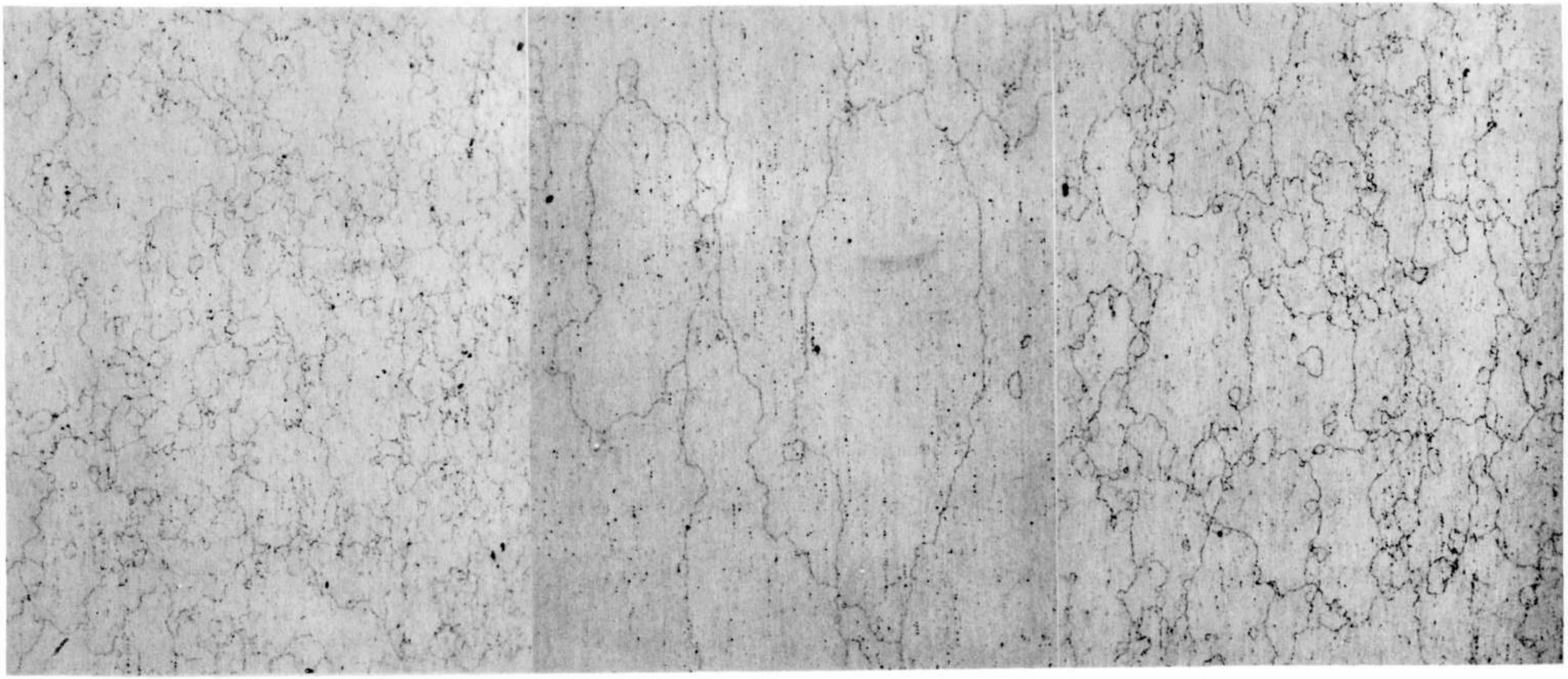


Edge Mid Center

1-3/4" x 4-1/4" Extruded Bar Heat Treated 1345° C(2450° F)/1-1/2 Hours - 250X

FIGURE 29

MICROSTRUCTURES IN RECRYSTALLIZED EXTRUDED BAR - HEAT 3925



Edge Mid Center

1-3/4" x 4-1/4" Extruded Bar-Rolled 15%-Heat Treated 1345°C(2450°F)/1-1/2 Hours - 250X

FIGURE 30

MICROSTRUCTURES IN RECRYSTALLIZED EXTRUDED AND ROLLED BAR - HEAT 3925

TABLE 53

1093° C(2000° F) TENSILE AND STRESS RUPTURE DATA - HEAT 3925

		UTS		YS		Elong.	RA	Stress Rupture		
History	Sample	MN/m^2	ksi	MN/m^2	ksi	%	%	MN/m^2	ksi	Hours
Extruded and Recrystallized	Longitudinal Mid Location	108	15.7	98	14.2	-	-	48	7.0	1.3
	Longitudinal Center Location	83	12.1	77	11.1	17.9	15.9	48	7.0	0.3
	Transverse Center Location	77	11.1	68	9.8	14.0	11.3	38	5.5	0.4
Extruded, Rolled and Recrystallized	Longitudinal Center Location	92	13.3	83	12.1	17.3	16.0	48	7.0	0.1
	Transverse Center Location	85	12.3	74	10.8	19.2	17.4	38	5.5	2.4

available for further working studies. An examination of a recrystallized cross section showed the presence of a mixed structure of large and very small grains.

The recrystallized structure from the extrusion from Heat 3940 also showed a non-uniform structure similar to that shown by the extrusion from Heat 3925. Rolling 15% at 1093° C (2000° F) appeared to cause some improvement in the uniformity of the recrystallized microstructure.

Rolling subsequent to extrusion appeared to offer some potential in obtaining a uniform grain structure. Consequently, three additional billets of the 5% Al alloy were committed to further extrusion and rolling experiments in the second part of the program. The extrusion data for these billets are also listed in Table 51.

Extrusion 3938 was made at 1205° C(2200° F) to determine whether the fine grains noted in the duplex structure of Heat 3937 could be eliminated by a higher extrusion temperature. The extrusion could not be recrystallized at 1340° C(2450° F). Subsequent rolling, 15% at 1093° C(2000° F), produced some recrystallization upon heat treatment at 1340° C(2450° F); the structure, however, was not uniform.

The extrusion from Heat 3934 was extruded under the same conditions as extrusion 3940. The same grain structure pattern was obtained on the recrystallized extruded section as for 3940. A series of rolling experiments were made, utilizing reductions of 10 and 15%, however, none were successful in eliminating the mixed structure.

Further plastic working on the extrusion process appeared to be necessary; therefore an extrusion was made with the billet from Heat 3936 utilizing a higher reduction ratio but holding the other extrusion conditions constant. The extrusion data are given in Table 51. Sections of the extrusion were also rolled 15% at 1093° C(2000° F) for evaluation.

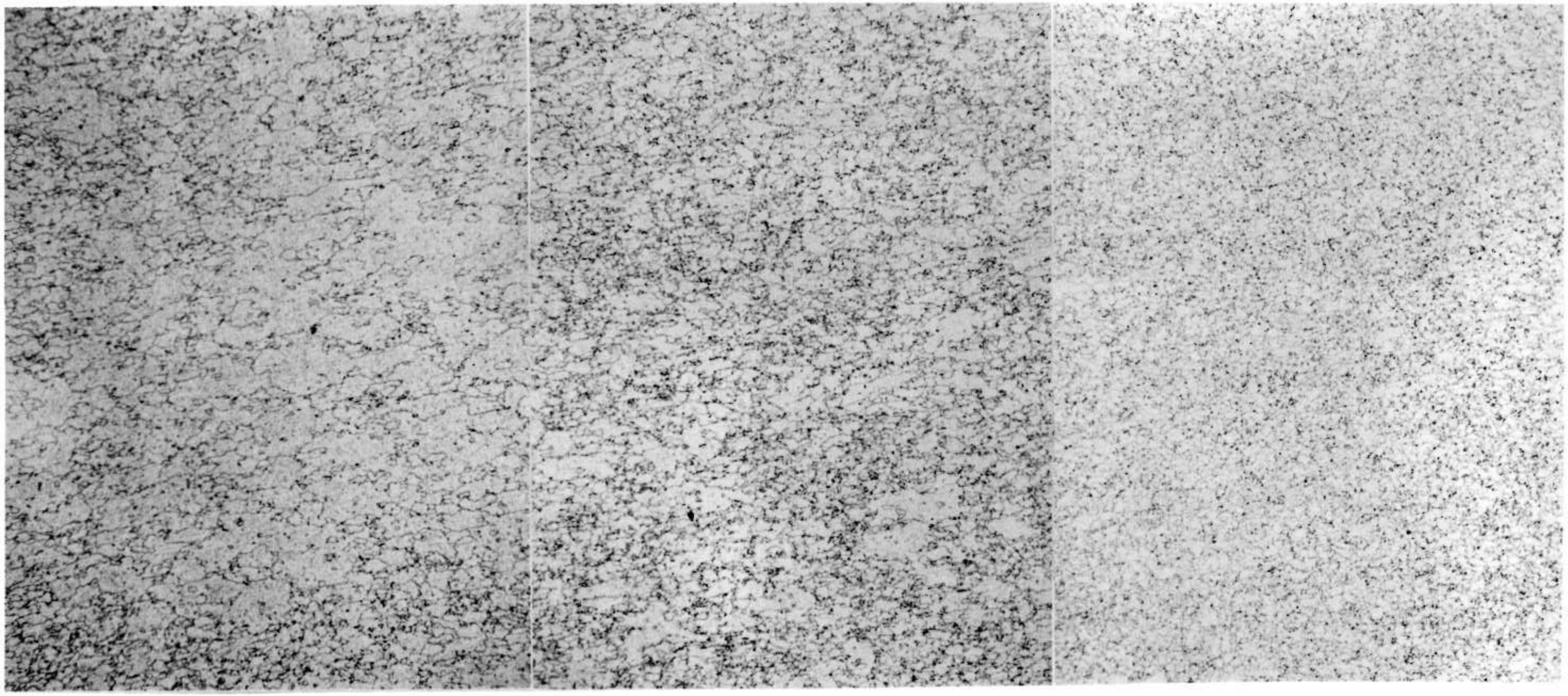
The structures both as-extruded and after rolling were more uniform than any of the previous extrusions. The microstructures are given in Figures 31 and 32 for the extruded and the rolled sections.

The extruded recrystallized sections, Figure 31, appeared more uniform, but were of a much smaller grain size than shown for 3925.

The extruded and rolled recrystallized structures appeared to be large grain size and fairly uniform, with a minimum of very small grains.

Elevated temperature stress rupture tests were made at NASA-Lewis on sections cut from the middle of the cross-sections. These data are given in Table 54. The excellent stress rupture properties indicate the potential of achieving properties comparable to TDNiCr in the TDNiCrAl material.

Five additional billets were prepared for use in other NASA studies, two of the 3.5% Al alloy and three of the 5.0% Al alloy. These were prepared in the same manner as the previous billets and the manufacturing data and sizes are given in Table 55.

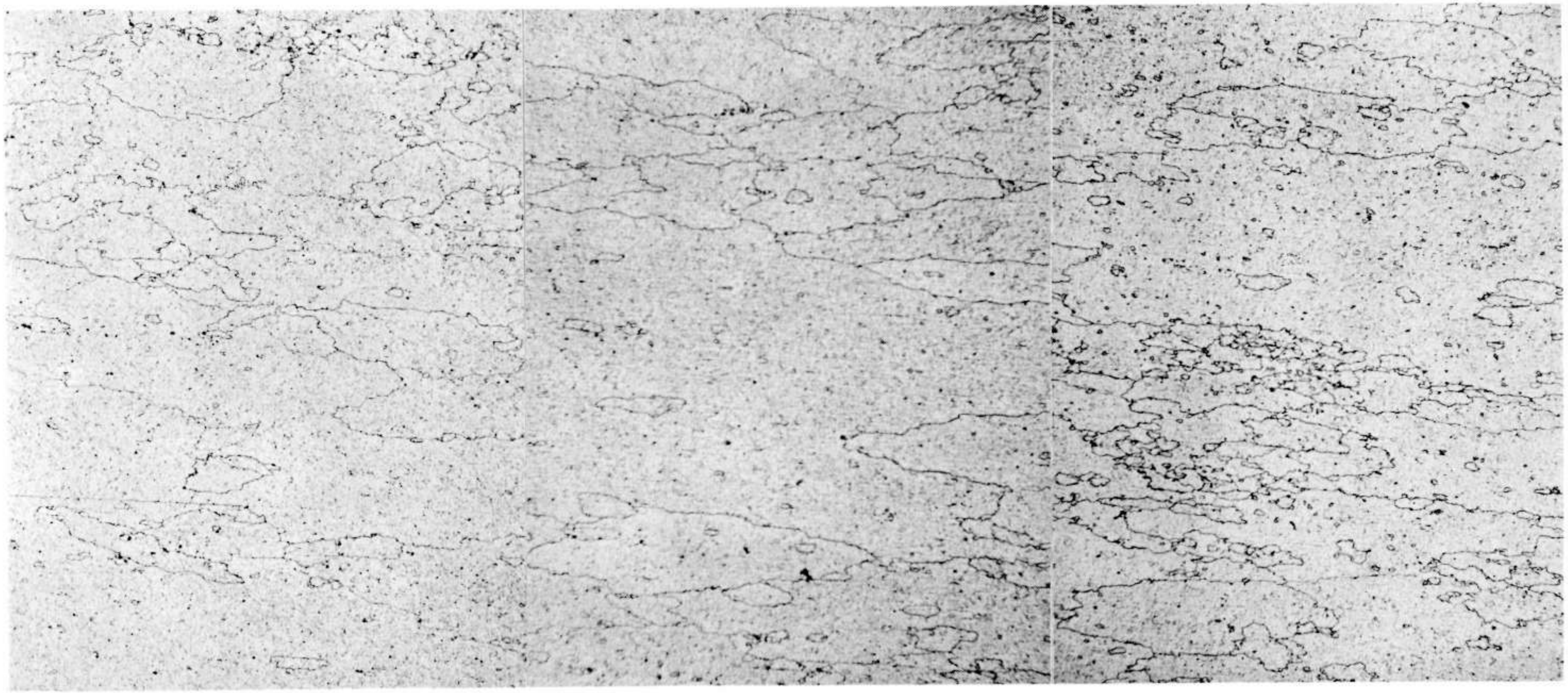


Edge Mid Center

1-1/4" x 4-5/8" Extruded Bar-Heat Treated 1345° C (2450° F)/1-1/2 Hours - 250X

FIGURE 31

MICROSTRUCTURES IN RECRYSTALLIZED EXTRUDED BAR - HEAT 3936



Edge Mid Center

1-1/4" x 4-5/8" Extruded Bar-Rolled 15%-Heat Treated 1345° C(2450° F)/1-1/2 Hours - 250X

FIGURE 32

MICROSTRUCUTRES IN RECRYSTALLIZED, EXTRUDED AND ROLLED BAR - HEAT 3936

TABLE 54

1093° C(2000° F) STRESS RUPTURE DATA - HEAT 3936

Sample	Stress		Rupture Life
	MN/m²	ksi	Hours
Extruded and ReX* Longitudinal Center	48	7.0	0.3
Extruded and ReX Transverse Center	38	5.5	1.6
Extruded, Rolled and ReX Longitudinal Center	48	7.0	187
Extruded, Rolled and ReX Transverse Center	38	5.5	170

*ReX - All samples recrystallized at 1345° C(2450° F) 1-1/2 hours.

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TABLE 55

TDNiCrAl CONSOLIDATED BILLETS

Heat No.	Alloy	Sinter Temperature		Upset Temperature		Weight		Size		Billet Quality
		°C	°F	°C	°F	kg.	lbs.	cm.	in.	
3931	3.5% Al	955	1750	1010	1800	83.0	183.0	19Øx36.5	7½Øx14-3/8	Flaw on butt end of billet extends 3.8 cm. (1.5 in.) deep
3932	3.5% Al	955	1750	1010	1800	79.6	175.5	19Øx34.3	7½Øx13-1/2	Circumferential flaw 16.5 cm. (6.5 in.) from butt of billet
3933	5.0% Al	955	1750	1010	1800	73.5	162.0	19Øx31.8	7½Øx12-1/2	No flaws
3935	5.0% Al	955	1750	1010	1800	78.9	174.0	19Øx34.3	7½Øx13-1/2	No flaws
3939	5.0% Al	955	1750	1010	1800	81.5	179.5	19Øx35.6	7½Øx14	Circumferential crack 14.0 cm. (5.5 in.) from butt of billet

A problem was encountered in the induction heating for the upset consolidation of these billets. This resulted in billet flaws which could be detected during machining for the final canning operation. The location of these flaws is noted in Table 55. All of the billets were canned for subsequent extrusion by NASA.

2. Extruded Product Shipments

A total of 166.4 kg.(366.4 lbs.) of TDNiCr fastener bar stock and 144.25 kg.(318.0 lbs.) of TDNiCrAl fastener bar stock was shipped to NASA or to NASA Contractors. Individual piece inventories are included in Appendix G of this report.

A total of 347.9 kg.(767.0 lbs.) of extruded rectangular section of TDNiCrAl stock was shipped to NASA-Lewis. Two densified billets of Ni-16Cr-3.5Al-$2ThO_2$ and three densified billets of Ni-16Cr-5.0Al-$2ThO_2$ were shipped to NASA-Lewis, a total of 396.5 kg.(874.0 lbs.). Individual piece inventories are included in Appendix G of this report.

Additional processing studies are required to develop the extrusion process for rectangular TDNiCrAl bar. The preliminary studies described herein indicate that more uniform deformation is required to achieve a uniform, recrystallized structure. Future studies should evaluate higher reductions and other process variables such as die geometry and extrusion shape that tend to improve uniformity in the extruded product. Also, reductions in ThO_2 content might be of assistance in achieving the desired structure.

SUMMARY OF RESULTS

The goals of the Part II program have been attained. The process for the manufacture of TDNiCr sheet has been scaled up to provide larger sheet sizes. An alternate process has been developed to provide thin TDNiCr sheet having improved quality and tolerances. High quality TDNiCr foil was produced by cold tension rolling. Sheet process development and extrusion development studies were conducted on two promising alloys. Quantities of TDNiCr and TDNiCrAl products were supplied to NASA.

The standard process for the manufacture of TDNiCr sheet, 61.0 x 152.4 x 0.025-0.103 cm.(24 x 60 x 0.010-0.040 in.) included the following steps:

- Powder Manufacture
- Hydrostatic Compaction
- Sinter Consolidate
- Decan, condition and sample
- Recan
- Roll to Intermediate Gauge Plate
- Decan
- Chemical Clean
- Preparation of Intermediate Gauge Plate for Canning
- Can
- Roll to Gauge
- Chemical Clean
- Recrystallization Heat Treat
- Finishing

The billet size was increased from 45.4 kg.(100 lb.) to 68.0 kg.(150 lb.) and a number of process improvements were developed.

In the alternate process for the manufacture of TDNiCr thin sheet, 61.0 x 152.4 x 0.025 to <0.038 cm.(24 x 60 x 0.010 to <0.015 in.) cold rolling was used instead of warm rolling for finish rolling to gauge.

The cold finish rolling resulted in improvements in both surface finish and tolerances. A reduction in high temperature strength was experienced when the cold rolling procedure was utilized. High quality TDNiCr foil was produced by cold tension rolling in gauges of 0.0051 to 0.0127 cm.(0.002 to 0.005 in.).

Two TDNiCrAl alloys were selected for further development, Ni-16%Cr-3.5%Al-2%ThO_2 and Ni-16%Cr-5.0%Al-2%ThO_2. The 3.5% Al level appears to afford the optimum combination of oxidation resistance, ductility and strength for space shuttle applications. The 5.0% Al level is believed to afford additional oxidation resistance for long term use in aircraft engine applications.

Limited sheet process studies demonstrated that TDNiCrAl sheet can readily be manufactured, but higher rolling temperatures are required than for TDNiCr. The high temperature properties attained on sheet containing 5.0% Al were lower than for TDNiCr. For the 3.5% Al alloy, properties comparable to TDNiCr were achieved in the experimental material.

Extrusion process studies demonstrated that bar and shapes can be produced by extrusion in a wide range of sizes. Extruded and recrystallized TDNiCrAl bar and shapes have good high temperature strength potential. Attainment of uniform microstructures desired for good high temperature strength will require additional development studies.

A total of 448.8 kg.(989.4 lb.) of TDNiCr plate, sheet and foil and 166.4 kg.(366.4 lb.) of extruded TDNiCr bar was manufactured and shipped to the NASA Centers or their contractors for space shuttle technology studies.

A total of 136.4 kg.(300.6 lb.) of TDNiCrAl sheet, 144.2 kg.(318 lb.) of TDNiCrAl extruded bar, and 347.9 kg. (767.0 lb.) of TDNiCrAl extruded shapes were manufactured and shipped to NASA for technology studies.

Five densified billets of TDNiCrAl alloys, 396.5 kg. (874 lb.) was manufactured and shipped to NASA for future extrusion development studies.

APPENDIX A

Alternate TDNiCr Sheet Process

Mechanical Properties-Laboratory Investigations

TABLE A-1

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3473-1)

Heat Treatment: 2 hours at 1177° C, Dry H_2

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			1093°C			Minimum Bend Radius	1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong		37.9 MN/m^2	31.0 MN/m^2
0	0.102	897.1	604.3	19.9	113.7	113.7	2.8	2.5T	10.5	
40	0.061	1018.3	764.8	17.5	97.8	97.8	2.0	3.0T		>20.0
45	0.056	992.8	682.1	20.0	93.7	93.7	3.0	2.0T		>20.0
50	0.051	984.6	681.4	19.5	86.8	86.8	2.0	0.8T		1.7
55	0.046	997.7	792.8	17.5	83.4	83.4	3.0	0.8T		2.0
60	0.041	972.9	697.3	20.0	74.4	74.4	2.5	0.8T		17.9
65	0.036	1014.2	894.3	11.9	81.3	81.3	4.0	0.8T		>20.0
70	0.031	1056.9	977.0	10.0	79.2	79.2	6.0	0.8T		>20.0
75	0.025	1016.3	870.9	12.5	76.0	76.0	7.0	0.8T		>20.0

TABLE A-1

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3473-1)

Heat Treatment: 2 hours at 2150° F, Dry H_2

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	130.2	87.7	19.9	16.5	16.5	2.8	2.5T	10.5	
40	0.024	147.8	111.0	17.5	14.9	14.2	2.0	3.0T		>20.0
45	0.022	144.1	99.0	20.0	14.7	13.6	3.0	2.0T		>20.0
50	0.020	142.9	98.9	19.5	12.8	12.6	2.0	0.8T		1.7
55	0.018	144.8	115.6	17.5	13.6	12.1	3.0	0.8T		2.0
60	0.016	141.2	101.2	20.0	12.0	10.8	2.5	0.8T		17.9
65	0.014	147.2	129.8	11.9	12.6	11.8	4.0	0.8T		>20.0
70	0.012	153.4	141.8	10.0	11.5	11.5	6.0	0.8T		>20.0
75	0.010	147.5	126.4	12.5	11.5	11.3	7.0	0.8T		>20.0

TABLE A-2

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3473-1)

Heat Treatment: Plant

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	897.1	604.3	19.9	113.7	113.7	2.5	2.0T	10.5	
5	0.097	896.4	649.0	16.0	130.9	130.9	2.5	3.0T	18.6	
10	0.092		Sample not obtained							
15	0.086	920.5	786.8	12.5	124.7	124.7	2.5	4.0T	13.7	
20	0.081	956.3	818.5	13.0	122.0	119.9	2.0	5.0T	23.3	
25	0.076	958.4	804.8	12.0	122.0	117.1	2.0	5.0T	11.3	
30	0.071		Sample not obtained							
35	0.066		Sample not obtained							
40	0.061	946.0	746.2	12.5	111.6	101.3	2.0	3.0T		>20
45	0.056	992.2	779.9	13.0	89.6	89.1	3.0	3.0T		>20
50	0.051	923.9	724.1	18.5	90.9	87.5	3.0	2.0T		>20
55	0.046	957.0	779.9	22.0	91.6	83.4	3.0	2.0T		>20
60	0.041	966.0	660.1	20.5	88.9	86.1	2.5	2.0T		1.7
65	0.036	973.6	826.8	11.5	78.5	76.5	4.5	2.0T		>20
70	0.031	1016.9	896.4	10.0	88.9	82.7	5.0	2.0T		>20
75	0.025	979.8	804.8	12.0	90.3	90.3	8.0	2.0T		>20

TABLE A-2

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3473-1)

Heat Treatment: Plant

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			2000°F			Minimum	2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Bend Radius	5.5 ksi	4.5 ksi
0	0.040	130.2	87.7	19.9	16.5	16.5	2.5	2.0T	10.5	
5	0.038	130.1	94.2	16.0	19.0	19.0	2.5	3.0T	18.6	
10	0.036		Sample not obtained							
15	0.034	133.6	114.2	12.5	18.1	18.1	2.5	4.0T	13.7	
20	0.032	138.8	118.8	13.0	17.7	17.4	2.0	5.0T	23.3	
25	0.030	139.1	116.8	12.0	17.7	17.0	2.0	5.0T	11.3	
30	0.028		Sample not obtained							
35	0.026		Sample not obtained							
40	0.024	137.3	108.3	12.5	16.2	14.7	2.0	3.0T		>20
45	0.022	144.0	113.2	13.0	13.0	12.9	3.0	3.0T		>20
50	0.020	134.1	105.1	18.5	13.2	12.7	3.0	2.0T		>20
55	0.018	138.9	113.2	22.0	13.3	12.1	3.0	2.0T		>20
60	0.016	140.2	95.8	20.5	12.9	12.5	2.5	2.0T		1.7
65	0.014	141.3	120.0	11.5	11.4	11.1	4.5	2.0T		>20
70	0.012	147.6	130.1	10.0	12.9	12.0	5.0	2.0T		>20
75	0.010	142.2	116.8	12.0	13.1	13.1	8.0	2.0T		>20

TABLE A-3

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3631-1)

Heat Treatment: 2 hours at 1177° C, Dry H_2

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	848.2	562.9	20.0	117.2	117.2	2.0	3.0T	>20.0	
40	0.061	965.3	715.9	17.5	114.4	112.3	2.5	6.0T		>20.0
45	0.056	949.4	673.8	18.0	101.3	96.5	2.0	3.5T		>20.0
50	0.051	942.6	675.9	17.5	99.2	94.4	2.5	2.5T		>20.0
55	0.046	923.9	672.5	19.5	88.9	81.3	4.8	2.5T		>20.0
60	0.041	941.9	673.2	17.0	76.5	68.9	3.5	2.5T		1.2
65	0.036	1015.6	869.5	11.9	75.1	73.0	3.0	2.5T		>20.0
70	0.031	1080.4	950.8	10.0	87.5	84.7	4.5	2.5T		>20.0
75	0.025	1034.9	866.8	12.5	86.1	77.9	5.0	2.5T		0.4

TABLE A-3

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3631-1)

Heat Treatment: 2 hours at 2150°F, Dry H_2

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	123.1	81.7	20.0	17.1	17.1	2.0	3.0T	>20.0	
40	0.024	140.1	103.9	17.5	16.6	16.3	2.5	6.0T		>20.0
45	0.022	137.8	97.7	18.0	14.7	14.0	2.0	3.5T		>20.0
50	0.020	136.8	98.1	17.5	14.4	13.7	2.5	2.5T		>20.0
55	0.018	134.1	97.6	19.5	12.9	11.8	4.8	2.5T		>20.0
60	0.016	136.7	97.7	17.0	11.1	10.0	3.5	2.5T		1.2
65	0.014	147.4	126.2	11.9	10.9	10.6	3.0	2.5T		>20.0
70	0.012	156.8	138.0	10.0	12.7	12.3	4.5	2.5T		>20.0
75	0.010	150.2	125.8	12.5	12.5	11.3	5.0	2.5T		0.4

TABLE A-4

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3631-1)

Heat Treatment: Plant

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	848.2	562.9	20.0	117.8	117.8	2.0	3.0T	>20	
5	0.097	870.2	673.2	20.0	135.0	135.0	2.5	4.0T	>20	
10	0.092	Sample not obtained								
15	0.086	909.5	765.5	12.5	136.4	130.9	2.5	4.0T	>20	
20	0.081	923.3	768.9	15.0	124.7	123.3	2.5	5.0T	>20	
25	0.076	929.5	824.7	10.0	135.7	135.0	3.0	5.0T	>20	
30	0.071	Sample not obtained								
35	0.066	930.8	768.2	10.0	126.1	119.2	3.5	5.0T	>20	
40	0.061				119.2	116.4	2.0	6.0T		>20
45	0.056	951.5	674.5	15.0	107.5	101.3	2.5	5.0T		>20
50	0.051	933.6	664.2	18.0	99.9	97.1	3.0	3.0T		>20
55	0.046				97.1	90.9	4.0	2.5T		>20
60	0.041	938.4	687.6	18.5	102.0	95.1	3.5	2.5T		>20
65	0.036	980.4	824.0	10.0	98.5	96.5	3.0	2.5T		>20
70	0.031	1043.8	921.9	10.0	89.6	89.6	3.5	2.5T		>20
75	0.025	1001.1	852.3	11.5	88.2	87.3	2.5	2.5T		>20

TABLE A-4

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3631-1)

Heat Treatment: Plant

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	123.1	81.7	20.0	17.1	17.1	2.0	3.0T	>20	
5	0.038	126.3	97.7	20.0	19.6	19.6	2.5	4.0T	>20	
10	0.036	Sample not obtained								
15	0.034	132.0	111.1	12.5	19.8	19.0	2.5	4.0T	>20	
20	0.032	134.0	111.6	15.0	18.1	17.9	2.5	5.0T	>20	
25	0.030	134.9	119.7	10.0	19.7	19.6	3.0	5.0T	>20	
30	0.028	Sample not obtained								
35	0.026	135.1	111.5	10.0	18.3	17.3	3.5	5.0T	>20	
40	0.024				17.3	16.9	2.0	6.0T		>20
45	0.022	138.1	97.9	15.0	15.6	14.7	2.5	5.0T		>20
50	0.020	135.5	96.4	18.0	14.5	14.1	3.0	3.0T		>20
55	0.018				14.1	13.2	4.0	2.5T		>20
60	0.016	136.2	99.8	18.5	14.8	13.8	3.5	2.5T		>20
65	0.014	142.3	119.6	10.0	14.3	14.0	3.0	2.5T		>20
70	0.012	151.5	133.8	10.0	13.0	13.0	3.5	2.5T		>20
75	0.010	145.3	123.7	11.5	12.7	12.7	2.5	2.5T		>20

TABLE A-5

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3418-1)

Heat Treatment: 2 hours at 1177° C, Dry H_2

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm. (0.35 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	809.6	598.7	17.0	108.2	108.2	2.0	4.0T	>20	
40	0.061	900.5	770.3	14.5	175.7	175.7	2.0	10.0T		>20
45	0.056	930.2	807.5	14.5	177.8	177.8	2.0	11.0T		>20
50	0.051	955.6	901.9	13.0	166.7	166.7	2.0	12.0T		>20
55	0.046	963.2	911.5	12.0	141.2	128.8	2.5	13.0T		>20
60	0.041	992.8	934.3	12.5	137.8	126.1	2.5	14.0T		>20
65	0.036	979.8	849.5	10.5	135.7	132.3	2.0	9.0T		>20
70	0.031	939.8	650.4	13.5	125.4	125.4	1.5	3.0T		>20
75	0.025	902.6	650.4	12.5	117.1	117.1	2.0	1.5T		>20

TABLE A-5

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3418-1)

Heat Treatment: 2 hours at 2150° F, Dry H_2

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	117.5	86.9	17.0	15.7	15.7	2.0	4.0T	>20	
40	0.024	130.7	111.8	14.5	25.2	25.2	2.0	10.0T		>20
45	0.022	135.0	117.2	14.5	25.8	25.8	2.0	11.0T		>20
50	0.020	138.7	130.9	13.0	24.2	24.2	2.0	12.0T		>20
55	0.018	139.8	132.3	12.0	20.5	18.7	2.5	13.0T		>20
60	0.016	144.1	135.6	12.5	20.0	18.3	2.5	14.0T		>20
65	0.014	142.2	123.3	10.5	19.7	19.2	2.0	9.0T		>20
70	0.012	136.4	94.4	13.5	18.2	18.2	1.5	3.0T		>20
75	0.010	131.0	94.4	12.5	17.0	17.0	2.0	1.5T		>20

TABLE A-6

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3418-1)

Heat Treatment: Plant

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			1093°C			Minimum Bend Radius	1093°C Stress Rupture Hrs. at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.		37.9 MN/m^2	31.0 MN/m^2
0	0.102	809.6	598.7	17.0	103.9	103.9	2.0	4.0T	>20	
40	0.061	917.7	850.9	16.6	199.8	199.8	2.5	8.0T		>20
45	0.056	944.6	882.6	15.9	181.2	172.9	2.6	12.0T		>20
50	0.051	956.3	909.5	14.0	181.2	181.2	2.5	11.0T		>20
55	0.046	948.1	911.5	15.0	158.5	141.2	2.7	12.0T		>20
60	0.041	990.8	941.9	17.0	139.9	137.1	2.5	12.0T		>20
65	0.036	967.4	868.1	12.0	115.8	109.6	2.0	12.0T		>20
70	0.031	875.4	619.4	12.5	108.2	108.2	2.0	6.0T		>20
75	0.025	878.5	620.1	10.5	105.4	104.0	2.0	3.0T		>20

TABLE A-6

MECHANICAL PROPERTIES VS. PERCENT REDUCTION (HEAT 3418-1)

Heat Treatment: Plant

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hrs. at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	117.5	86.9	17.0	15.7	15.7	2.0	4.0T	>20	
40	0.024	133.2	123.5	16.6	29.0	29.0	2.5	8.0T		>20
45	0.022	137.1	128.1	15.9	26.3	25.1	2.6	12.0T		>20
50	0.020	138.8	132.0	14.0	26.3	26.3	2.5	11.0T		>20
55	0.018	137.6	132.3	15.0	23.0	20.5	2.7	12.0T		>20
60	0.016	143.8	136.7	17.0	20.3	19.9	2.5	12.0T		>20
65	0.014	140.4	126.0	12.0	16.8	15.9	2.0	12.0T		>20
70	0.012	127.6	89.9	12.5	15.7	15.7	2.0	6.0T		>20
75	0.010	127.5	90.0	10.5	15.3	15.1	2.0	3.0T		>20

TABLE A-7

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3473-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: 2 hours at 1177°C, dry H_2

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	897.1	604.3	19.9	113.7	113.7	2.8	2.5T	10.5	
40	0.061	1023.9	935.7	12.0	127.5	125.4	1.5	7.0T		>20.0
45	0.056	1032.8	874.3	12.5	125.4	123.3	3.0	8.0T		>20.0
50	0.051	1008.7	771.0	15.0	135.0	132.3	2.0	3.0T		>20.0
55	0.046	959.8	748.9	15.5	117.8	115.1	2.5	2.5T		4.1
60	0.041	992.8	740.0	17.5	94.4	88.9	4.5	2.0T		>20.0
65	0.036	973.6	773.7	19.5	84.1	73.0	5.0	1.1T		>20.0
70	0.031	981.1	771.7	19.5	84.1	73.0	4.5	1.2T		>20.0

TABLE A-7

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3473-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: 2 hours at 2150°F, dry H_2

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	130.2	87.7	19.9	16.5	16.5	2.8	2.5T	10.5	
40	0.024	148.6	135.8	12.0	18.5	18.2	1.5	7.0T		>20.0
45	0.022	149.9	126.9	12.5	18.2	17.9	3.0	8.0T		>20.0
50	0.020	146.4	111.9	15.0	19.6	19.2	2.0	3.0T		>20.0
55	0.018	139.3	108.7	15.5	17.1	16.7	2.5	2.5T		4.1
60	0.016	144.1	107.4	17.5	13.7	12.9	4.5	2.0T		>20.0
65	0.014	141.3	112.3	19.5	12.2	10.6	5.0	1.1T		>20.0
70	0.012	142.4	113.0	19.5	12.2	10.6	4.5	1.2T		>20.0

TABLE A-8

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3473-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: Plant

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	897.1	604.3	19.9	113.7	113.7	2.8	2.5T	10.5	
40	0.061	990.8	862.6	13.0	117.1	111.3	1.5	7.0T		>20.0
45	0.056	994.9	782.7	14.5	102.0	98.5	3.5	4.5T		>20.0
50	0.051	963.2	715.9	17.5	123.3	117.1	2.5	2.5T		>20.0
55	0.046	967.4	714.5	17.5	111.3	104.0	2.5	2.0T		>20.0
60	0.041	938.4	647.0	20.0	93.7	84.1	3.0	2.0T		17.9
65	0.036	941.9	704.2	19.0	84.1	76.5	5.0	1.5T		16.3
70	0.031	949.4	718.6	20.5	68.2	62.7	7.0	2.0T		>20.0

TABLE A-8

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3473-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: Plant

Initial Micro Grain Size: 0.035 mm
Initial Macro Grain Size: 0.05 cm.(0.02 in.)

		Room Temperature			2000°F			Minimum	2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Bend Radius	5.5 ksi	4.5 ksi
0	0.040	130.2	87.7	19.9	16.5	16.5	2.8	2.5T	10.5	
40	0.024	143.8	125.2	13.0	17.0	16.3	1.5	7.0T		>20.0
45	0.022	144.4	113.6	14.5	14.8	14.3	3.5	4.5T		>20.0
50	0.020	139.8	103.9	17.5	17.9	17.0	2.5	2.5T		>20.0
55	0.018	140.4	103.7	17.5	16.3	15.1	2.5	2.0T		>20.0
60	0.016	136.2	93.9	20.0	13.6	12.2	3.0	2.0T		17.9
65	0.014	136.7	102.2	19.0	12.2	11.1	5.0	1.5T		16.3
70	0.012	137.8	104.3	20.5	9.9	9.1	7.0	2.0T		>20.0

TABLE A-9

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3631-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: 2 hours at 1177°C, dry H_2

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm. (0.03 in.)

		Room Temperature			1093°C			Minimum	1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m²	YS MN/m²	% Elong	UTS MN/m²	YS MN/m²	% Elong.	Bend Radius	37.9 MN/m²	31.0 MN/m²
0	0.102	848.2	562.9	20.0	117.8	117.8	2.0	3.5T	>20.0	
40	0.061	965.3	886.7	12.5	110.2	110.2	2.5	7.5T	>20.0	
45	0.056	964.6	766.2	14.5	116.4	110.2	2.5	6.5T	11.1	
50	0.051	962.5	708.3	15.5	102.0	95.8	2.5	3.0T	1.0	>20.0
55	0.046	955.6	690.4	15.5	106.8	105.4	2.5	1.8T		>20.0
60	0.041	948.8	712.4	18.0	86.1	84.1	4.0	2.0T		0.4
65	0.036	931.5	702.8	17.5	68.9	64.1	4.5	1.0T		0.7
70	0.031	955.0	702.1	17.0	67.5	67.5	7.0	1.2T		>20.0

TABLE A-9

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3631-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: 2 hours at 2150°F, dry H_2

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			2000°F			Minimum Bend Radius	2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.		5.5 ksi	4.5 ksi
0	0.040	123.1	81.7	20.0	17.1	17.1	2.0	3.5T	>20.0	
40	0.024	140.1	128.7	12.5	16.0	16.0	2.5	7.5T	>20.0	
45	0.022	140.0	111.2	14.5	16.9	16.0	2.5	6.5T	11.1	
50	0.020	139.7	102.8	15.5	14.8	13.9	2.5	3.0T	1.0	>20.0
55	0.018	138.7	100.2	15.5	15.5	15.3	2.5	1.8T		>20.0
60	0.016	137.7	103.4	18.0	12.5	12.2	4.0	2.0T		0.4
65	0.014	135.2	102.0	17.5	10.0	9.3	4.5	1.0T		0.7
70	0.012	138.6	101.9	17.0	9.8	9.8	7.0	1.2T		>20.0

TABLE A-10

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3631-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: Plant

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	848.2	562.9	20.0	117.8	117.8	2.0	3.5	>20.0	
40	0.061	957.2	877.1	13.5	125.4	124.0	2.0	7.0T		>20.0
45	0.056	965.3	787.5	13.0	111.6	106.8	2.5	7.0T		>20.0
50	0.051	954.3	716.6	17.0	101.3	94.4	2.5	3.7T		>20.0
55	0.046	948.1	686.2	18.0	111.6	106.8	2.5	1.8T		>20.0
60	0.041	948.1	700.0	17.0	86.8	77.2	2.5	1.0T		>20.0
65	0.036	941.2	706.9	19.0	71.0	67.5	6.0	1.0T		>20.0
70	0.030	960.5	755.1	20.0	73.7	63.4	10.0	1.2T		>20.0

TABLE A-10

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3631-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: Plant

Initial Micro Grain Size: 0.050 mm
Initial Macro Grain Size: 0.08 cm.(0.03 in.)

		Room Temperature			2000°F			Minimum Bend Radius	2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.		5.5 ksi	4.5 ksi
0	0.040	123.1	81.7	20.0	17.1	17.1	2.0	3.5	>20.0	
40	0.024	138.2	127.3	13.5	18.2	18.0	2.0	7.0T		>20.0
45	0.022	140.1	114.3	13.0	16.2	15.5	2.5	7.0T		>20.0
50	0.020	138.5	104.0	17.0	14.7	13.7	2.5	3.7T		>20.0
55	0.018	137.6	99.6	18.0	16.2	15.5	2.5	1.8T		>20.0
60	0.016	137.6	101.6	17.0	12.6	11.2	2.5	1.0T		>20.0
65	0.014	136.6	102.6	19.0	10.3	9.8	6.0	1.0T		>20.0
70	0.012	139.4	109.6	20.0	10.7	9.2	10.0	1.2T		>20.0

TABLE A-11

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3418-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: 2 hours at 1177°C, dry H_2

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	809.6	598.7	17.0	108.2	108.2	2.0	4.0T	>20.0	
40	0.061	838.5	787.9	12.0	179.1	179.1	1.5	9.0T	>20.0	
45	0.056	877.8	796.5	12.5	185.3	185.3	2.0	8.0T		>20.0
50	0.051	921.2	865.4	12.5	190.9	188.1	2.5	9.0T		>20.0
55	0.046	941.9	877.1	14.5	159.2	155.0	2.0	9.0T		>20.0
60	0.041	966.0	910.9	14.5	160.5	160.5	1.0	11.0T		>20.0
65	0.036	988.7	886.1	16.0	117.8	115.8	1.5	11.0T		>20.0
70	0.030	990.9	826.1	10.5	122.0	119.2	2.0	6.0T		>20.0

TABLE A-11

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3418-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: 2 hours at 2150°F, dry H_2

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	117.5	86.9	17.0	15.7	15.7	2.0	4.0T	>20.0	
40	0.024	121.7	110.0	12.0	26.0	26.0	1.5	9.0T	>20.0	
45	0.022	127.4	115.6	12.5	26.9	26.9	2.0	8.0T		>20.0
50	0.020	133.7	125.6	12.5	27.7	27.3	2.5	9.0T		>20.0
55	0.018	136.7	127.3	14.5	23.1	22.5	2.0	9.0T		>20.0
60	0.016	140.2	132.2	14.5	23.3	23.3	1.0	11.0T		>20.0
65	0.014	143.5	128.6	16.0	17.1	16.8	1.5	11.0T		>20.0
70	0.012	143.8	119.9	10.5	17.7	17.3	2.0	6.0T		>20.0

TABLE A-12

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3418-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: Plant

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			1093°C				1093°C Stress Rupture Hours at	
% Red.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	37.9 MN/m^2	31.0 MN/m^2
0	0.102	809.6	598.7	17.0	108.2	108.2	2.0	4.0T	>20.0	
40	0.061	843.3	776.5	14.5	180.5	180.5	1.5	10.0T		>20.0
45	0.056	884.0	789.6	15.0	185.3	184.6	1.5	10.0T		>20.0
50	0.051	892.9	837.1	12.0	184.6	175.7	2.5	10.0T		>20.0
55	0.046	938.4	885.4	13.0	180.5	180.5	2.0	12.0T		>20.0
60	0.041	966.0	903.3	9.5	141.9	141.9	1.5	12.0T		>20.0
65	0.036	999.1	888.8	12.0	113.0	108.2	2.5	10.0T		>20.0
70	0.031	950.8	733.8	11.0	101.3	97.8	2.0	3.0-6.0T		>20.0

TABLE A-12

MECHANICAL PROPERTIES VS. PERCENT REDUCTION FOR HEAT 3418-2 WITH INTERMEDIATE HEAT TREATMENT AT 30% REDUCTION

Heat Treatment: Plant

Initial Micro Grain Size: 0.4-0.6 mm
Initial Macro Grain Size: 0.9 cm.(0.35 in.)

		Room Temperature			2000°F				2000°F Stress Rupture Hours at	
% Red.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	5.5 ksi	4.5 ksi
0	0.040	117.5	86.9	17.0	15.7	15.7	2.0	4.0T	>20.0	
40	0.024	122.4	112.7	14.5	26.2	26.2	1.5	10.0T		>20.0
45	0.022	128.3	114.6	15.0	26.9	26.8	1.5	10.0T		>20.0
50	0.020	129.6	121.5	12.0	26.8	25.5	2.5	10.0T		>20.0
55	0.018	136.2	128.5	13.0	26.2	26.2	2.0	12.0T		>20.0
60	0.016	140.2	131.1	9.5	20.6	20.6	1.5	12.0T		>20.0
65	0.014	145.0	129.0	12.0	16.4	15.7	2.5	10.0T		>20.0
70	0.012	138.0	106.5	11.0	14.7	14.2	2.0	3.0-6.0T		>20.0

APPENDIX B

Alternate TDNiCr Sheet Process

Mechanical Properties-Production Trials

TABLE B-1

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3830

Starting Gauge: 0.058 cm.(0.023 in.)
Initial Micro Grain Size: 0.080 mm
Initial Macro Grain Size: 0.15 cm.(0.06 in.)
Heat Treated at 0.051 cm.(0.020 in.)

	Room Temperature			1093°C				1093°C
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
0.025	826.1	608.4	11.9	86.1	86.1	1.5	1.5T	>20

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	Room Temperature			2000°F				2000°F
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
0.010	119.9	88.3	11.9	12.5	12.5	1.5	1.5T	>20

TABLE B-2

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3469

Starting Gauge: 0.064 cm. (0.025 in.)
Initial Micro Grain Size: 0.130 mm
Initial Macro Grain Size: 0.25 cm. (0.10 in.)
Heat Treated at 0.051 cm. (0.020 in.)

	Room Temperature			1093°C			Minimum	1093°C
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
0.025	890.9	593.9	14.0	124.7	124.7	-	<3.0T	>20

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	Room Temperature			2000°F			Minimum	2000°F
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Bend Radius	Stress Rupture Hours at 4.5 ksi
0.010	129.3	86.2	14.0	18.1	18.1	-	<3.0T	>20

TABLE B-3

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3872-S

Starting Gauge: 0.064 cm.(0.025 in.)
Initial Micro Grain Size: 0.100 mm
Initial Macro Grain Size: 0.18 cm.(0.07 in.)
Heat Treated at 0.051 cm.(0.020 in.)

	Room Temperature			1093°C				1093°C
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
0.025	921.2	655.2	14.0	135.7	130.9	2.0	<3.0T	>20

	Room Temperature			2000°F				2000°F
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
0.010	133.7	95.1	14.0	19.7	19.0	2.0	<3.0T	>20

TABLE B-4

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3796

Starting Gauge: 0.076 cm.(0.030 in.)
Initial Micro Grain Size: 0.080 mm
Initial Macro Grain Size: 0.15 cm.(0.06 in.)
Heat Treated at 0.051 cm.(0.020 in.)

Gauge cm.	Room Temperature			1093°C			Minimum Bend Radius	1093°C Stress Rupture Hours at 31.0 MN/m^2
	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.		
0.025	885.4	649.0	18.5	77.2	75.1	3.0	0.6T	.9

Gauge in.	Room Temperature			2000°F			Minimum Bend Radius	2000°F Stress Rupture Hours at 4.5 ksi
	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.		
0.010	128.5	94.2	18.5	11.2	10.9	3.0	0.6T	.9

TABLE B-5

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3795

Starting Gauge: 0.076 cm.(0.030 in.)
Initial Micro Grain Size: 0.130 mm
Initial Macro Grain Size: 0.25 cm.(0.10 in.)
Heat Treated at 0.051 cm.(0.020 in.)

	Room Temperature			1093°C				1093°C
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
0.025	877.8	626.3	18.5	83.4	83.4	3.0	0.6T	>20

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	Room Temperature			2000°F				2000°F
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
0.010	127.4	90.9	18.5	12.1	12.1	3.0	0.6T	>20

TABLE B-6

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3794

Starting Gauge: 0.076 cm.(0.030 in.)
Initial Micro Grain Size: 0.110 mm
Initial Macro Grain Size: 0.15 cm.(0.08 in.)
Heat Treated at 0.051 cm.(0.020 in.)

	Room Temperature			1093°C				1093°C
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
0.025	910.2	665.1	19.5	79.2	77.2	3.0	0.6T	>20

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	Room Temperature			2000°F				2000°F
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
0.010	132.1	96.6	19.5	11.5	11.2	3.0	0.6T	>20

TABLE B-7

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3792

Starting Gauge: 0.076 cm.(0.030 in.)
Initial Micro Grain Size: 0.063 mm
Initial Macro Grain Size: 0.10 cm.(0.04 in.)
Heat Treated at 0.051 cm.(0.020 in.)

		Room Temperature			1093°C				1093°C Stress Rupture
Ident.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Hours at 31.0 MN/m^2
1.	0.025	886.1	840.6	15.5	58.6	57.9	5.0	0.6T	>20
2.	0.025	886.7	632.5	21.5	70.3	70.3	7.0	0.6T	>20

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		Room Temperature			2000°F				2000°F Stress Rupture
Ident.	Gauge in.	UTS ksi	YS ksi	% Elong	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Hours at 4.5 ksi
1.	0.010	128.6	122.0	15.5	8.5	8.4	5.0	0.6T	>20
2.	0.010	128.7	91.8	21.5	10.2	10.2	7.0	0.6T	>20

TABLE B-8

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3783

Starting Gauge: 0.076 cm.(0.030 in.)
Initial Micro Grain Size: 0.080 mm
Initial Macro Grain Size: 0.15 cm.(0.06 in.)
Heat Treated at 0.051 cm.(0.020 in.)

	Room Temperature			1093°C			Minimum Bend Radius	1093°C Stress Rupture Hours at 31.0 MN/m^2
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.		
0.025	941.9	702.1	20.0	58.6	58.6	5.0	0.6T	>20

	Room Temperature			2000°F			Minimum Bend Radius	2000°F Stress Rupture Hours at 4.5 ksi
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.		
0.010	136.7	101.9	20.0	8.5	8.5	5.0	0.6T	>20

TABLE B-9

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3693

Starting Gauge: 0.076 cm. (0.030 in.)
Initial Micro Grain Size: 0.160 mm
Initial Macro Grain Size: 0.32 cm. (0.125 in.)
Heat Treated at 0.051 cm. (0.020 in.)

		Room Temperature			1093°C				1093°C
Ident.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
A1	0.025	908.8	642.1	18.5	82.0	79.2	3.0	0.5T	>20
A2	0.025	904.0	655.2	21.5	86.8	77.2	3.5	0.5T	>20
B1	0.025	924.6	649.7	19.5	97.1	92.3	2.0	0.5T	>20
B2	0.025	929.5	659.4	19.5	89.6	86.8	2.5	0.5T	>20
C1	0.025	988.7	782.0	12.5	115.1	115.1	2.0	0.5T	>20
C2	0.025	943.9	678.0	19.5	79.2	79.2	2.5	0.5T	>20

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		Room Temperature			2000°F				2000°F
Ident.	Gauge in.	UTS ksi	YS ksi	% Elong	UTS ksi	YS ksi	% Elong	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
A1	0.010	131.9	93.2	18.5	11.9	11.5	3.0	0.5T	>20
A2	0.010	131.2	95.1	21.5	12.6	11.2	3.5	0.5T	>20
B1	0.010	134.2	94.3	19.5	14.1	13.4	2.0	0.5T	>20
B2	0.010	134.9	95.7	19.5	13.0	12.6	2.5	0.5T	>20
C1	0.010	143.5	113.5	12.5	16.7	16.5	2.0	0.5T	>20
C2	0.010	137.0	98.4	19.5	11.5	11.5	2.5	0.5T	>20

TABLE B-10

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3707

Starting Gauge: 0.102 cm.(0.040 in.)
Initial Micro Grain Size: 0.015 mm
Initial Macro Grain Size: 0.02 cm.(0.01 in.)
Heat Treated at 0.051 cm.(0.020 in.)

	Room Temperature			1093°C				1093°C Stress Rupture
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Hours at $31.0\ MN/m^2$
0.025	942.6	706.9	20.0	82.0	74.4	6.5	1.0T	>20

	Room Temperature			2000°F				2000°F Stress Rupture
Gauge in.	UTS ksi	YS ksi	% Elong	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Hours at 4.5 ksi
0.010	136.8	102.6	20.0	11.9	10.8	6.5	1.0T	>20

TABLE B-11

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3702

Starting Gauge: 0.102 cm.(0.040 in.)
Initial Micro Grain Size: 0.140 mm
Initial Macro Grain Size: 0.32 cm.(0.125 in.)

		Room Temperature			1093° C				1093° C
Ident.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
1.	0.025	869.5	632.5	17.0	80.6	78.6	3.5	2.5T	>20
2.	0.025	958.4	830.9	10.0	72.3	67.5	4.0	2.0T	>20

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		Room Temperature			2000° F				2000° F
Ident.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
1.	0.010	126.2	91.8	17.0	11.7	11.4	3.5	2.5T	>20
2.	0.010	139.1	120.6	10.0	10.5	9.8	4.0	2.0T	>20

1. Heat treated at 0.051 cm.(0.020 in.) and 0.025 cm.(0.010 in.)

2. Heat treated at 0.071 cm.(0.028 in.) and 0.036 cm.(0.014) and 0.025 cm.(0.010 in.)

TABLE B-12

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3695

Starting Gauge: 0.051 cm. (0.020 in.)
Initial Micro Grain Size: 0.120 mm
Initial Macro Grain Size: 0.25 cm. (0.10 in.)

		Room Temperature			1093°C				1093°C
Ident.	Gauge cm	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
1.	0.025	890.9	609.1	16.0	115.1	114.4	2.0	3.5T	>20
2.	0.025	914.3	622.9	18.0	99.2	99.2	3.0	3.5T	>20
3.	0.025	791.7	522.3	14.5	104.0	100.6	2.0	3.5T	>20
4.	0.025	853.7	555.3	15.0	112.3	112.3	2.0	3.5T	>20
5.	0.025	846.1	563.6	15.5	115.1	115.1	2.5	3.5T	>20

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		Room Temperature			2000°F				2000°F
Ident.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
1.	0.010	129.3	88.4	16.0	16.7	16.6	2.0	3.5T	>20
2.	0.010	132.7	90.4	18.0	14.4	14.4	3.0	3.5T	>20
3.	0.010	114.9	75.8	14.5	15.1	14.6	2.0	3.5T	>20
4.	0.010	123.9	80.6	15.0	16.3	16.3	2.0	3.5T	>20
5.	0.010	122.8	81.8	15.5	16.7	16.7	2.5	3.5T	>20

TABLE B-13

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3690

Starting Gauge: 0.051 cm.(0.020 in.)
Initial Micro Grain Size: 0.120
Initial Macro Grain Size: 0.25 cm.(0.10 in.)

		Room Temperature			1093°C				1093°C Stress Rupture
Ident.	Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Hours at 31.0 MN/m^2
1.	0.025	912.2	565.0	16.0	133.0	133.0	2.0	3.5T	>20
2.	0.025	901.2	660.1	16.0	128.2	128.2	2.0	3.5T	>20
3.	0.025	784.1	573.9	8.0	98.5	94.3	2.5	3.5T	>20
4.	0.025	850.2	566.3	14.0	113.0	113.0	2.0	3.5T	>20
5.	0.025	859.2	611.1	14.5	110.9	110.9	2.0	3.5T	>20

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		Room Temperature			2000°F				2000°F Stress Rupture
Ident.	Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Hours at 4.5 ksi
1.	0.010	132.4	82.0	16.0	17.2	17.2	2.0	3.5T	>20
2.	0.010	130.8	95.8	16.0	19.3	19.3	2.0	3.5T	>20
3.	0.010	113.8	83.3	8.0	18.6	18.6	2.5	3.5T	>20
4.	0.010	123.4	82.2	14.0	14.3	13.7	2.0	3.5T	>20
5.	0.010	124.7	88.7	14.5	16.4	16.4	2.0	3.5T	>20

TABLE B-14

MECHANICAL PROPERTIES OF COLD ROLLED HEAT 3497

Starting Gauge: 0.203 cm.(0.080 in.)
Initial Micro Grain Size: 0.110 mm
Initial Macro Grain Size: 0.23 cm.(0.09 in.)
Heat Treated After Each 50% Reduction

	Room Temperature			1093°C				1093°C
Gauge cm.	UTS MN/m^2	YS MN/m^2	% Elong.	UTS MN/m^2	YS MN/m^2	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 31.0 MN/m^2
0.025	1022.5	850.9	15.0	76.5	73.0	5.0	1.5T	>20

	Room Temperature			2000°F				2000°F
Gauge in.	UTS ksi	YS ksi	% Elong.	UTS ksi	YS ksi	% Elong.	Minimum Bend Radius	Stress Rupture Hours at 4.5 ksi
0.010	148.4	123.5	15.0	11.1	10.6	5.0	1.5T	>20

APPENDIX C

TDNiCr Exploratory Studies

1. Chromium Oxide Control

The Cr_2O_3 particles in the microstructure of TDNiCr are considered to be a contaminant. Efforts were carried out in order to reduce the size and content of these particles. The approaches employed were powder reduction prior to compaction and carbon additions in conjunction with special sinter cycles and/or compaction techniques.

a. Hydrogen Reduction of Powder

Two 68.0 kg.(150 lb.) batches of powder were prepared in a conventional manner and identified as Heats 3783 and 3784. Each batch of powder was then subjected to a temperature of 850° C(1562° F) for a period of 2-1/2 hours in a reducing atmosphere of dry hydrogen. Subsequent to removal of the powder batches from the furnace, one batch was screened through a 20 mesh sieve and the second batch was screened through a 30 mesh sieve in order to break up any coarse agglomerates that might have been formed during the treatment cycle. No differences could be observed between the two batches of powder.

In both Heats 3783 and 3784 the amount of powder that could be loaded into the hydrostatic compaction hardware was limited to approximately 63.5 kg.(140 lb.) rather than the 68.0 kg.(150 lb.) normally attainable. Therefore, a change in powder character was indicated as a result of the reduction operation.

Additional processing was carried out by standard procedures. These consisted of compaction at 413.4 MN/m^2 (60.0 ksi), sintering at 954° C(1750° F) and roll consolidation at 1010° C (1850° F) to a hot rolled slab thickness of 2.54 cm.(1.0 in.).

During both the sintering operation in the Harper Furnace and the preheat cycle for roll consolidation in the Pereny Furnace, a chromatograph was utilized to monitor the composition of the gases exiting from the canned billets. It was observed that large volumes of nitrogen gas were evolved in the temperature range of 910° C(1670° F) to 938° C(1720° F). This evolution was found to decrease at the initiation of the 954° C(1750° F) soak and was completely gone at the end of the two hour soak. Since nitrogen evolution has not been noted for billets prepared by standard procedures, it was concluded that nitrogen must have been picked up during the above heating cycle prior to the compaction operation.

At the 2.54 cm.(1.0 in.) stage of processing, samples were obtained from the slab for thoria size determination and metallographic evaluation.

Metallographic examination in the as-polished condition exhibited what appeared to be a greater quantity of dispersed Cr_2O_3 particles than found in comparably fabricated material containing powder not subjected to the special heat treat cycle prior to compaction.

Thoria sizes of both Heats 3783 and 3784 in the as hot rolled condition at the 2.54 cm.(1.0 in.) thickness were found to be 15 mμ. For this reason, both heats were subjected to a temperature of 1093° C(2000° F) for two hours subsequent to conditioning in order to grow the thoria to a more satisfactory size prior to the 760° C(1400° F) rolling.

After conditioning, canning and thoria size control heat treatment, Heats 3783 and 3784 were isothermally rolled to intermediate gauge plate of 0.254 cm.(0.1 in.) thickness. At this stage of processing, samples were again secured. Thoria sizes were again determined on the as-rolled plate and chemical analysis was performed.

Thoria sizes of Heats 3783 and 3784 at a thickness of 0.254 cm.(0.1 in.) were found to be 20 mμ and 19 mμ respectively.

Chemical analysis results for Heats 3783 and 3784 are shown below:

Constituent %	Heat 3783	Heat 3784
Thoria	2.05	2.06
Chromium	19.64	19.67
Carbon	.033	.030
Sulfur	.005	.003
Nitrogen	.006	.006
Total Oxygen	.731	.622

It may be noted that total oxygen appears to be slightly higher than that generally experienced on TDNiCr sheet. For example, conventional material is in the range of 3500 to 5500 ppm where the results on Heats 3783 and 3784 run in the range of 6200 to 7300 ppm which verified the greater quantity of Cr_2O_3 present according to metallographic evaluation. It was, therefore, concluded that this procedure would not be employed for any future processing.

b. Carbon Addition

Two units identified as Heats 3822 and 3823 weighing 45.4 kg.(100 lb.) each were prepared with carbon additions of 50 and 75 grams respectively. They were conventionally compacted at 413.3 MN/m² (60.0 ksi) and subsequently placed in mild steel "loose type" cans for sintering in the Harper Furnace.

Sintering was carried out using the conventional stepped temperature cycle and a maximum temperature of 1177° C(2150° F). The effluent hydrogen was monitored for carbon and oxygen containing gases by gas chromatograph. Early in the cycle carbon was lost in the form of methane. At the end of the cycle at temperatures of 1149° C(2100° F) and above carbon monoxide was evolved. The sinter run was terminated after two hours at 1177° C(2150° F) to avoid excessive ThO_2 growth and because the rate of carbon monoxide evolution had dropped considerably.

Sample drillings from edge to center of the two sintered slabs showed considerable carbon loss from the surface material but 85 to 90% residual at the middle of the slab; see Table C-1. Sample drill holes were refilled by insertion of mechanically compacted slugs of TDNiCr powder. These were compacted at the same pressure used for the slabs themselves. The units were then recanned in conventional roll consolidation cans, placed

TABLE C-1

CARBON ANALYSES FOR SLABS 3822 AND 3823

Heat No.	% Carbon added to powder	Slab Condition	% Carbon vs. Depth					
			Surface---------------------------→					Center
3822	0.11	as-sintered	0.02	0.04	0.06	0.07	0.10	0.08
		consolidated		0.02				0.03
3823	0.16	as-sintered	0.03	0.04		0.10	0.14	0.14
		consolidated		0.06				0.05

in the Pereny Furnace for preheat at 1177° C(2150° F) and subsequently roll consolidated and hot rolled by standard procedures to a slab thickness of 2.54 cm.(1.0 in.).

The preheat for roll consolidation was also monitored with the gas chromatograph. Carbon monoxide evolution was of the same order of magnitude as that evolved in the previous sinter cycle.

The total preheat time at 1177° C(2150° F) was a period of four hours.

After decanning at the 2.54 cm.(1.0 in.) slab stage of processing, the two units were chemically and metallographically evaluated. Analysis for carbon at this stage showed considerable carbon removal even to the center of the slabs (Table C-1). Residual levels are not much higher than those obtained in slabs made from powder without carbon additions. This significant carbon removal in the roll consolidation preheat was unexpected in view of the limited removal obtained in the original sinter cycle. Metallographic examination of the rolled slab showed considerable reduction in residual Cr_2O_3 particles though cleanliness improvement was variable, ranging from slightly better than a conventionally processed slab to considerably better. This same variable improvement was noted in sheet product from the two slabs. Cleanliness differences between the two slabs or between their sheet products did not appear significant.

As a means of enhancing the reduction of residual Cr_2O_3 by a carbon addition to the TDNiCr powder, another approach was evaluated. This consisted of compaction by powder rolling of the TDNiCr powder with the carbon addition. It was anticipated that sintering of a composite of thin compacted sheet would enable more effective and consistent carbon reduction of Cr_2O_3 because of the accessibility of the interior of the unit to the sinter gases and the ease of evolution of the reduction products.

Two 4.5 kg.(10 lb.) units, Heats 3913 and 3914, were processed to slab by roll consolidation. The powder in 3913 was standard TDNiCr powder to which 1800 ppm carbon was added. The powder for Heat 3914 was subjected to a heat treatment in a reducing atmosphere after the addition of the carbon and before roll compaction.

Each of these heats of powder was compacted into thin strips by powder rolling carried out on a Fenn rolling mill equipped with a powder feed hopper and a pair of mating rolls. The male roll had a 25.4 cm.(10 in.) wide face which fit between the shoulders on the female roll. Powder feeding of the heat treated powder was satisfactory because of agglomeration which had taken place in the heat treatment. The untreated powder had poor flowability and did not feed well. Irregular thickness and much strip breakage occurred during the roll compaction of this powder.

The powder rolled strip was cut to size and stacked in a roll consolidation can. The units were given a combined sinter and heatup for roll consolidation to prevent pickup of oxygen which might have occurred had they been exposed to air for recanning for a separate roll consolidation step. The combined sinter-consolidation heatup utilized temperature arrests at 204°, 315.6°, 454° C(400°, 600°, 850° F) and a final 1121° C(2050° F) at which points -56.7° C(-70° F) effluent hydrogen dewpoints were achieved.

The roll consolidation slabs were sampled along a mid-section line so that microexamination of edge and center could be made. Heat 3913 whose powder was unheat treated showed approximately 90 percent of the cross section to be devoid of visible Cr_2O_3 particles. Islands of Cr_2O_3 rich material could be observed on both the edge and center samples. On the other hand, Heat 3914, whose powder was subjected to a reduction heat treatment prior to powder rolling, showed no reduction in Cr_2O_3 over standard product.

This investigation demonstrated the feasibility of utilizing a carbon additive to the TDNiCr powder in conjunction with a composite of thinly compacted material as a means of effecting a reduction of residual Cr_2O_3 particles in the finished product microstructure. However, this technique would require additional refinement prior to adoption as a standard procedure for the manufacture of TDNiCr sheet. For example, a method of assuring greater homogeneity of the carbon additive is required, improvement of flowability of standard powder for powder rolling, and finally, qualification of the powder rolling technique of compaction in lieu of hydrostatic compaction. Since this effort would fall outside the present scope of this contract, no change in the standard process was considered during the Part II program.

2. Alternate Powder Compaction Techniques

Two powder compaction processes alternate to the hydrostatic compaction of single units were evaluated at small scale. Composite units of powder rolled strip and mechanically compacted wafers were processed to sheet for evaluation.

One 4.54 kg.(10 lb.) unit consisting of 5.71 cm.(2-1/4 in.) high stack of roll compacted pieces as shown in Figure C-1 was identified as Heat 3800. The stack of compacted pieces was placed in a mild steel can as shown in Figure C-2, both before and after assembly. The unit was subsequently rolled to 0.051 and 0.127 cm.(0.025 and 0.050 in.) thick sheet by conventional procedures.

In the case of the mechanically compacted wafers, two 4.54 kg.(10 lb.) units were prepared. One unit contained wafers with a zinc stearate additive for lubrication and a second one contained wafers without the additive. These units were identified as Heats 3791 and 3790 respectively. A typical wafer stack along with roll consolidation can components is shown in Figure C-3. Figure C-4 shows the roll consolidation can containing a typical wafer stack before

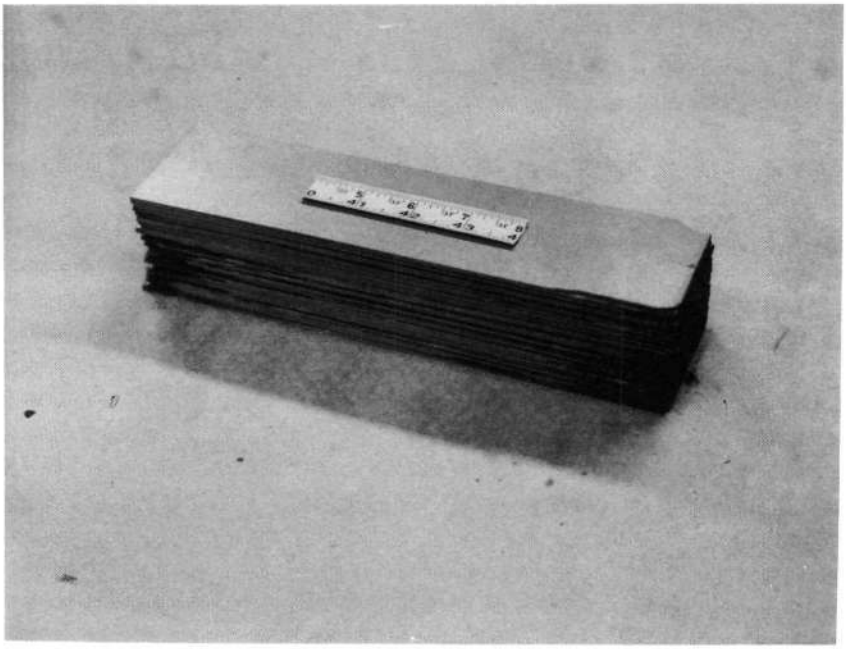

FIGURE C-1

ROLL COMPACTED STACK - HEAT 3800

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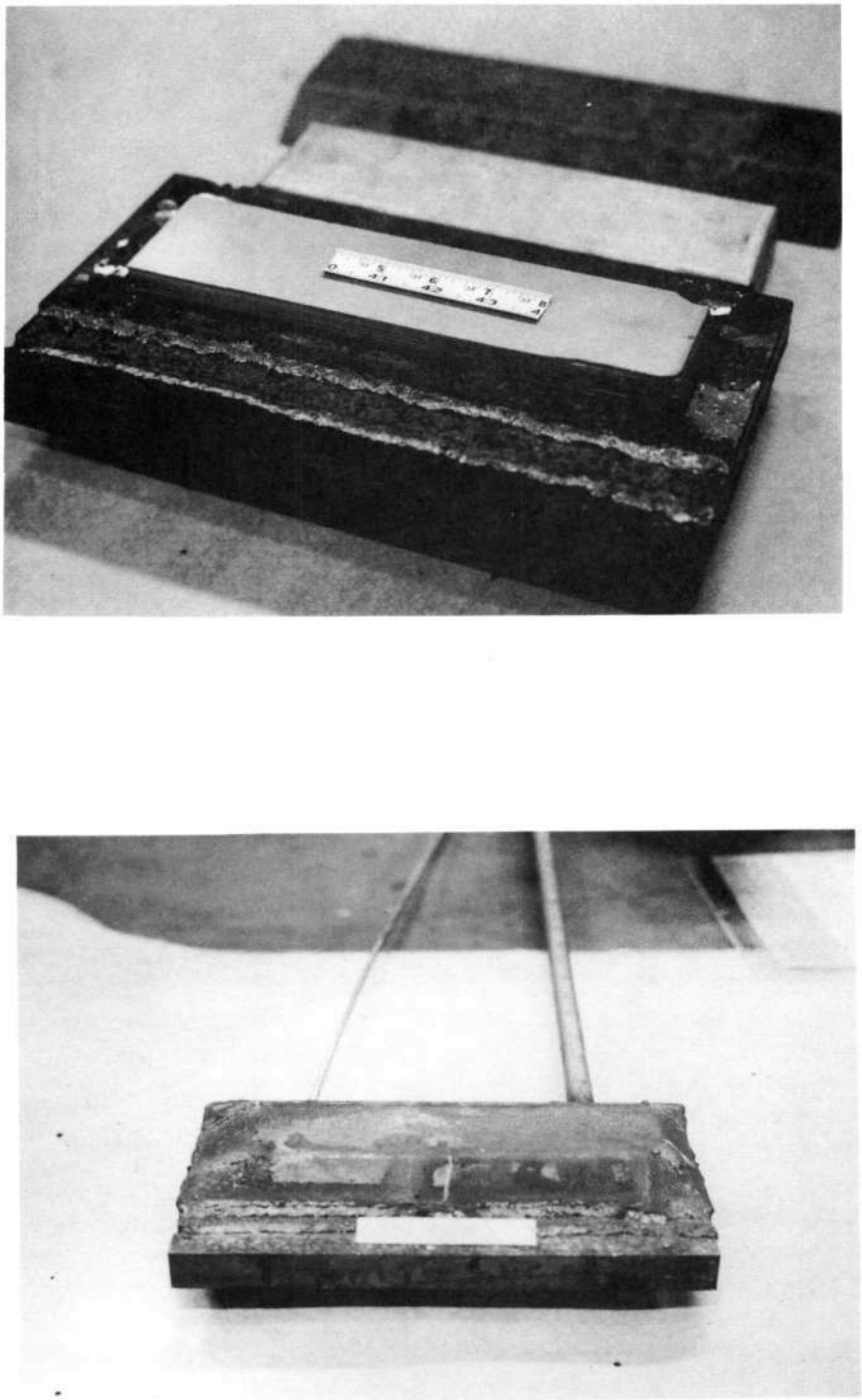


FIGURE C-2

POWDER ROLLED MATERIAL - CAN ASSEMBLY - HEAT 3800

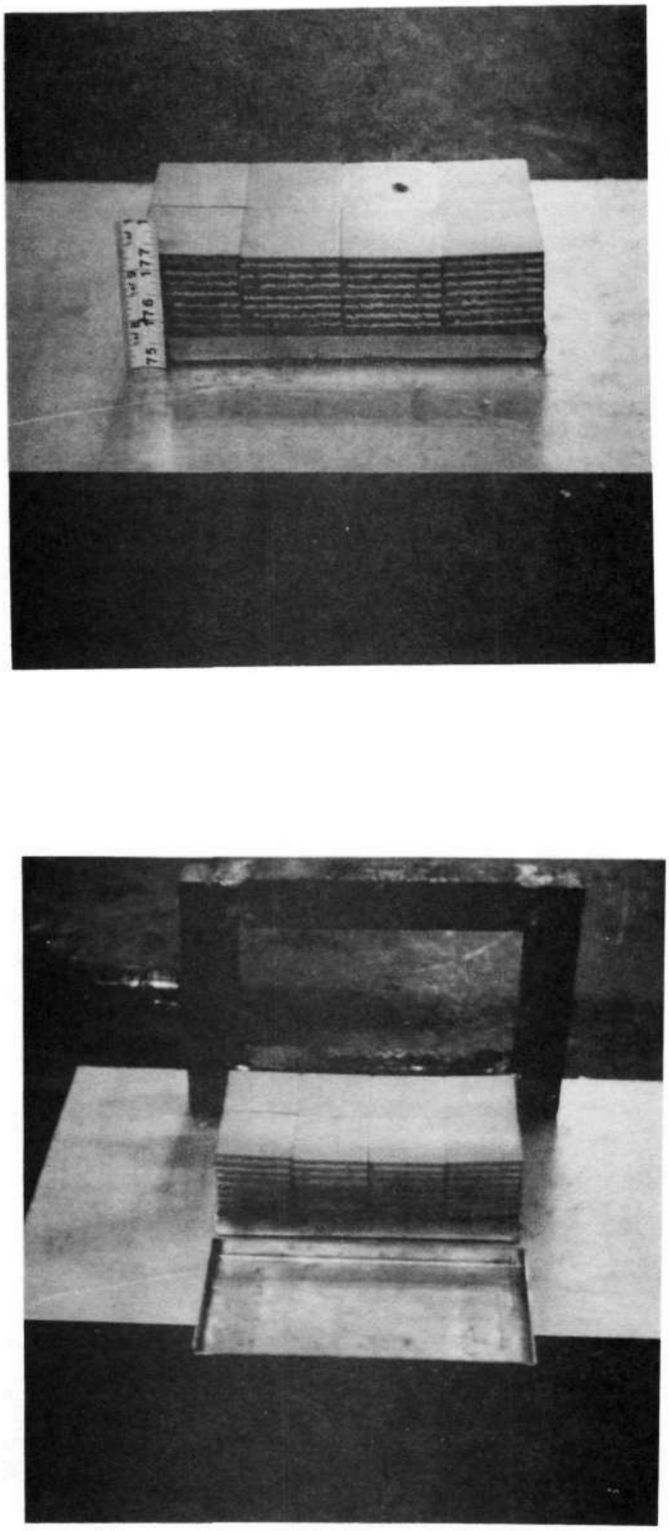


FIGURE C-3

MECHANICAL WAFER STACK AND CAN PARTS - HEAT 3790

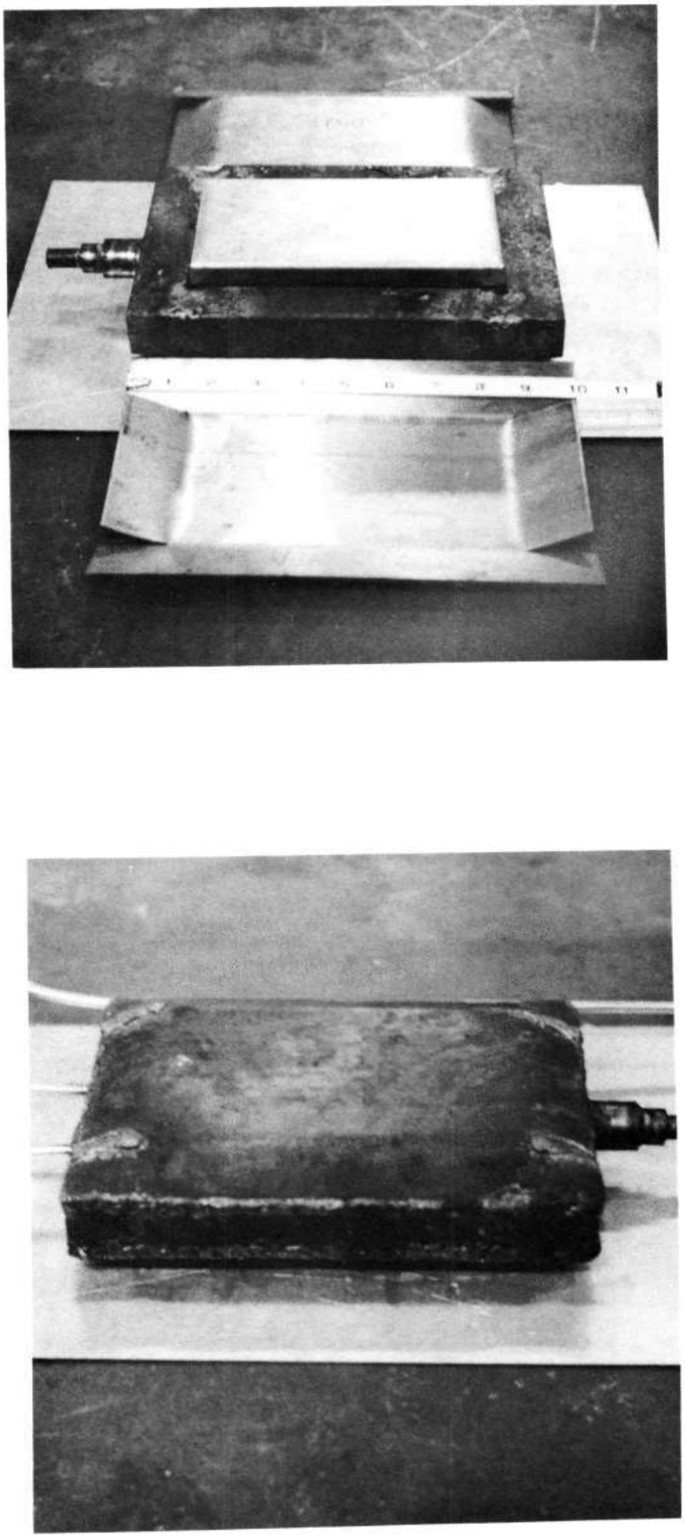


FIGURE C-4

MECHANICAL WAFER UNIT - CAN ASSEMBLY - HEAT 3790 and after weld positioning of the can cover plates. As in the case of the powder rolled unit, these units were further fabricated to 0.051 and 0.127 cm.(0.025 and 0.050 in.) thick sheet by conventional procedures for further evaluation.

Chemical analysis data obtained at the 0.254 cm.(0.100 in.) intermediate gauge plate stage of processing from Heats 3790, 3791 and 3800 are shown in Table C-2. As indicated, Heats 3790 and 3791 contain rather high total oxygen contents which are comparable to those reported for Heats 3783 and 3784 in this report. Powder for all four of these heats was subjected to a special heat treatment in a reducing atmosphere prior to compaction in an effort to reduce the size and content of Cr_2O_3 particles. Powder utilized for Heat 3800 had been given a similar treatment; however, a subsequent sinter and roll consolidation temperature of 1177° C(2150° F) instead of a conventional 1010° C(1850° F) temperature apparently was responsible for some reduction in the total oxygen and carbon contents.

Thoria sizes were obtained at the hot rolled-consolidated slab stage of processing on Heats 3790, 3791 and 3800. They were found to be 14.0, 15.5 and 20.0 mμ respectively. Accordingly, Heats 3790 and 3791 were subjected to a 1093° C(2000° F) heat treatment for two hours prior to further fabrication in order to attain proper thoria size.

Evaluation of the product sheet for soundness indicated that the sheet contained no trace of any flaws resulting from its lamellar origin.

TABLE C-2

CHEMICAL ANALYSIS FOR HEATS 3790, 3791 AND 3800

Heat No.	Chemical Constituents in Percent C	Cr	S	ThO_2	Total O_2	Ni
3790	0.023	20.38	0.004	2.21	0.770	Bal.
3791	0.016	19.70	0.005	2.21	0.675	Bal.
3800	0.007	19.60	0.003	2.25	0.594	Bal.

Mechanical properties were not utilized as an evaluation criterion since the small scale developmental units were not subjected to sufficient 760°C(1400°F) deformation to establish proper microstructure for good properties.

Follow-on work with compaction composites included the fabrication of two full scale units of approximately 45.4 kg. (100 lb.) of mechanically compacted slabs. Powder for both units had been subjected to the same pre-compaction reduction cycle as was used for the small scale units.

Mechanical compaction was carried out at Fansteel's North Chicago Plant on a Baldwin 17.8 MN(2000 ton) press. The compacts were pressed to 10.2 x 30.5 x 1.2 cm.(4 x 12 x .480 in.) and weighed approximately 2.0 kg.(4.4 lb.) each. Calculated density of the compacts was 60 to 65% of theoretical.

The first composite, Heat 3839, was consolidated by roll consolidation. Seven layers of compacts were assembled in a stack 30.5 cm.(12 in.) square and 8.3 cm.(3-1/4 in.) high with the long dimension alternated in each succeeding layer. Figure C-5 shows the stacking. The weight of the composite was 42 kg.(93 lb.) and the compacted slabs were sintered at 1121°C(2050°F) in the stacked configuration. They were then recanned for roll consolidation. Processing to 2.54 cm. (1.0 in.) slab stage was by conventional techniques. The slab is shown as-rolled in Figure C-6 and after decanning in Figure C-7. Bonding of the compacts was effective except for some of the interfaces that were transverse to the rolling

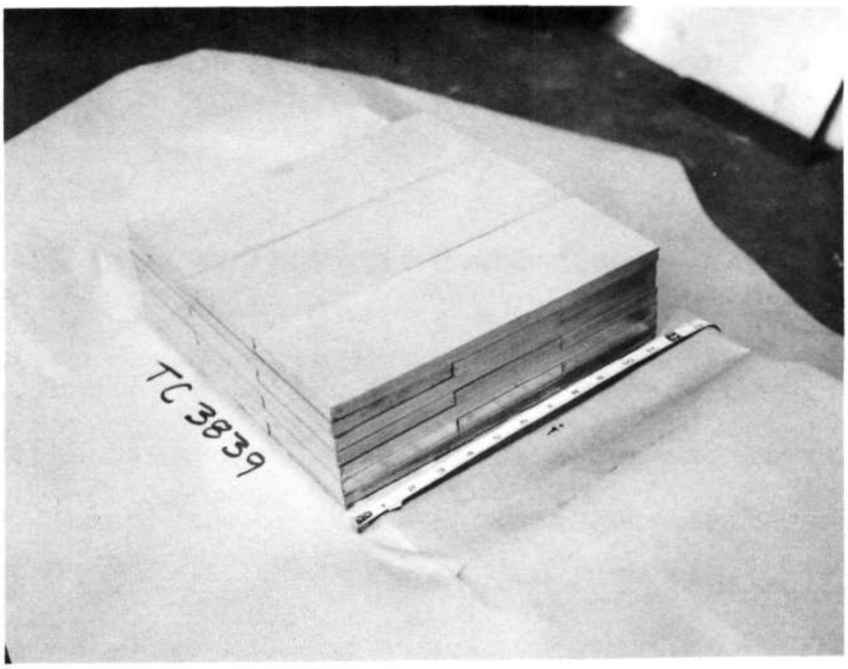


FIGURE C-5

LARGE SCALE MECHANICAL COMPACT UNIT - HEAT 3839

FIGURE C-6

MECHANICAL COMPACT UNIT AS-ROLLED - HEAT 3839

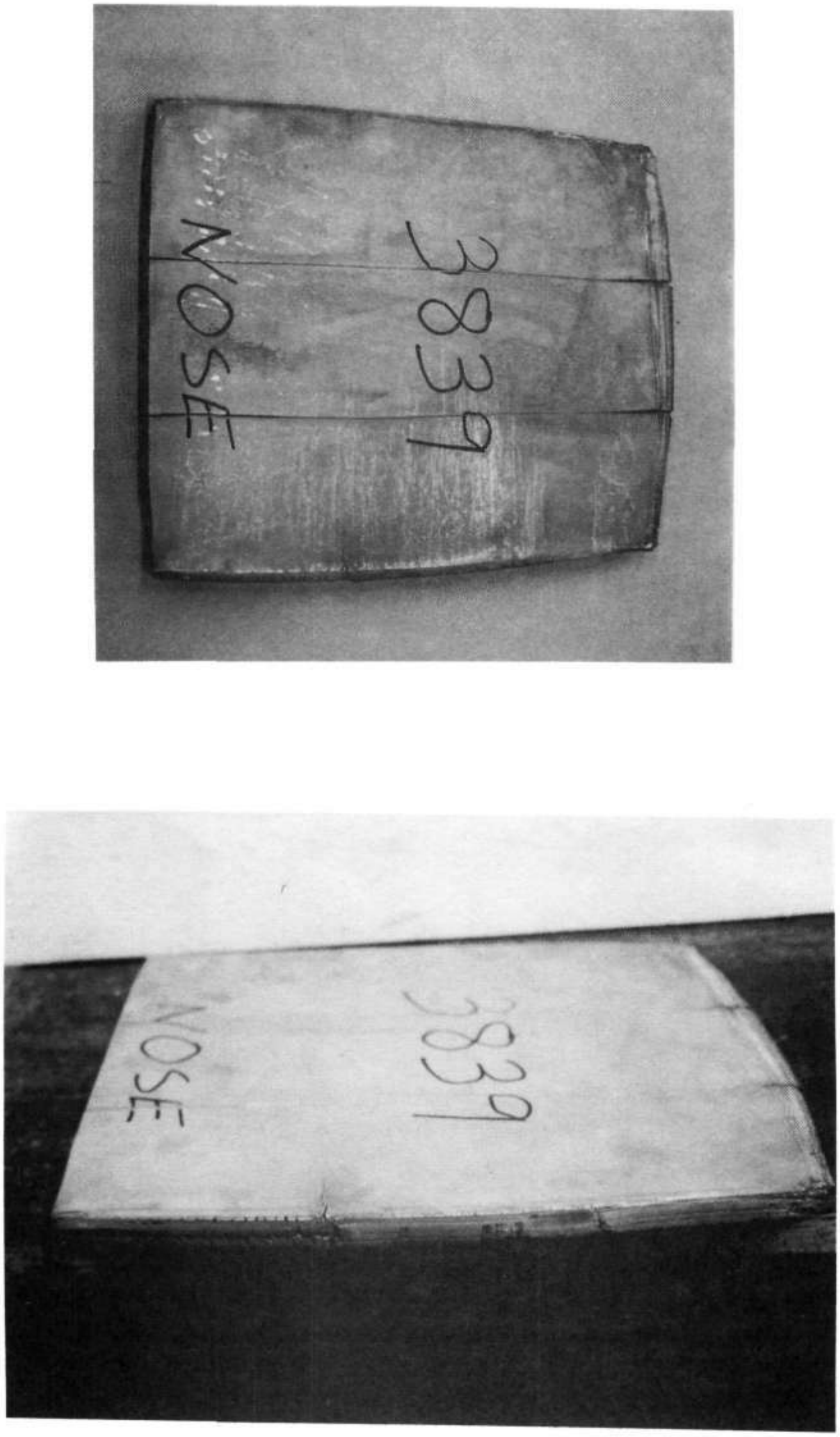


FIGURE C-7

MECHANICAL COMPACT UNIT AFTER DECANNING - HEAT 3839 direction. Apparently, the longitudinal tensile forces of the rolling process tend to inhibit bonding in the transverse direction. The decanned slab was conditioned by milling and cropping and examined by dye penetrant which showed no defects. In subsequent rolling to sheet by conventional techniques, however, heavy edge cracking occurred, Figure C-8. The origin of the cracking is believed to be in undiscovered defects residual from imperfect bonding in roll consolidation.

The second slab composite was Heat 3815 which was stacked and sintered by the same procedures used for Heat 3839 with the exception that eight layers were used resulting in a total weight of 48 kg.(106 lb.). This unit was forge consolidated in a closed die having dimensions of 37 x 37 x 4.4 cm.(14 x 14 x 1-3/4 in.). In the same manner as preheating for roll consolidation, a hydrogen flow was used during heating for forging. A forging temperature of 1121°C(2050°F) was used. Figure C-9 shows the slab after forging. Subsequent to forging the slab was conditioned by pickle decanning and milling. It was crack checked by dye penetrant. Bonding of the compacts appeared better than with roll consolidation. In subsequent rolling to sheet the forged composite also exhibited edge cracks, presumably at sites of undetected imperfect bonds.

The feasibility of producing slabs by consolidation of mechanically compacted units has been demonstrated. However, additional experimentation would be required to assure crack-free consolidation on a reproducible basis.

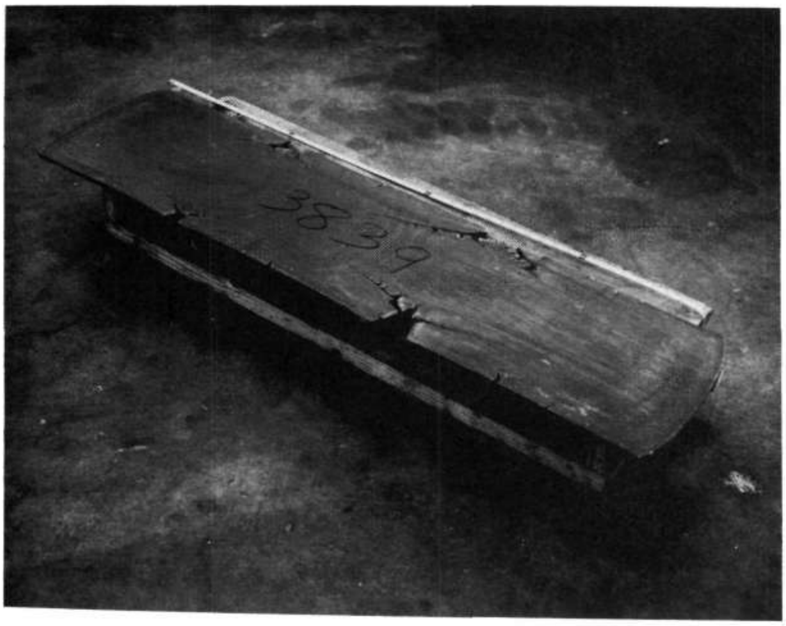


FIGURE C-8

MECHANICAL COMPACT UNIT DECANNED AFTER WARM ROLLING
HEAT 3839

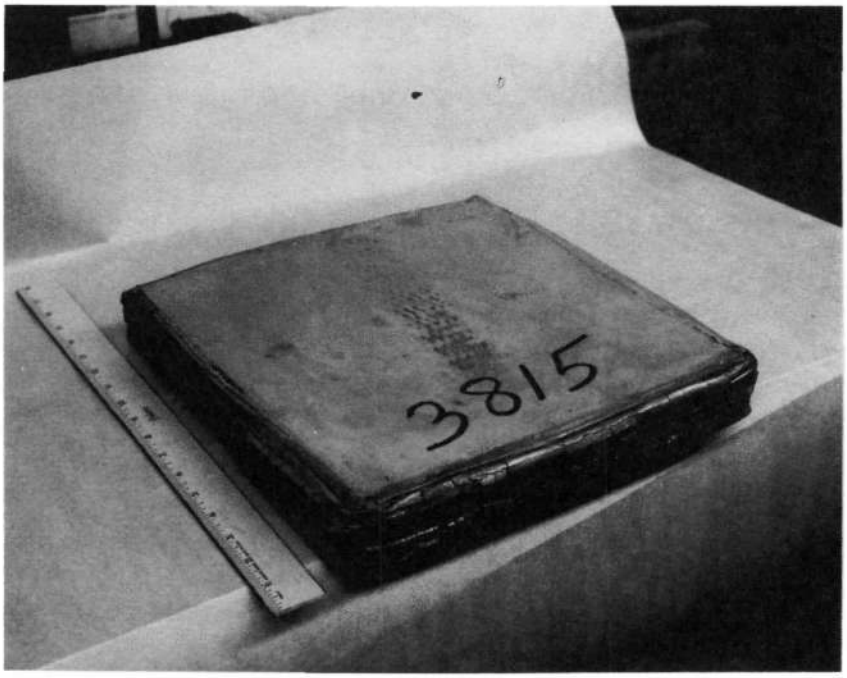


FIGURE C-9

FORGED MECHANICAL COMPACT UNIT AFTER DECANNING
HEAT 3815

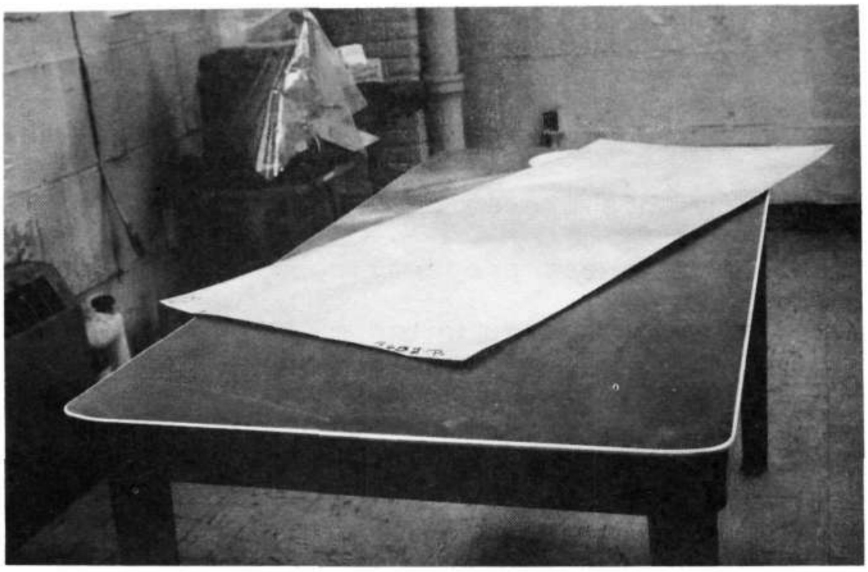
3. Creep Flattening

It was found during Part I that TDNiCr is capable of being flattened in the unrecrystallized condition in the 704-816° C (1300-1500° F) temperature range. Previously material was sent to Titanium Metals Corp. of America in Toronto, Ohio, where it was creep flattened at 760° C(1400° F) in a specially designed fixture. Upon subsequent recrystallization at 1177° C(2150° F) however, the flatness improvement was lost. A creep flattening fixture was therefore designed and was fabricated for flattening in the Fansteel Baltimore heat treatment furnace. It consisted of two 0.635 cm.(1/4 in.) stainless steel plates weighing 42.6 kg.(94 lb.) which were clamped to the bottom of a TDNiCr sheet such that a uniaxial load was applied. TDNiCr bolts and nuts were used for attachment. It was hoped that this load would creep flatten the sheets in the 704-816° C (1300-1500° F) temperature range, and would maintain this flatness up to 1177° C(2150° F). No metallurgical changes were expected to occur since the applied stress was only one tenth the one hundred hour rupture strength at 1177° C(2150° F).

Figure C-10 shows the results of this work. Two 0.025 cm. (0.010 in.) sheets from Heat 3689 with identical flatness before heat treatment, were utilized. One sheet was inserted in the creep flattening fixture and the other simply hung, which is standard procedure. Flatness readings after heat treatment were:

Creep Flattened	- 0.5%
Control (Standard Procedures)	- 7.0%

No deleterious quality effects resulted during heat treatment.



Control Sheet - 7.0% Flatness

Creep Flattened Sheet - 0.5% Flatness

FIGURE C-10

COMPARISON OF CREEP FLATTENED VS. CONTROL SHEETS

Mechanical property results were obtained for both pieces and are shown in Table C-3. Apparently no property degradation resulted from this operation. Both the creep flattened sheet and the control sheet property results are listed in Table C-3. The type of damage which might be expected to occur are cracks normal to the direction of loading. Consequently, many longitudinal bend tests were run on the creep flattened sheet. If cracks did exist, the minimum bend radius would have been increased. However, the longitudinal bend radius was less than 3T for each test.

TABLE C-3

MECHANICAL PROPERTIES OF CREEP FLATTENING EXPERIMENT
HEAT 3689

	Room Temperature					1093°C(2000°F)						1093°C(2000°F) Stress Rupture
Sheet	UTS MN/m^2	ksi	YS MN/m^2	ksi	% Elong.	UTS MN/m^2	ksi	YS MN/m^2	ksi	% Elong.	Min. Bend Radius	Hrs. at 31.0 MN/m^2 (4.5 ksi)
Control	760.7	110.4	524.3	76.1	18.0	85.4	12.4	85.4	12.4	2.0	3T	>20
Flattened	727.6	105.6	562.9	81.7	18.0	70.3	10.2	70.3	10.2	2.0	3T	>20

APPENDIX D

Production TDNiCr

Flat Product Inventory

TABLE D-1

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.0076x30.48x60.96	.100	3528	520	Cold Rolled
1	0.0076x50.16x62.23	.168	3528	521	"
1	0.0076x50.80x62.23	.172	3528	522	"
1	0.0076x50.80x62.23	.168	3528	523	"
1	0.0076x50.80x62.23	.181	3528	524	"
1	0.0076x50.80x62.23	.181	3528	525	"
1	0.0076x50.80x62.23	.218	3528	526	"
1	0.0076x50.80x62.23	.213	3528	527	"
1	0.0076x50.80x62.23	.213	3528	528	"
1	0.0076x50.80x62.23	.168	3528	529	"
1	0.0076x50.80x62.23	.213	3528	530	"
1	0.0076x50.80x62.23	.186	3528	531	"
1	0.0076x50.80x62.23	.181	3528	532	"
1	0.0076x50.80x62.23	.209	3528	533	"
1	0.0076x50.80x62.23	.209	3528	534	"
1	0.0076x50.80x62.23	.209	3528	535	"
1	0.0076x50.80x62.23	.222	3528	536	"
1	0.0076x50.80x62.23	.222	3528	537	"
1	0.0076x50.80x62.23	.222	3528	538	"
1	0.0076x50.80x62.23	.218	3528	539	"
SUB-TOTAL	20 pieces	3.880	3528		
1	0.0076x60.96x579.12	1.81	3702	-	Cold Rolled
1	0.0076x48.26x243.84	.45	3702	-	"
1	0.0076x50.48x708.66	2.40	3702	-	"
1	0.0076x61.28x384.17	1.68	3702	-	"
1	0.0076x62.23x83.82	.36	3702	-	"
SUB-TOTAL	5 pieces	6.72	3702		
1	0.0076x49.53x212.72	.68	3524	-	Cold Rolled
1	0.0076x50.48x80.64	.32	3524	-	"
1	0.0076x50.80x32.38	.09	3524	-	"
SUB-TOTAL	3 pieces	1.09	3524		

TABLE D-1

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.003x12x24	0.22	3528	520	Cold Rolled
1	0.003x19-3/4x24-1/2	0.37	3528	521	"
1	0.003x20x24-1/2	0.38	3528	522	"
1	0.003x20x24-1/2	0.37	3528	523	"
1	0.003x20x24-1/2	0.40	3528	524	"
1	0.003x20x24-1/2	0.40	3528	525	"
1	0.003x20x24-1/2	0.48	3528	526	"
1	0.003x20x24-1/2	0.47	3528	527	"
1	0.003x20x24-1/2	0.47	3528	528	"
1	0.003x20x24-1/2	0.37	3528	529	"
1	0.003x20x24-1/2	0.47	3528	530	"
1	0.003x20x24-1/2	0.41	3528	531	"
1	0.003x20x24-1/2	0.40	3528	532	"
1	0.003x20x24-1/2	0.46	3528	533	"
1	0.003x20x24-1/2	0.46	3528	534	"
1	0.003x20x24-1/2	0.46	3528	535	"
1	0.003x20x24-1/2	0.49	3528	536	"
1	0.003x20x24-1/2	0.49	3528	537	"
1	0.003x20x24-1/2	0.49	3528	538	"
1	0.003x20x24-1/2	0.48	3528	539	"
SUB-TOTAL	20 pieces	8.54	3528		
1	0.003x24x228	4.00	3702	-	Cold Rolled
1	0.003x19x96	1.00	3702	-	"
1	0.003x19-7/8x279	5.30	3702	-	"
1	0.003x24-1/8x151-1/4	3.70	3702	-	"
1	0.003x24-1/2x33	0.80	3702	-	"
SUB-TOTAL	5 pieces	14.80	3702		
1	0.003x19-1/2x83-3/4	1.50	3524	-	Cold Rolled
1	0.003x19-7/8x31-3/4	0.70	3524	-	"
1	0.003x20x12-3/4	0.20	3524	-	"
SUB-TOTAL	3 pieces	2.40	3524		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.0076x49.53x108.90	.36	3523	-	Cold Rolled
1	0.0076x50.80x306.70	1.13	3523	-	"
1	0.0076x51.12x82.87	.23	3523	-	"
1	0.0076x50.48x124.46	.41	3523	-	"
1	0.0076x50.80x192.40	.54	3523	-	"
1	0.0076x50.16x153.04	.50	3523	-	"
1	0.0076x51.12x89.53	.27	3523	-	"
1	0.0076x51.12x140.97	.41	3523	-	"
SUB-TOTAL	8 pieces	3.86	3523		
1	0.0076x58.42x609.60	2.72	3695	-	Cold Rolled
SUB-TOTAL	1 piece	2.72	3695		
1	0.0140x45.72x109.22	.59	3437	706	Cold Rolled
1	0.0140x45.72x109.22	.59	3437	707	"
SUB-TOTAL	2 pieces	1.18	3437		
1	0.0140x62.86x396.24	2.90	3783	-	Cold Rolled
1	0.0140xWxL	.45	3783	-	"
SUB-TOTAL	2 pieces	3.35	3783		
1	0.0140x67.63x457.20	3.95	3792	-	Cold Rolled
1	0.0140x67.94x426.72	3.81	3792	-	"
SUB-TOTAL	2 pieces	7.76	3792		
1	0.0140x60.96x731.52	5.44	3796	-	Cold Rolled
SUB-TOTAL	1 piece	5.44	3796		
1	0.0140x65.09x548.64	4.63	3794	-	Cold Rolled
SUB-TOTAL	1 piece	4.63	3794		
1	0.0140x57.15x335.28	2.22	3795	-	Cold Rolled
1	0.0140x58.42x731.52	5.44	3795	-	"
SUB-TOTAL	2 pieces	7.66	3795		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.003x19-1/2x42-7/8	0.80	3523	-	Cold Rolled
1	0.003x20x120-3/4	2.50	3523	-	"
1	0.003x20-1/8x32-5/8	0.50	3523	-	"
1	0.003x19-7/8x49	0.90	3523	-	"
1	0.003x20x75-3/4	1.20	3523	-	"
1	0.003x19-3/4x60-1/4	1.10	3523	-	"
1	0.003x20-1/8x35-1/4	0.60	3523	-	"
1	0.003x20-1/8x55-1/2	0.90	3523	-	"
SUB-TOTAL	8 pieces	8.50	3523		
1	0.003x23x240	6.00	3695	-	Cold Rolled
SUB-TOTAL	1 piece	6.00	3695		
1	0.0055x18x43	1.30	3437	706	Cold Rolled
1	0.0055x18x43	1.30	3437	707	"
SUB-TOTAL	2 pieces	2.60	3437		
1	0.0055x24-3/4x156	6.40	3783	-	Cold Rolled
1	0.0055xWxL	1.00	3783	-	"
SUB-TOTAL	2 pieces	7.40	3783		
1	0.0055x26-5/8x180	8.70	3792	-	Cold Rolled
1	0.0055x26-3/4x168	8.40	3792	-	"
SUB-TOTAL	2 pieces	17.10	3792		
1	0.0055x24x288	12.00	3796	-	Cold Rolled
SUB-TOTAL	1 piece	12.00	3796		
1	0.0055x25-5/8x216	10.2	3794	-	Cold Rolled
SUB-TOTAL	1 piece	10.2	3794		
1	0.0055x22-1/2x132	4.9	3795	-	Cold Rolled
1	0.0055x23x288	12.0	3795	-	"
SUB-TOTAL	2 pieces	16.9	3795		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.025x59.05x146.05	2.04	3690	-	Alternate Process
1	0.025x59.05x162.88	2.27	3690	-	"
1	0.025x59.05x154.60	2.22	3690	-	"
1	0.025x59.05x161.61	2.22	3690	-	"
SUB-TOTAL	4 pieces	8.75	3690		
1	0.025x58.74x127.00	1.81	3707	-	Alternate Process
SUB-TOTAL	1 piece	1.81	3707		
1	0.025x53.34x356.23	4.40	3497	-	Alternate Process
SUB-TOTAL	1 piece	4.40	3497		
1	0.025x45.08x93.66	.86	3711	-	C.R.A.
SUB-TOTAL	1 piece	.86	3711		
1	0.025x44.77x126.05	1.18	3711	662	C.R.A.
1	0.025x46.99x125.73	1.22	3711	663	"
1	0.025x46.04x124.46	1.22	3711	664	"
1	0.025x47.62x125.09	1.22	3711	665	"
1	0.025x46.67x124.46	1.22	3711	666	"
1	0.025x47.31x124.46	1.22	3711	667	"
1	0.025x34.29x125.73	.91	3711	668	"
1	0.025x45.72x91.44	.91	3711	661	"
SUB-TOTAL	8 pieces	9.12	3711		
1	0.025x61.28x108.90	1.45	3701	669	C.R.A.
1	0.025x59.69x116.20	1.54	3701	670	"
1	0.025x61.59x94.61	1.27	3701	671	"
1	0.025x61.28x89.53	1.22	3701	672	"
1	0.025x61.28x60.32	.86	3701	673	"
1	0.025x60.96x110.49	1.50	3701	674	"
1	0.025x60.32x92.71	1.22	3701	675	"
1	0.025x48.26x112.39	1.13	3701	676	"
SUB-TOTAL	8 pieces	10.21	3701		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.010x23-1/4x57-1/2	4.5	3690	-	Alternate Process
1	0.010x23-1/4x64-1/8	5.0	3690	-	"
1	0.010x23-1/4x60-3/4	4.9	3690	-	"
1	0.010x23-1/4x63-5/8	4.9	3690	-	"
SUB-TOTAL	4 pieces	19.3	3690		
1	0.010x23-1/8x50	4.0	3707	_	Alternate Process
SUB-TOTAL	1 piece	4.0	3707		
1	0.010x21x140-1/4	9.7	3497	-	Alternate Process
SUB-TOTAL	1 piece	9.7	3497		
1	0.010x17-3/4x36-7/8	1.9	3711	-	C.R.A.
SUB-TOTAL	1 piece	1.9	3711		
1	0.010x17-5/8x49-5/8	2.6	3711	662	C.R.A.
1	0.010x18-1/2x49-1/2	2.7	3711	663	"
1	0.010x18-1/8x49	2.7	3711	664	"
1	0.010x18-3/4x49-1/4	2.7	3711	665	"
1	0.010x18-3/8x49	2.7	3711	666	"
1	0.010x18-5/8x49	2.7	3711	667	"
1	0.010x13-1/2x49-1/2	2.0	3711	668	"
1	0.010x18x36	2.0	3711	661	"
SUB-TOTAL	8 pieces	20.1	3711		
1	0.010x24-1/8x42-7/8	3.2	3701	669	C.R.A.
1	0.010x23-1/2x45-3/4	3.4	3701	670	"
1	0.010x24-1/4x37-1/4	2.8	3701	671	"
1	0.010x24-1/8x35-1/4	2.7	3701	672	"
1	0.010x24-1/8x23-3/4	1.9	3701	673	"
1	0.010x24x43-1/2	3.3	3701	674	"
1	0.010x23-3/4x36-1/2	2.7	3701	675	"
1	0.010x19x44-1/4	2.5	3701	676	"
SUB-TOTAL	8 pieces	22.5	3701		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.025x48.26x50.80	.50	3689	643	Ground
1	0.025x52.70x70.48	.73	3689	642	"
1	0.025x57.47x97.79	1.22	3689	641	"
1	0.025x53.97x127.63	1.59	3689	640	"
1	0.025x53.97x124.46	1.54	3689	639	"
1	0.025x57.15x99.06	1.18	3689	638	"
1	0.025x50.48x132.71	1.45	3689	637	"
1	0.025x57.15x99.38	1.22	3689	636	"
1	0.025x57.15x97.47	1.22	3689	635	"
SUB-TOTAL	9 pieces	10.66	3689		
1	0.025x43.18x125.73	1.13	3714	611	Ground
1	0.025x41.27x71.74	.68	3714	612	"
1	0.025x41.27x104.77	.95	3714	613	"
1	0.025x40.00x103.50	.86	3714	614	"
1	0.025x40.00x106.68	.95	3714	615	"
1	0.025x41.91x107.31	1.00	3714	616	"
1	0.025x43.18x107.95	1.04	3714	617	"
1	0.025x39.37x83.82	.64	3714	618	"
1	0.025x41.27x125.41	1.04	3714	619	"
1	0.025x43.81x126.36	1.13	3714	620	"
1	0.025x40.64x122.55	1.00	3714	621	"
1	0.025x43.50x38.42	.36	3714	622	"
1	0.025x43.81x88.58	.86	3714	622A	"
SUB-TOTAL	13 pieces	11.66	3714		
1	0.025x56.20x155.89	1.91	3700	634	C.R.A.
1	0.025x56.51x85.72	1.04	3700	633	"
1	0.025x55.88x106.68	1.32	3700	632	"
1	0.025x54.93x144.78	1.68	3700	631	"
1	0.025x55.24x161.92	1.86	3700	630	"
1	0.025x51.43x143.83	1.59	3700	629	"
SUB-TOTAL	6 pieces	9.39	3700		
1	0.025x50.80x115.57	1.18	3524	591	C.R.A.
1	0.025x50.16x120.01	1.18	3524	588	"
1	0.025x45.72x107.95	.91	3524	587	"
1	0.025x47.62x126.68	1.09	3524	585	"
1	0.025x47.62x113.35	1.00	3524	584	"
SUB-TOTAL	5 pieces	5.35	3524		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.010x19x20	1.1	3689	643	Ground
1	0.010x20-3/4x27-3/4	1.6	3689	642	"
1	0.010x22-5/8x38-1/2	2.7	3689	641	"
1	0.010x21-1/4x50-1/4	3.5	3689	640	"
1	0.010x21-1/4x49	3.4	3689	639	"
1	0.010x22-1/2x39	2.6	3689	638	"
1	0.010x19-7/8x52-1/4	3.2	3689	637	"
1	0.010x22-1/2x39-1/8	2.7	3689	636	"
1	0.010x22-1/2x38-3/8	2.7	3689	635	"
SUB-TOTAL	9 pieces	23.5	3689		
1	0.010x17x49-1/2	2.5	3714	611	Ground
1	0.010x16-1/4x28-1/4	1.5	3714	612	"
1	0.010x16-1/4x41-1/4	2.1	3714	613	"
1	0.010x15-3/4x40-3/4	1.9	3714	614	"
1	0.010x15-3/4x42	2.1	3714	615	"
1	0.010x16-1/2x42-1/4	2.2	3714	616	"
1	0.010x17x42-1/2	2.3	3714	617	"
1	0.010x15-1/2x33	1.4	3714	618	"
1	0.010x16-1/4x49-3/8	2.3	3714	619	"
1	0.010x17-1/4x49-3/4	2.5	3714	620	"
1	0.010x16x48-1/4	2.2	3714	621	"
1	0.010x17-1/8x15-1/8	0.8	3714	622	"
1	0.010x17-1/4x34-7/8	1.9	3714	622A	"
SUB-TOTAL	13 pieces	25.7	3714		
1	0.010x22-1/8x61-3/8	4.2	3700	634	C.R.A.
1	0.010x22-1/4x33-3/4	2.3	3700	633	"
1	0.010x22x42	2.9	3700	632	"
1	0.010x21-5/8x57	3.7	3700	631	"
1	0.010x21-3/4x63-3/4	4.1	3700	630	"
1	0.010x20-1/4x56-5/8	3.5	3700	629	"
SUB-TOTAL	6 pieces	20.7	3700		
1	0.010x20x45-1/2	2.6	3524	591	C.R.A.
1	0.010x19-3/4x47-1/4	2.6	3524	588	"
1	0.010x18x42-1/2	2.0	3524	587	"
1	0.010x18-3/4x49-7/8	2.4	3524	585	"
1	0.010x18-3/4x44-5/8	2.2	3524	584	"
SUB-TOTAL	5 pieces	11.8	3524		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.025x45.72x154.62	1.45	3635	592	C.R.A.
SUB-TOTAL	1 piece	1.45	3635		
1	0.025x45.08x141.92	1.36	3521	593	C.R.A.
1	0.025x46.35x60.96	.54	3521	595	"
1	0.025x57.78x107.95	1.22	3521	595A	"
SUB-TOTAL	3 pieces	3.12	3521		
1	0.025x30.48x109.22	.68	3694	519	Ground
1	0.025x38.10x130.17	.91	3694	518	"
1	0.025x38.73x155.58	1.13	3694	517	"
1	0.025x38.73x153.03	1.18	3694	516	"
1	0.025x38.73x148.91	1.13	3694	515	"
1	0.025x38.42x149.54	1.13	3694	514	"
SUB-TOTAL	6 pieces	6.16	3694		
1	0.025x46.35x121.28	1.18	3525	574	C.R.A.
1	0.025x45.08x144.14	1.41	3525	575	"
1	0.025x46.99x140.33	1.45	3525	576	"
1	0.025x45.40x116.84	1.09	3525	577	"
1	0.025x41.59x142.87	1.22	3525	578	"
1	0.025x46.35x133.35	1.36	3525	579	"
1	0.025x45.08x96.52	.91	3525	580	"
1	0.025x46.35x93.98	.95	3525	581	"
1	0.025x45.08x141.60	1.45	3525	582	"
1	0.025x45.72x137.79	1.45	3525	583	"
SUB-TOTAL	10 pieces	12.47	3525		
1	0.025x48.26x84.14	1.00	3698	608	C.R.A.
1	0.025x41.91x42.54	.45	3698	608A	"
1	0.025x56.51x141.60	1.77	3698	607	"
1	0.025x56.51x140.02	1.72	3698	606	"
1	0.025x56.51x75.25	.86	3698	604	"
1	0.025x56.20x166.69	1.91	3698	603	"
1	0.025x56.51x95.88	1.13	3698	602	"
1	0.025x56.51x80.64	.91	3698	600	"
1	0.025x56.20x97.47	1.04	3698	599	"
1	0.025x56.20x158.75	1.95	3698	598	"
1	0.025x55.88x96.84	1.36	3698	597	"
SUB-TOTAL	11 pieces	14.10	3698		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.010x18x60-7/8	3.2	3635	592	C.R.A.
SUB-TOTAL	1 piece	3.2	3635		
1	0.010x17-3/4x55-7/8	3.0	3521	593	C.R.A.
1	0.010x18-1/4x24	1.2	3521	595	"
1	0.010x22-3/4x42-1/2	2.7	3521	595A	"
SUB-TOTAL	3 pieces	6.9	3521		
1	0.010x12x43	1.5	3694	519	Ground
1	0.010x15x51-1/4	2.0	3694	518	"
1	0.010x15-1/4x61-1/4	2.5	3694	517	"
1	0.010x15-1/4x60-1/4	2.6	3694	516	"
1	0.010x15-1/4x58-5/8	2.5	3694	515	"
1	0.010x15-1/8x58-7/8	2.5	3694	514	"
SUB-TOTAL	6 pieces	13.6	3694		
1	0.010x18-1/4x47-3/4	2.6	3525	574	C.R.A.
1	0.010x17-3/4x56-3/4	3.1	3525	575	"
1	0.010x18-1/2x55-1/4	3.2	3525	576	"
1	0.010x17-7/8x46	2.4	3525	577	"
1	0.010x16-3/8x56-1/4	2.7	3525	578	"
1	0.010x18-1/4x52-1/2	3.0	3525	579	"
1	0.010x17-3/4x38	2.0	3525	580	"
1	0.010x18-1/4x37	2.1	3525	581	"
1	0.010x17-3/4x55-3/4	3.2	3525	582	"
1	0.010x18x54-1/4	3.2	3525	583	"
SUB-TOTAL	10 pieces	27.5	3525		
1	0.010x19x33-1/8	2.2	3698	608	C.R.A.
1	0.010x16-1/2x16-3/4	1.0	3698	608A	"
1	0.010x22-1/4x55-3/4	3.9	3698	607	"
1	0.010x22-1/4x55-1/8	3.8	3698	606	"
1	0.010x22-1/4x29-5/8	1.9	3698	604	"
1	0.010x22-1/8x65-5/8	4.2	3698	603	"
1	0.010x22-1/4x37-3/4	2.5	3698	602	"
1	0.010x22-1/4x31-3/4	2.0	3698	600	"
1	0.010x22-1/8x38-3/8	2.3	3698	599	"
1	0.010x22-1/8x62-1/2	4.3	3698	598	"
1	0.010x22x38-1/8	3.0	3698	597	"
SUB-TOTAL	11 pieces	31.1	3698		

B

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.025x52.70x120.97	1.22	3691	652	C.R.A.
1	0.025x51.43x127.95	1.27	3691	653	"
1	0.025x52.70x142.87	1.68	3691	654	"
1	0.025x53.34x146.68	1.45	3691	655	"
1	0.025x51.43x148.59	1.50	3691	656	"
1	0.025x53.02x122.87	1.22	3691	657	"
1	0.025x33.65x121.28	.82	3691	658	"
1	0.025x51.43x120.01	1.32	3691	659	"
1	0.025x53.34x125.73	1.45	3691	660	"
SUB-TOTAL	9 pieces	11.93	3691		
1	0.025x60.01x153.99	1.91	3697	628	C.R.A.
1	0.025x56.69x152.40	2.00	3697	626	"
1	0.025x60.32x153.03	2.09	3697	625	"
1	0.025x60.01x159.07	2.09	3697	624	"
1	0.025x60.01x161.29	2.09	3697	623	"
SUB-TOTAL	5 pieces	10.18			
1	0.025x46.04x111.44	1.09	3637	557	C.R.A.
1	0.025x46.35x97.15	.95	3637	558	"
1	0.025x45.72x95.25	.95	3637	559	"
1	0.025x42.54x109.85	1.00	3637	560	"
1	0.025x46.35x113.66	1.09	3637	561	"
1	0.025x42.54x109.54	1.00	3637	562	"
1	0.025x44.08x102.87	1.04	3637	458	"
1	0.025x43.18x95.25	.91	3637	457	"
1	0.025x43.18x100.97	.95	3637	563	"
1	0.025x41.27x90.80	.73	3637	564	"
1	0.025x42.54x68.58	.59	3637	565	"
1	0.025x42.23x111.76	.86	3637	566	"
1	0.025x45.72x91.44	.86	3637	567	"
1	0.025x42.54x114.62	.95	3637	568	"
1	0.025x42.54x94.93	.82	3637	569	"
1	0.025x44.45x109.22	1.04	3637	570	"
SUB-TOTAL	16 pieces	14.83	3637		
1	0.025x46.04x134.62	1.32	3875	730	C.R.A.
1	0.025x46.04x113.67	1.36	3875	731	"
1	0.025x45.72x169.54	1.45	3875	732	"
1	0.025x45.72x173.04	1.68	3875	733	"
1	0.025x45.72x140.02	1.45	3875	734	"
1	0.025x45.72x173.35	1.77	3875	735	"
SUB-TOTAL	6 pieces	9.03	3875		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.010x20-3/4x47-5/8	2.7	3691	652	C.R.A.
1	0.010x20-1/4x50-3/8	2.8	3691	653	"
1	0.010x20-3/4x56-1/4	3.7	3691	654	"
1	0.010x21x57-3/4	3.2	3691	655	"
1	0.010x20-1/4x58-1/2	3.3	3691	656	"
1	0.010x20-7/8x48-3/8	2.7	3691	657	"
1	0.010x13-1/4x47-3/4	1.8	3691	658	"
1	0.010x20-1/4x47-1/4	2.9	3691	659	"
1	0.010x21x49-1/2	3.2	3691	660	"
SUB-TOTAL	9 pieces	26.3	3691		
1	0.010x23-5/8x60-5/8	4.2	3697	628	C.R.A.
1	0.010x23-1/2x60	4.4	3697	626	"
1	0.010x23-3/4x60-1/4	4.6	3697	625	"
1	0.010x23-5/8x62-5/8	4.6	3697	624	"
1	0.010x23-5/8x63-1/2	4.6	3697	623	"
SUB-TOTAL	5 pieces	22.4	3697		
1	0.010x18-1/8x43-7/8	2.4	3637	557	C.R.A.
1	0.010x18-1/4x38-1/4	2.1	3637	558	"
1	0.010x18x37-1/2	2.1	3637	559	"
1	0.010x16-3/4x43-1/4	2.2	3637	560	"
1	0.010x18-1/4x44-3/4	2.4	3637	561	"
1	0.010x16-3/4x43-1/8	2.2	3637	562	"
1	0.010x17-3/4x40-1/2	2.3	3637	458	"
1	0.010x17x37-1/2	2.0	3637	457	"
1	0.010x17x39-3/4	2.1	3637	563	"
1	0.010x16-1/4x35-3/4	1.6	3637	564	"
1	0.010x16-3/4x27	1.3	3637	565	"
1	0.010x16-5/8x44	1.9	3637	566	"
1	0.010x18x36	1.9	3637	567	"
1	0.010x16-3/4x45-1/8	2.1	3637	568	"
1	0.010x16-3/4x37-3/8	1.8	3637	569	"
1	0.010x17-1/2x43	2.3	3637	570	"
SUB-TOTAL	16 pieces	32.7	3637		
1	0.010x18-1/8x53	2.9	3875	730	C.R.A.
1	0.010x18-1/8x52-5/8	3.0	3875	731	"
1	0.010x18x66-3/4	3.2	3875	732	"
1	0.010x18x68-1/8	3.7	3875	733	"
1	0.010x18x55-5/8	3.2	3875	734	"
1	0.010x18x68-1/4	3.9	3875	735	"
SUB-TOTAL	6 pieces	19.9	3875		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.025x46.99x119.38	1.04	3873	753	C.R.A.
1	0.025x46.99x93.98	.82	3873	754	"
1	0.025x46.99x85.09	.77	3873	755	"
1	0.025x46.99x120.97	1.09	3873	756	"
1	0.025x46.99x93.03	.86	3873	757	"
1	0.025x46.99x98.42	.95	3873	758	"
1	0.025x46.99x125.73	1.22	3873	759	"
SUB-TOTAL	7 pieces	6.75	3873		
1	0.025x66.36x136.52	2.04	3918	748	C.R.A.
1	0.025x66.67x127.95	1.77	3918	750	"
1	0.025x66.67x136.21	1.91	3918	751	"
1	0.025x66.67x135.25	1.86	3918	752	"
SUB-TOTAL	4 pieces	7.58	3918		
1	0.025x55.88x157.80	1.68	3919	747	C.R.A.
1	0.025x55.88x151.45	1.68	3919	748	"
SUB-TOTAL	2 pieces	3.36	3919		
1	0.025x59.69x108.90	1.32	3917	716	C.R.A.
1	0.025x56.83x128.27	1.50	3917	717	"
1	0.025x52.39x127.00	1.32	3917	718	"
1	0.025x52.39x127.32	1.22	3917	719	"
1	0.025x52.07x110.49	1.22	3917	720	"
1	0.025x52.07x129.86	1.32	3917	721	"
1	0.025x57.15x127.32	1.63	3917	722	"
1	0.025x59.69x105.41	1.32	3917	723	"
1	0.025x57.47x125.09	1.59	3917	724	"
1	0.025x60.01x106.04	1.36	3917	725	"
1	0.025x52.07x127.00	1.27	3917	726	"
1	0.025x54.93x129.54	1.59	3917	727	"
1	0.025x62.86x102.87	1.27	3917	728	"
1	0.025x59.69x109.22	1.32	3917	729	"
SUB-TOTAL	14 pieces	19.25	3917		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.010x18-1/2x47	2.3	3873	753	C.R.A.
1	0.010x18-1/2x37	1.8	3873	754	"
1	0.010x18-1/2x33-1/2	1.7	3873	755	"
1	0.010x18-1/2x47-5/8	2.4	3873	756	"
1	0.010x18-1/2x36-5/8	1.9	3873	757	"
1	0.010x18-1/2x38-3/4	2.1	3873	758	"
1	0.010x18-1/2x49-1/2	2.7	3873	759	"
SUB-TOTAL	7 pieces	14.9	3873		
1	0.010x26-1/8x53-3/4	4.5	3918	749	C.R.A.
1	0.010x26-1/4x50-3/8	3.9	3918	750	"
1	0.010x26-1/4x53-5/8	4.2	3918	751	"
1	0.010x26-1/4x53-1/4	4.1	3918	752	"
SUB-TOTAL	4 pieces	16.7	3918		
1	0.010x22x62-1/8	3.7	3919	747	C.R.A.
1	0.010x22x59-5/8	3.7	3919	748	"
SUB-TOTAL	2 pieces	7.4	3919		
1	0.010x23-1/2x42-7/8	2.9	3917	716	C.R.A.
1	0.010x22-3/8x50-1/2	3.3	3917	717	"
1	0.010x20-5/8x50	2.9	3917	718	"
1	0.010x20-5/8x50-1/8	2.7	3917	719	"
1	0.010x20-1/2x43-1/2	2.7	3917	720	"
1	0.010x20-1/2x51-1/8	2.9	3917	721	"
1	0.010x22-1/2x50-1/8	3.6	3917	722	"
1	0.010x23-1/2x41-1/2	2.9	3917	723	"
1	0.010x22-5/8x49-1/4	3.5	3917	724	"
1	0.010x23-5/8x41-3/4	3.0	3917	725	"
1	0.010x20-1/2x50	2.8	3917	726	"
1	0.010x21-5/8x51	3.5	3917	727	"
1	0.010x24-3/4x40-1/2	2.8	3917	728	"
1	0.010x23-1/2x43	2.9	3917	729	"
SUB-TOTAL	14 pieces	42.4	3917		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.038x27.62x88.58	.86	3716	688	Ground
1	0.038x36.19x78.74	.91	3716	687	"
1	0.038x41.91x79.69	1.09	3716	686	"
1	0.038x36.83x82.23	1.00	3716	685	"
1	0.038x36.83x81.28	1.00	3716	684	"
1	0.038x40.64x77.79	1.00	3716	682	"
1	0.038x36.83x79.99	1.00	3716	681	"
1	0.038x40.00x81.91	1.13	3716	680	"
1	0.038x39.05x86.36	1.09	3716	679	"
1	0.038x44.45x82.55	1.22	3716	677	"
SUB-TOTAL	10 pieces	10.30	3716		
1	0.051x59.69x106.68	2.81	3876	708	Ground
1	0.051x57.15x116.20	2.77	3876	709	"
1	0.051x57.15x123.19	3.04	3876	710	"
1	0.051x59.05x116.84	3.04	3876	711	"
1	0.051x57.78x122.55	2.95	3876	712	"
1	0.051x59.69x104.14	2.63	3876	713	"
1	0.051x58.42x104.14	2.63	3876	714	"
1	0.051x58.42x86.36	2.18	3876	715	"
SUB-TOTAL	8 pieces	22.05	3876		
1	0.051x43.81x107.63	1.95	3712	644	Ground
1	0.051x45.72x109.22	2.18	3712	645	"
1	0.051x47.62x112.08	2.36	3712	646	"
1	0.051x45.08x109.22	2.22	3712	647	"
1	0.051x45.72x108.58	2.27	3712	648	"
1	0.051x46.99x109.22	2.18	3712	649	"
1	0.051x46.35x107.95	2.13	3712	650	"
1	0.051x46.35x112.08	2.27	3712	651	"
SUB-TOTAL	8 pieces	17.56	3712		
1	0.051x64.45x98.42	2.86	3706	559	Ground
1	0.051x64.13x93.03	2.72	3706	555	"
1	0.051x63.50x88.58	2.49	3706	556	"
1	0.051x33.65x31.43	.50	3706	548	"
1	0.051x34.61x38.10	.59	3706	549	"
1	0.051x34.61x36.51	.59	3706	550	"
1	0.051x26.35x37.46	.45	3706	551	"
1	0.051x26.99x32.07	.36	3706	552	"
1	0.051x25.72x34.61	.36	3706	553	"
SUB-TOTAL	9 pieces	10.92	3706		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.015x10-7/8x34-7/8	1.9	3716	688	Ground
1	0.015x14-1/4x31	2.0	3716	687	"
1	0.015x16-1/2x31-3/8	2.4	3716	686	"
1	0.015x14-1/2x32-3/8	2.2	3716	685	"
1	0.015x14-1/2x32	2.2	3716	684	"
1	0.015x16x30-5/8	2.2	3716	682	"
1	0.015x14-1/2x31-1/2	2.2	3716	681	"
1	0.015x15-3/4x32-1/4	2.5	3716	680	"
1	0.015x15-3/8x34	2.4	3716	679	"
1	0.015x17-1/2x32-1/2	2.7	3716	677	"
SUB-TOTAL	10 pieces	22.7	3716		
1	0.020x23-1/2x42	6.2	3876	708	Ground
1	0.020x22-1/2x45-3/4	6.1	3876	709	"
1	0.020x22-1/2x48-1/2	6.7	3876	710	"
1	0.020x23-1/4x46	6.7	3876	711	"
1	0.020x22-3/4x48-1/4	6.5	3876	712	"
1	0.020x23-1/2x41	5.8	3876	713	"
1	0.020x23x41	5.8	3876	714	"
1	0.020x23x34	4.8	3876	715	"
SUB-TOTAL	8 pieces	48.6	3876		
1	0.020x17-1/4x42-3/8	4.3	3712	644	Ground
1	0.020x18x43	4.8	3712	645	"
1	0.020x18-3/4x44-1/8	5.2	3712	646	"
1	0.020x17-3/4x43	4.9	3712	647	"
1	0.020x18x42-3/4	5.0	3712	648	"
1	0.020x18-1/2x43	4.8	3712	649	"
1	0.020x18-1/4x42-1/2	4.7	3712	650	"
1	0.020x18-1/4x44-1/8	5.0	3712	651	"
SUB-TOTAL	8 pieces	38.7	3712		
1	0.020x25-3/8x38-3/4	6.3	3706	559	Ground
1	0.020x25-1/4x36-5/8	6.0	3706	555	"
1	0.020x25x34-7/8	5.5	3706	556	"
1	0.020x13-1/4x12-3/8	1.1	3706	548	"
1	0.020x13-5/8x15	1.3	3706	549	"
1	0.020x13-5/8x14-3/8	1.3	3706	550	"
1	0.020x10-3/8x14-3/4	1.0	3706	551	"
1	0.020x10-5/8x12-5/8	0.8	3706	552	"
1	0.020x10-1/8x13-5/8	0.8	3706	553	"
SUB-TOTAL	9 pieces	24.1	3706		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.051x44.45x186.69	3.63	3709	540	Ground
1	0.051x45.08x194.94	3.90	3709	541	"
1	0.051x44.77x192.09	3.58	3709	542	"
1	0.051x44.77x193.04	3.81	3709	543	"
SUB-TOTAL	4 pieces (UnRx)	14.92	3709		
1	0.076x64.45x93.34	3.99	3796	704	Ground
1	0.076x64.45x74.93	3.36	3796	705	"
SUB-TOTAL	2 pieces	7.35	3796		
1	0.102x67.94x134.94	7.98	3918	736	Ground
1	0.102x51.43x92.71	4.31	3918	738	"
SUB-TOTAL	2 pieces	12.29	3918		
1	0.102x63.50x152.40	8.62	3874	739	Ground
1	0.102x64.77x139.06	7.94	3874	740	"
SUB-TOTAL	2 pieces	16.56	3874		
1	0.102x64.77x173.99	9.75	3919	741	Ground
1	0.102x58.74x93.98	4.90	3919	742	"
SUB-TOTAL	2 pieces	14.65	3919		
1	0.102x63.82x89.22	5.31	3920	743	Ground
1	0.102x60.96x71.44	3.95	3920	744	"
SUB-TOTAL	2 pieces	9.26	3920		
1	0.102x55.24x102.87	4.81	3708	547	Ground
1	0.102x62.25x97.79	5.44	3708	544	"
SUB-TOTAL	2 pieces	10.25	3708		
1	0.102x48.26x124.78	5.22	3715	571	Ground
1	0.102x52.07x126.68	5.76	3715	572	"
1	0.102x49.53x129.54	5.58	3715	573	"
SUB-TOTAL	3 pieces	16.56	3715		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.020x17-1/2x73-1/2	8.0	3709	540	Ground
1	0.020x17-3/4x76-3/4	8.6	3709	541	"
1	0.020x17-5/8x75-5/8	7.9	3709	542	"
1	0.020x17-5/8x76	8.4	3709	543	"
SUB-TOTAL	4 pieces (UnRx)	32.9	3709		
1	0.030x25-3/8x36-3/4	8.8	3796	704	Ground
1	0.030x25-3/8x29-1/2	7.4	3796	705	"
SUB-TOTAL	2 pieces	16.2	3796		
1	0.040x26-3/4x53-1/8	17.6	3918	736	Ground
1	0.040x20-1/4x36-1/2	9.5	3918	738	"
SUB-TOTAL	2 pieces	27.1	3918		
1	0.040x25x60	19.0	3874	739	Ground
1	0.040x25-1/2x54-3/4	17.5	3874	740	"
SUB-TOTAL	2 pieces	36.5	3874		
1	0.040x25-1/2x68-1/2	21.5	3919	741	Ground
1	0.040x23-1/8x37	10.8	3919	742	"
SUB-TOTAL	2 pieces	32.3	3919		
1	0.040x25-1/8x35-1/8	11.7	3920	743	Ground
1	0.040x24x28-1/8	8.7	3920	744	"
SUB-TOTAL	2 pieces	20.4	3920		
1	0.040x21-3/4x40-1/2	10.6	3708	547	Ground
1	0.040x24-1/2x38-1/2	12.0	3708	544	"
SUB-TOTAL	2 pieces	22.6	3708		
1	0.040x19x49-1/8	11.5	3715	571	Ground
1	0.040x20-1/2x49-7/8	12.7	3715	572	"
1	0.040x19-1/2x51	12.3	3715	573	"
SUB-TOTAL	3 pieces	36.5	3715		

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(cm.)	Wt. Kgs.	Heat No.	Serial No.	Finish
1	0.203x22.86x45.72	1.81	3692	690	Ground
SUB-TOTAL	1 piece	1.81	3692		
1	0.025x72.07x108.58	18.14	3829	703	Ground
SUB-TOTAL	1 piece (UnRx)	18.14	3829		
1	0.064x26.99x69.85	11.29	3699	496	Ground
SUB-TOTAL	1 piece	11.29	3699		
1	0.064x45.72x26.67	7.30	3827	701	Ground
1	0.064x32.38x33.65	6.89	3827	702	"
SUB-TOTAL	2 pieces (UnRx)	14.19	3827		
GRAND TOTAL	256 pieces	448.81			

TABLE D-1 (CONT'D.)

TDNiCr SHEET PRODUCT

Quantity Pcs.	Size(in.)	Wt. Lbs.	Heat No.	Serial No.	Finish
1	0.80x9x18	4.0	3692	690	Ground
SUB-TOTAL	1 piece	4.0	3692		
1	0.10x28-3/8x42-3/4	40.0	3829	703	Ground
SUB-TOTAL	1 piece (UnRx)	40.0	3829		
1	0.25x10-5/8x27-1/2	24.9	3699	496	Ground
SUB-TOTAL	1 piece	24.9	3699		
1	0.25x18x10-1/2	16.1	3827	701	Ground
1	0.25x12-3/4x13-1/4	15.2	3827	702	"
SUB-TOTAL	2 pieces (UnRx)	31.3	3827		
GRAND TOTAL	256 pieces	989.4			

APPENDIX E

TDNiCrAl Exploratory Studies

1. Alloy Studies

A number of techniques were explored for manufacturing the powder of the desired composition for the TDNiCrAl alloys.

All of the aluminum containing small scale studies reported in Part I utilized master alloy powders manufactured by Cerac Inc. of Cr-Al or Cr-Al-Y. They were prepared by induction melting in carbon-lined crucibles. The master alloys were melted in small charges and ground under argon in ball mills to provide master alloy powders of -325 mesh.

No beneficial effects could be accredited to the presence of Y in the master alloys, consequently the scale-up studies in Part II were restricted to master alloys of Cr-Al.

Attempts to scale up the melting of alloys of Cr-Al were not successful.

Two other approaches were successful in producing master alloy powders of Cr-Al in the range of 15 to 25% Al.

One technique utilized high purity Shieldalloy flake chromium powder which was wet ground in a 15S Union Process Company attritor with pure aluminum powder. The ground powders were then heated under argon in a push-pull furnace starting at 232°C(450°F) and increasing to 510°C(950°F) and holding for one hour. The resulting powders were alloyed, of good purity and remained as fine powders.

A proprietary thermite type process developed by Reading Alloys Inc. produced Cr-Al master alloys which were reproducible, of good purity, and of relatively low cost. The powders were ground by Reading Alloys, Inc. to -100 mesh. All of the TDNiCrAl data reported in the Part II study were obtained on billets made with powder from Cr-Al master alloys provided by Reading Alloys, Inc.

The Reading Cr-Al master alloys were ground in a 15S Union Process Company attritor for 8 hours using 1.75 cm.(3/16 in.) diameter high-chromium steel balls. The powders were immersed in an organic solvent "Soltrol" manufactured by Phillips Petroleum and covered with a blanket of argon in the attritor. The master alloy was ground to approximately 3μ diameter in the 8 hour period and was then air dried.

The TDNiCrAl powders were prepared by pre-blending (1/2 hour) the appropriate amounts of Ni-ThO_2 powder prepared by the Du Pont-Fansteel coprecipitation technique together with the ground Cr-Al master alloy powder in a cone blender. The blended alloy was then ground in 45.4 kg.(100 lb.) batches in a 15S Union Process Company attritor. The powders were attritored under "Soltrol" and argon for three hours. The powders were then screened through a 60 mesh screen and air dried preparatory for compaction.

2. TDNiCrAl Scale-Up Studies

During Part II a series of 45.4 kg.(100 lb.) scale-up TDNiCrAl billets was fabricated and evaluated. Nominal compositions and heat numbers of these units were as follows:

Heat No.	Nominal Composition
3806	$Ni-16Cr-2Al-2.0ThO_2$
3807	$Ni-16Cr-3Al-2.0ThO_2$
3808	$Ni-16Cr-3Al-2.0ThO_2$
3809	$Ni-16Cr-3Al-2.0ThO_2$
3810	$Ni-16Cr-3Al-2.0ThO_2$
3811	$Ni-20Cr-2Al-2.0ThO_2$
3812	$Ni-20Cr-3Al-2.0ThO_2$
3816	$Ni-16Cr-3Al-2.0ThO_2$
3831	$Ni-16Cr-4Al-2.0ThO_2$
3848	$Ni-16Cr-3Al-1.5ThO_2$
3849	$Ni-16Cr-4Al-1.5ThO_2$

All of these heats were compacted by conventional techniques, canned in "loose type" mild steel containers and sintered at 1067° C(1950° F), recanned in standard roll consolidation cans and rolled to the 2.54 cm.(1.0 in.) slab stage of processing. Subsequent fabrication studies were described in Section II-3a of this report.

a. Carbide Studies

Chromium carbides were suspected of contributing to fabrication problems in the forming of TDNiCrAl alloy sheet. Solution treating followed by rapid cool have shown some improvement in bend behavior of some of the TDNiCrAl alloys.

A scouting study was initiated to explore the potential of forming other stable carbides which might be beneficial rather than detrimental.

C

Three small units of Ni-16Cr-5Al-2ThO$_2$ alloys containing 0.5% additions of Mo, Ta or HF were sintered at 1035° C(1900° F) and then forged to density at 1090° C(2000° F). Sections of the forgings were then heat treated at a series of temperatures and times. Subsequent to each heat treatment, carbides and oxides were extracted with a 1:1:1 solution of H_2O, HCl and HNO_3. X-ray diffraction patterns were obtained for the purpose of determining the nature of the carbides which had been precipitated as a result of each heat treatment. Table E-1 lists the results obtained.

From the data it is seen that Cr carbides are those which are stable in the Ni-Cr-Al-ThO$_2$ matrix. The preponderance of ThO$_2$ and Al$_2$O$_3$ in the extracts made it difficult to differentiate between Cr_7C_3 and $Cr_{23}C_6$. This was done by comparing the peak height intensity of the (422) line of $Cr_{23}C_6$ with those of the (411), (202) and (102) lines of Cr_7C_3.

The alloy additions did not appear effective and no further carbide studies were made.

3. TDNiCr Oxidation Evaluation

When TDNiCr is exposed to air at elevated temperatures the oxidation products and kinetics are chiefly a function of temperature, time, gas velocity, angle of impingement, stress, total dynamic pressure and substrate grain size and orientation. Since it is possible that standard TDNiCr may not survive the environmental conditions anticipated for the

TABLE E-1.

CARBIDES FORMED IN SELECTED Ni-Cr-Al-ThO_2 ALLOYS

Alloy Composition	Heat Treatment	Carbides Present
Ni-16Cr-5Al- $2ThO_2$-0.5Ta	As Forged	TaC
	+ 1 Hr.1260° C(2300° F)-F.C.	$Cr_{23}C_6 + Cr_7C_3$
	+ 4 Hrs.1038° C(1900° F)-F.C.	$Cr_7C_3 + Cr_{23}C_6$
	+ 4 Hrs. 927° C(1700° F)-A.C.	$Cr_7C_3 + Cr_{23}C_6$
Ni-16Cr-5Al- $2ThO_2$-0.5Mo	As Forged	$Cr_7C_3 + Cr_{23}C_6$
	+ 1 Hr.1260° C(2300° F)-F.C.	$Cr_{23}C_6 + Cr_7C_3$
	+ 4 Hrs.1038° C(1900° F)-F.C.	$Cr_{23}C_6 + Cr_7C_3$
	+ 4 Hrs. 927° C(1700° F)-A.C.	$Cr_{23}C_6 + Cr_7C_3$
Ni-16Cr-5Al $2ThO_2$-0.5Hf	As Forged	Cr_7C_3 + HfC
	+ 1 Hr. 1260° C(2300° F)-F.C.	$Cr_7C_3 + Cr_{23}C_6$
	+ 4 Hrs.1038° C(1900° F)-F.C.	$Cr_7C_3 + Cr_{23}C_6$
	+ 4 Hrs. 927° C(1700° F)-A.C.	Cr_7C_3

space shuttle, this portion of the program was aimed at modifying the base composition to further improve its oxidation resistance without seriously impairing other requisite properties.

At the program's inception it was generally regarded that the major detriment to the performance of TDNiCr was the conversion of the protective oxide, Cr_2O_3, to CrO_3 gas. Under space shuttle conditions some early estimates of this reaction rate precluded the use of TDNiCr at a temperature of 1204° C (2200° F). This served as the basis for the selection of the alloy systems to be evaluated.

One approach taken was to alloy the TDNiCr with elements which tend to form complex oxides or spinels with Cr_2O_3. Of major interest here were additions of Mn and Fe which showed promise in some earlier work. Si had been found to be of further benefit when used in conjunction with these additives owing to the formation of a sub-scale of SiO_2. In addition, La had also been found to stabilize Mn Cr_2O_4 in alloys such as Rene Y and was included in the study.

The second and major approach was that of utilizing a minimum of 3 percent Al in the nickel-chromium alloy matrix. In this manner, it was expected that Cr_2O_3 formation would be greatly suppressed in favor of the non-volatile Al_2O_3 and $NiAl_2O_4$ species.

A dynamic oxidation test rig was assembled at Fansteel Baltimore for testing the alloys of interest. A test specimen consisted of a flat coupon, 1.59 cm x 2.54 cm. x 0.038 cm. (5/8 in. x 1 in. x 0.015 in.) fitted into slots in a metallic specimen holder. Initially four and later eight such specimens and holders were placed in 5.1 cm.(2 in.) diameter tubes in a furnace at 1204°C(2200°F) and exposed to a 914.4-152.4 cm./sec. (30-50 ft./sec.) stream of combustion products from gas burners located at one end of the tubes. Each test, of a nominal 40-50 hour duration with cooling cycles every half hour to below 537°C(1000°F), utilized one or two standard TDNiCr specimens (Heat 3304) and three or six test alloy specimens. Weight change data were recorded, initially at one hour intervals and later at approximately 8 hour intervals. Each four hours specimens were rotated from position to position to eliminate any such effect on results. After testing, the specimens were subjected to metallographic examination. In addition, the surfaces were analyzed by X-ray diffraction and electron microprobe analysis where warranted.

As a result of the first year's effort, it was determined that Al modified Ni-Cr-ThO_2 alloys afforded the most promise for further development. One major problem associated with these alloys, that of subsurface oxidation, was overcome through the use of a H_2 preheat treatment prior to oxidation testing. The preformed Al_2O_3 film remained intact for at least 50 hours of testing at all temperatures from 870°C (1600°F) to 1204°C(2200°F). Another problem, common to all the materials tested during the first year, was that of apparent porosity which developed within the test specimens; the degree of porosity showed a heat to heat as well as a compositional dependence. A factor which obscured the picture was that the porosity, unless extremely severe, could be revealed only by electropolishing or etching. No porosity was generally observed by vibrator polishing.

Three approaches to pinning down this problem were taken as follows:

1. Specimens from Heats 3848 ($Ni-16Cr-3.5Al-1.5ThO_2$) and 3849 ($Ni-16Cr-4.0Al-1.5ThO_2$) were subjected to dynamic oxidation testing at 1204° C and 898° C(2200° F and 1650° F) and sampled every 1, 4, 16, and 64 hours. These specimens were examined metallographically and subjected to electron microprobe analyses.
2. During the course of the above test, specimens from Heat 3336 ($Ni-16Cr-3.5Al-2ThO_2$) which had previously been given varying types of heat treatments, were examined by scanning electron microscopy. These heat treatments included as-received material (2 hours at 1315° C(2400° F) in A_2), 48 hours at 1204° C(2200° F) in H_2 and 100 hours at 1260° C(2300° F) in air.
3. Towards the end of the program specimens of widely varying grain sizes were made available. The specimens were of a nominal $Ni-16Cr-3.5Al-2ThO_2$ composition from Heats 3903 and 3915. These specimens were tested in the dynamic oxidation rig for 45 hours at 1204° C (2200° F) and subjected to metallographic analyses.

4. The final group of specimens available for oxidation testing were from Heats 3925, 3927, 3928 and 3929 of a nominal Ni-16Cr-5Al-2ThO_2 composition. Tests as in (3) were performed.

The above tests should have revealed the effects, if any, of time, temperature, environment, Al content and grain size on porosity.

From the tests on Heats 3848 and 3849 it was found that no porosity developed in specimens oxidized for times to 64 hours at 870° C(1600° F). Those specimens tested at 1204° C (2200° F), however, showed that porosity increases with time as can be seen in Figures E-1 and E-2. Specimens of Heats 3925, 3927, 3928 and 3929, heated for 45 hours at 1204° C (2200° F), are shown in Figures E-3 and E-4. These show the presence of some porosity. The same specimen oxidized for 100 hours at 1260° C(2300° F) showed more porosity. From these facts, it can be postulated that Kirkendall porosity in TDNiCrAl alloys develops as a result of the outward diffusion of Al. As a result, vacancies are generated eventually forming visible pores. These results are in agreement with those of Whittenberger* in his studies with various metallic diffusion couples.

Scanning electron microscopy also showed that within the pores were varying amounts of Al_2O_3, ThO_2 and Fe rich

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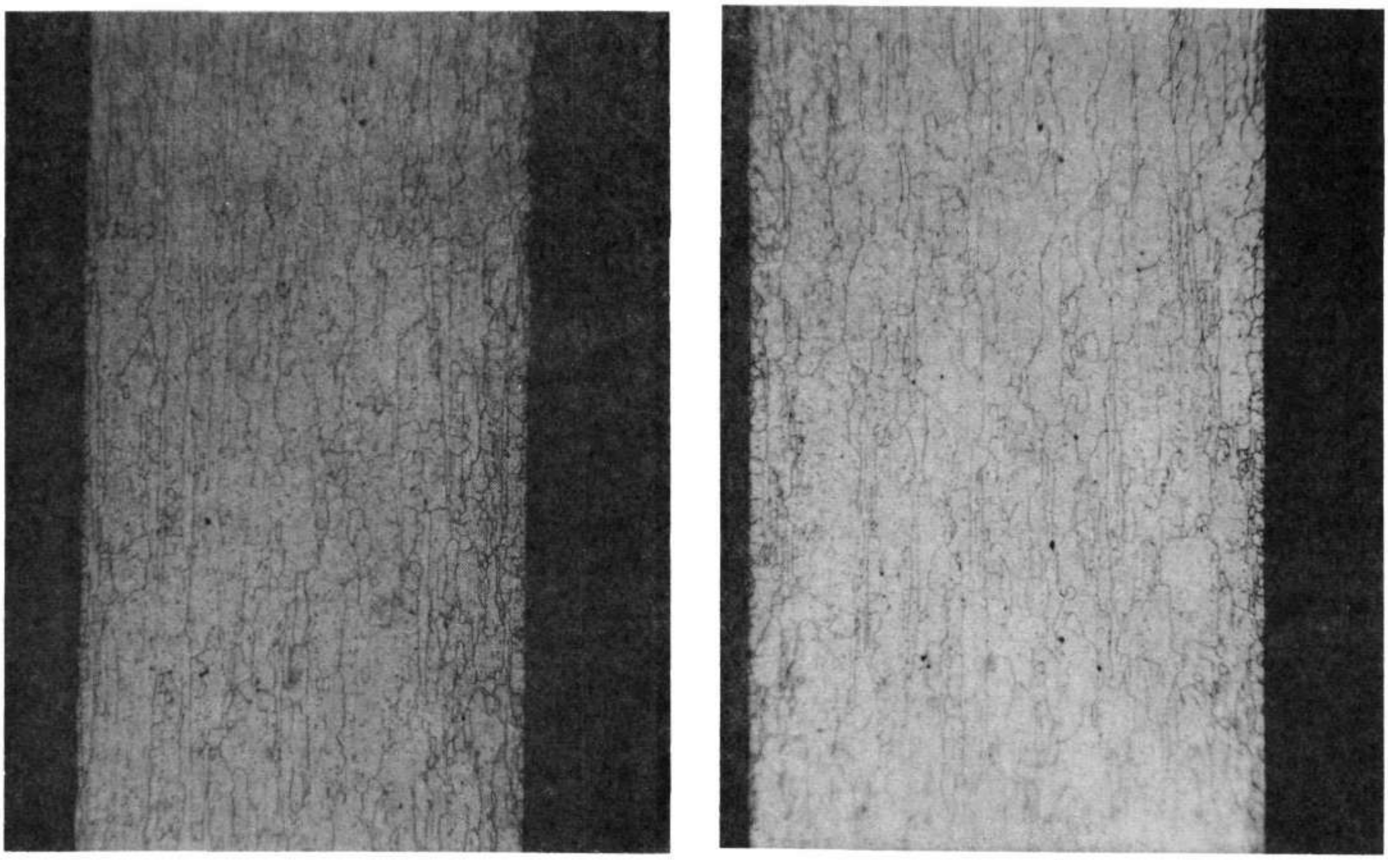
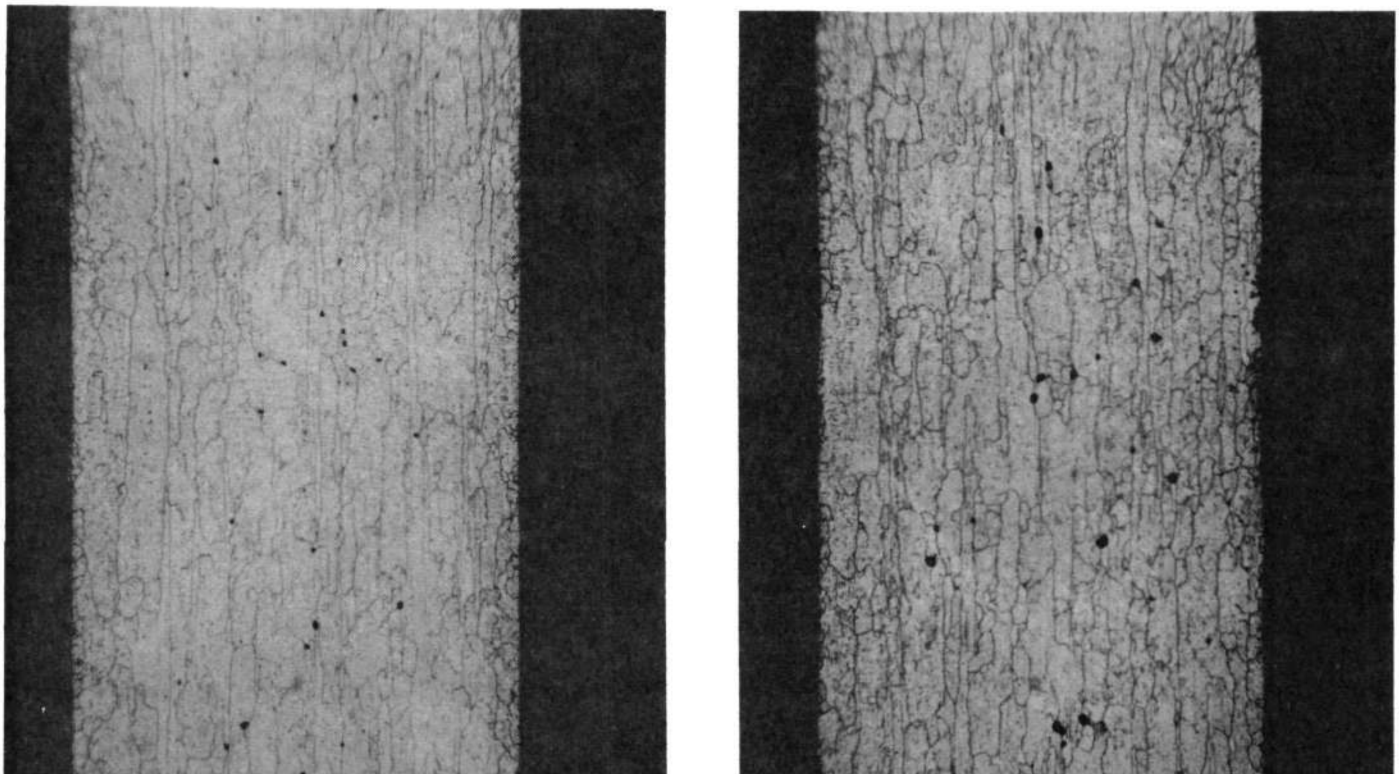
*Whittenberger, J. D., NASA TN D-6797, Lewis Research Center

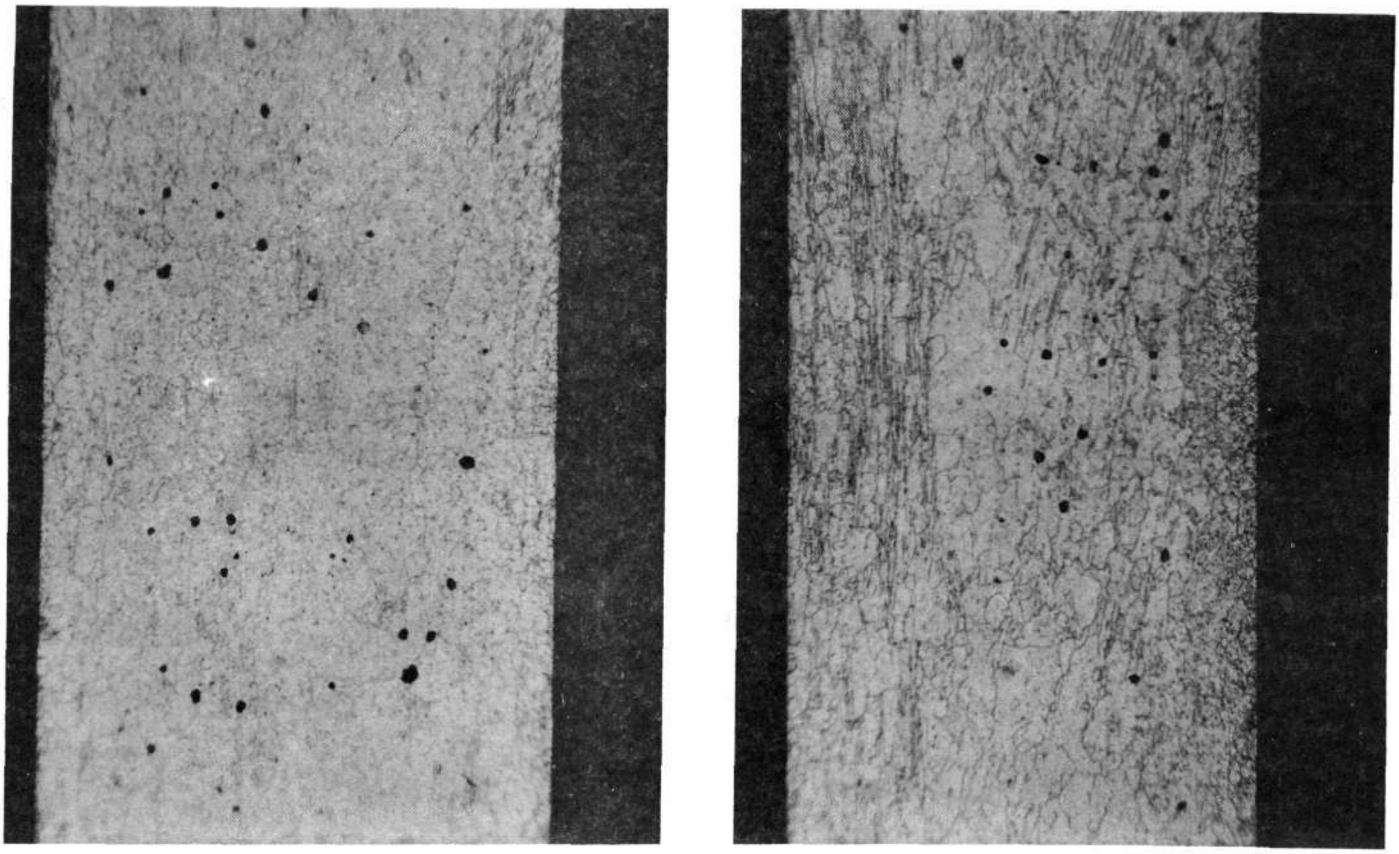


1 Hour 4 Hours



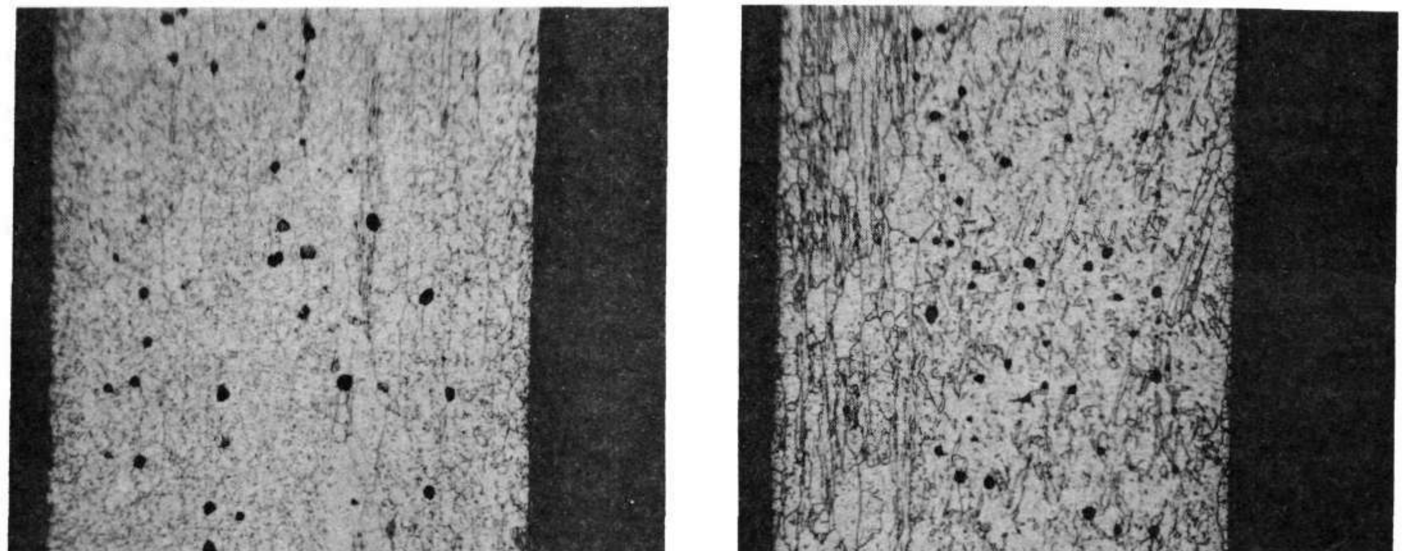
16 Hours 64 Hours

FIGURE E-1. CYCLIC OXIDATION TESTS AT 1204°C(2200°F)
0.038 cm.(0.015 in.) SHEET, HEAT 3848, MAG. 200X



1 Hour

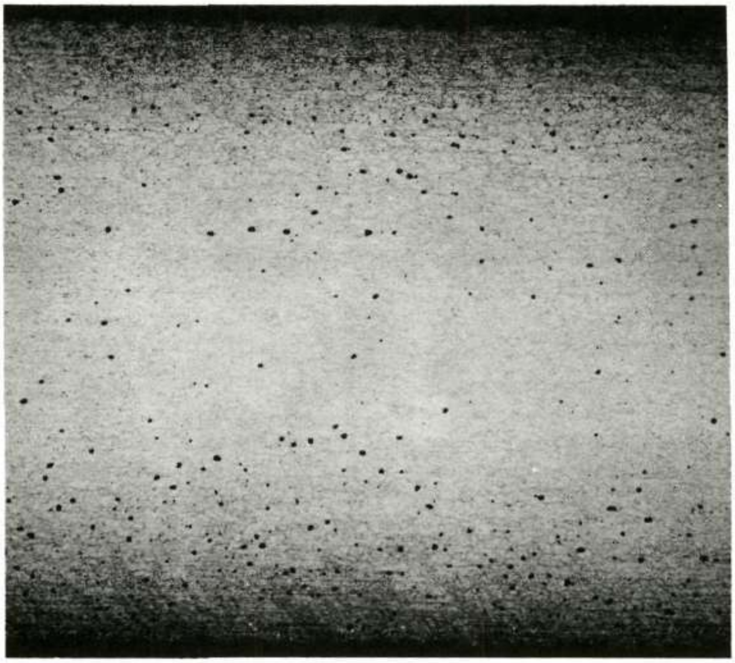
4 Hours



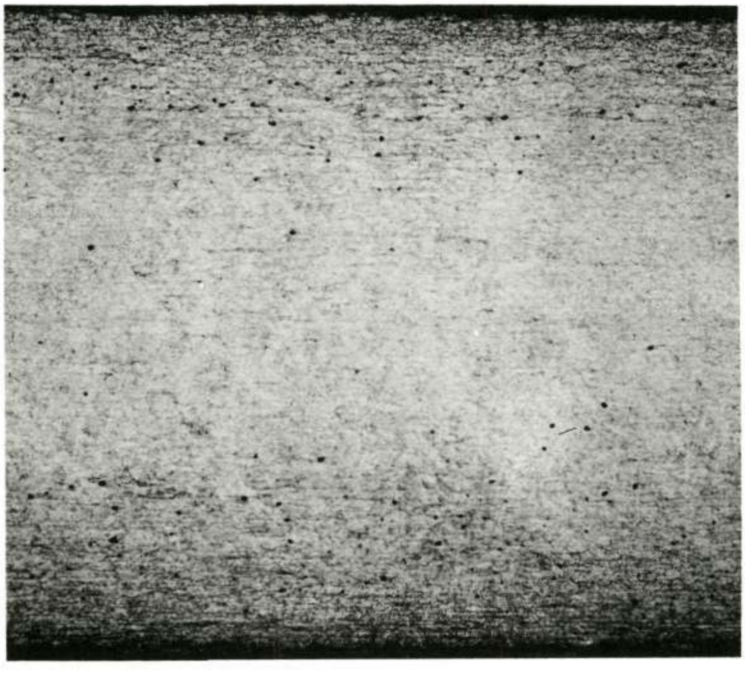
16 Hours

64 Hours

FIGURE E-2. CYCLIC OXIDATION TESTS AT 1204° C(2200° F) ON 0.038 cm.(0.015 in.) SHEET, HEAT 3849, MAG. 200X

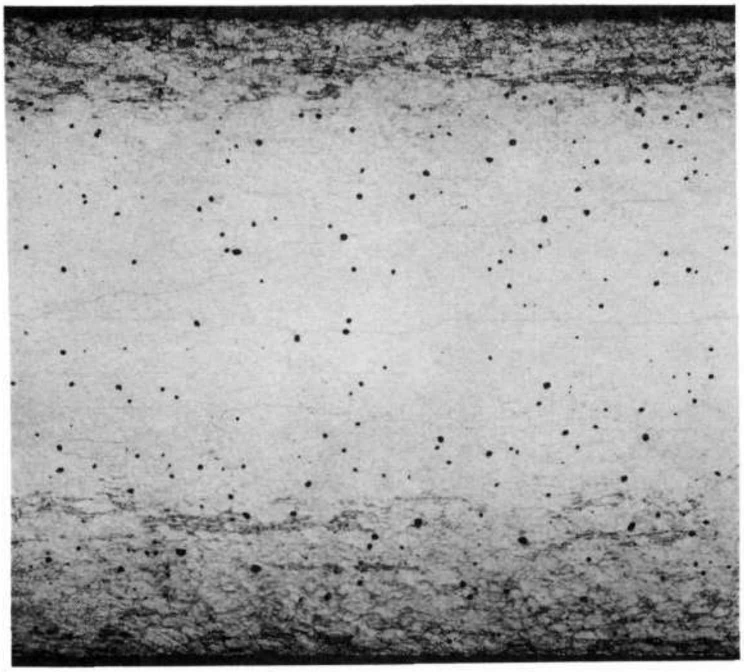


Heat 3925. Grain Size 0.013 mm at Surface
0.100 mm at Center

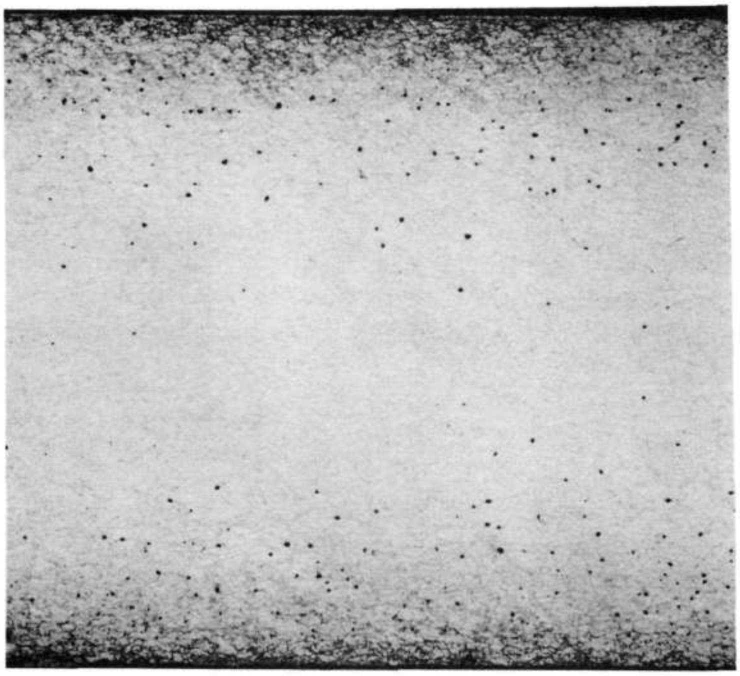


Heat 3927. Grain Size 0.015 mm

FIGURE E-3. PHOTOMICROGRAPHS OF Ni-16Cr-5Al-2ThO_2 ALLOYS AFTER 45 HOURS OF CYCLIC OXIDATION AT 1204° C (2200° F) 60X



Heat 3928. Grain Size 0.015 mm



Heat 3929. Grain Size 0.015 mm

FIGURE E-4. PHOTOMICROGRAPHS OF Ni-16Cr-5Al-2ThO$_2$ ALLOYS AFTER 45 HOURS OF CYCLIC OXIDATION TESTING AT 1204° C (2200° F) 60X particles. The Fe rich particles were not necessarily associated with oxygen; it was not possible to determine if carbon was present as well. At present no explanation can be afforded as to why Fe rich particles can remain stable in a Ni matrix for extended periods of time at 1204° C(2200° F).

The effect of grain size on degree of porosity was clearly established by the tests on Heats 3903 and 3915. Table E-2 shows the approximate percent porosity developed in these TDNiCrAl specimens as a function of grain size; also included in the table for comparative purposes are the Hastelloy-X and TDNiCr results. It is clear that porosity increases with increasing grain size. Again, this agrees with Whittenberger's observations; the explanation for the results is that fine grained materials have a greater capacity than coarse grained materials for absorbing the generated vacancies.

From the above it would not be expected that Al content per se would have a significant effect on the extent of porosity developed. If surface oxides are similar and have the same degree of adherence, then alloys of high and low Al content perform similarly; that is, the outward diffusion rate of Al in both cases should be essentially the same.

The specimens from Heats 3927, 3928 and 3929 all had grain sizes of 0.015 mm; the specimen from Heat 3925 had a surface region grain size of 0.013 mm and a central region grain size of 0.1 mm. A direct quantitative comparison between these specimens and those of their 3.5 percent Al

TABLE E-2

THE EFFECT OF GRAIN SIZE ON POROSITY FOR SELECTED ALLOYS AS A RESULT OF CYCLIC OXIDATION TESTING AT 1200° C (2200° F) FOR 45 HOURS

Heat No.	Nominal Composition	Grain Size (mm.)	Approximate Percent Porosity
3915	Ni-16Cr-3.5Al-2ThO_2	0.008	0.0
3915	Ni-16Cr-3.5Al-2ThO_2	0.013	0.5
3903	Ni-16Cr-3.5Al-2ThO_2	0.020	1.5
3903	Ni-16Cr-3.5Al-2ThO_2	0.100	2.0
3304	Ni-20Cr-2ThO_2	Coarse	4.0
Hastelloy-X	-	Coarse	2.0

counterpart regarding the extent of porosity cannot be made since the former were approximately three times the thickness of the latter. However, a visual comparison suggests fairly similar behavior.

The conclusions reached as a result of the alloy development with regard to oxidation behavior are as follows:

1. The most oxidation resistant alloys developed were of the class Ni-16%Cr-3.5 to 5.0%Al-2%ThO_2.
2. A preheat treatment in dry H_2 at 1177°C(2150°F) formed an Al_2O_3 film on the surfaces of specimens which inhibited the tendency of such alloys to exhibit anomalous oxidation behavior, at least for 50 hours at 1204°C(2200°F).
3. Although surface recession during the course of testing at temperatures to 1260°C(2300°F) was minimal, Kirkendall porosity sometimes developed; the extent of porosity increased with increasing temperature, time of exposure and increasing grain size.

4. Alloy Definition

On the basis of the data presented in the previous sections, two alloy compositions were chosen for further development. These alloys were as follows:

Ni-16Cr-3.5Al-2ThO_2

Ni-16Cr-5.0Al-2ThO_2

The former alloy containing the 3.5 percent aluminum level appears to afford the optimum combination of oxidation resistance, ductility and strength for space shuttle application.

The second alloy containing the 5.0 percent aluminum level is believed to afford additional oxidation resistance for long term use in aircraft engine applications.

APPENDIX F

TDNiCrAl Flat Product Inventory

TABLE F-1

TDNiCrAl FLAT PRODUCT SHIPMENTS

Quantity Pcs.	Size (cm.)	Size (in.)	Weight		Heat No.
			Kgs.	Lbs.	
1 (UnRx)	0.038x46.04x 71.12	0.015x18-1/8x28	1.00	2.2	3831
1 (UnRx)	0.038x40.64x103.19	0.015x16 x40-5/8	1.32	2.9	3831
1	0.038x41.91x 30.80	0.015x16-1/2x12-1/8	0.41	0.9	3831
1	0.038x46.04x 97.79	0.015x18-1/8x38-1/2	1.41	3.1	3831
1	0.038x42.23x 65.40	0.015x16-5/8x25-3/4	0.86	1.9	3831
SUB-TOTAL	5 pieces		4.99	11.0	3831
1	0.038x40.64x34.92	0.015x16 x13-3/4	0.41	0.9	3739
1	0.038x33.97x25.40	0.015x13-3/8x10	0.27	0.6	3739
1	0.038x20.00x15.24	0.015x 7-7/8x6	0.09	0.2	3739
1	0.038x24.76x27.30	0.015x 9-3/4x10-3/4	0.23	0.5	3739
1	0.038x24.76x10.48	0.015x 9-3/4x 4-1/8	0.09	0.2	3739
1	0.038x13.02x27.30	0.015x 5-1/8x10-3/4	0.09	0.2	3739
SUB-TOTAL	6 pieces		1.18	2.6	3739
1	0.038x35.56x34.29	0.015x14 x13-1/2	0.36	0.8	3738
1	0.038x36.83x36.83	0.015x14-1/2x14-1/2	0.41	0.9	3738
1	0.038x40.64x33.97	0.015x16 x13-3/8	0.36	0.8	3738
1	0.038x15.56x21.91	0.015x 6-1/8x 8-5/8	0.09	0.2	3738
1	0.038x11.43x16.51	0.015x 4-1/2x 6-1/2	0.05	0.1	3738
1	0.038x25.72x18.87	0.015x10-1/8x 6-1/4	0.14	0.3	3738
SUB-TOTAL	6 pieces		1.41	3.1	3738
1	0.038x39.37x24.13	0.015x15-1/2x 9-1/2	0.32	0.7	3774
SUB-TOTAL	1 piece		0.32	0.7	3774
1	0.038x24.45x25.72	0.015x 9-5/8x10-1/8	0.18	0.4	3741
1	0.038x25.40x29.53	0.015x10 x11-5/8	0.23	0.5	3741
1	0.038x22.54x10.16	0.015x 8-7/8x 4	0.09	0.2	3741
1	0.038x31.75x29.84	0.015x12-1/2x11-3/4	0.27	0.6	3741
1	0.038x16.83x10.16	0.015x 6-5/8x 4	0.05	0.1	3741
1	0.038x10.16x10.16	0.015x 4 x 4	0.05	0.1	3741
1	0.038x31.75x41.91	0.015x12-1/2x16-1/2	0.41	0.9	3741
SUB-TOTAL	7 pieces		1.27	2.8	3741

TABLE F-1 (CONT'D.)

TDNiCrAl FLAT PRODUCT SHIPMENTS

Quantity			Weight		Heat
Pcs.	Size (cm.)	Size (in.)	Kgs.	Lbs.	No.
1	0.038x18.41x39.69	0.015x 7-1/4x15-5/8	0.23	0.5	3740
1	0.038x12.70x39.37	0.015x 5 x15-1/2	0.18	0.4	3740
1	0.038x17.14x38.10	0.015x 6-3/4x15	0.18	0.4	3740
1	0.038x19.05x35.24	0.015x 7-1/2x13-7/8	0.18	0.4	3740
1	0.038x42.54x36.83	0.015x16-3/4x14-1/2	0.45	1.0	3740
1	0.038x38.73x32.38	0.015x15-1/4x12-3/4	0.36	0.8	3740
SUB-TOTAL	6 pieces		1.59	3.5	3740
1	0.038x30.48x37.15	0.015x12 x14-5/8	0.32	0.7	3771
1	0.038x33.02x41.59	0.015x13 x16-3/8	0.41	0.9	3771
1	0.038x29.21x40.00	0.015x11-1/2x15-3/4	0.36	0.8	3771
1	0.038x29.21x38.73	0.015x11-1/2x15-1/4	0.32	0.7	3771
1	0.038x29.84x38.42	0.015x11-3/4x15-1/8	0.32	0.7	3771
SUB-TOTAL	5 pieces		1.72	3.8	3771
1	0.038x29.53x41.27	0.015x11-5/8x16-1/4	0.36	0.8	3772
1	0.038x10.16x25.40	0.015x 4 x10	0.09	0.2	3772
1	0.038x41.91x37.15	0.015x16-1/2x14-5/8	0.45	1.0	3772
1	0.038x40.00x39.69	0.015x15-3/4x15-5/8	0.45	1.0	3772
1	0.038x40.64x38.73	0.015x16 x15-1/4	0.45	1.0	3772
1	0.038x40.32x29.21	0.015x15-7/8x11-1/2	0.32	0.7	3772
1	0.038x13.33x10.16	0.015x 5-1/4x 4	0.05	0.1	3772
1	0.038x38.10x36.83	0.015x15 x14-1/2	0.36	0.8	3772
1	0.038x10.16x10.16	0.015x 4 x 4	0.05	0.1	3772
SUB-TOTAL	9 pieces		2.59	5.7	3772
1	2.54x44.45x19.05	1.0x17-1/2x7-1/2	14.83	32.7	3848
SUB-TOTAL	1 piece		14.83	32.7	3848
1	2.54x42.23x20.0	1.0x16-5/8x7-7/8	15.65	34.5	3849
SUB-TOTAL	1 piece		15.65	34.5	3949
1	2.54x45.40x17.78	1.0x17-7/8x7	18.01	39.7	3812
SUB-TOTAL	1 piece		18.01	39.7	3812
1	4.44x10.79x45.72	1.75x4-1/4x18	17.46	38.5	3929
SUB-TOTAL	1 piece		17.46	38.5	3929

TABLE F-1 (CONT'D.)

TDNiCrAl FLAT PRODUCT SHIPMENTS

Quantity Pcs.	Size (cm.)	Size (in.)	Weight		Heat No.
			Kgs.	Lbs.	
1	0.69x15.24x27.94	0.270x6x11	2.45	5.4	3926
1	0.63x24.13x24.13	0.245x9-1/2x9-1/2	3.08	6.8	3926
SUB-TOTAL	2 pieces		5.53	12.2	3926
1	0.038x34.29x97.79	0.015x13-1/2x38-1/2	1.04	2.3	3905
1	0.038x33.02x96.52	0.015x13 x38	1.13	2.5	3905
1	0.038x33.02x83.50	0.015x13 x32-7/8	0.77	1.7	3905
1	0.038x36.19x79.99	0.015x14-1/4x31-1/2	1.00	2.2	3905
1(UnRx)	0.038x33.65x98.42	0.015x13-1/4x38-3/4	1.04	2.3	3905
1(UnRx)	0.038x35.56x102.23	0.015x14 x40-1/4	1.18	2.6	3905
1(UnRx)	0.038x34.92x97.15	0.015x13-3/4x38-1/4	1.09	2.4	3905
1(UnRx)	0.038x34.29x79.29	0.015x13-1/2x29-1/4	0.82	1.8	3905
SUB-TOTAL	8 pieces		8.07	17.8	3905
1(UnRx)	0.127x41.27x136.52	0.050x16-1/4x53-3/4	5.81	12.8	3927
SUB-TOTAL	1 piece		5.81	12.8	3927
1(UnRx)	0.127x41.91x80.64	0.050x16-1/2x31-3/4	3.40	7.5	3928
1(UnRx)	0.127x41.91x78.74	0.050x16-1/2x31	3.36	7.4	3928
1(UnRx)	0.127x90.64x77.47	0.050x16 x30-1/2	3.04	6.7	3928
SUB-TOTAL	3 pieces		9.80	21.6	3928
1(UnRx)	0.127x43.81x133.98	0.050x17-1/4x52-3/4	6.17	13.6	3929
1(UnRx)	0.127x43.81x162.56	0.050x17-1/4x64	6.85	15.1	3929
SUB-TOTAL	2 pieces		13.02	28.7	3929
1(UnRx)	0.127x43.50x141.60	0.050x17-1/8x55-3/4	5.99	13.2	3925
1(UnRx)	0.127x44.77x146.05	0.050x17-5/8x57-1/2	5.90	13.0	3925
SUB-TOTAL	2 pieces		11.89	26.2	3925
1	0.038x37.46x67.94	0.015x14-3/4x26-3/4	0.77	1.7	3903
SUB-TOTAL	1 piece		0.77	1.7	3903
GRAND TOTAL	68 pieces		136.47	300.6	

APPENDIX G

TDNiCrAl Extruded Product Inventory

TABLE G-1

FASTENER STOCK SHIPMENTS - TDNiCr

Quantity Pcs.	Nominal Size(cm.)	Nominal Size(in.)	Weight Kgs.	Weight Lbs.	Heat No.	Condition
1	3.18x181.60	1.250x71-1/2	11.20	24.7	3817	UnRx
1	2.86x128.90	1.125x50-3/4	7.89	17.4	3817	UnRx
2	1.91x30.48	0.750x12	1.45	3.2	3817	UnRx
SUB-TOTAL	4 pieces		20.55	45.3	3817	
3	0.96x152.40	0.375x60	3.49	7.7	3818	Rx
9	0.96x152.40	0.375x60	10.07	22.2	3818	UnRx
3	0.64x151.13	0.250x59-1/2	1.59	3.5	3818	Rx
8	0.64x151.13	0.250x59-1/2	3.81	8.4	3818	UnRx
SUB-TOTAL	23 pieces		18.96	41.8	3818	
3	1.02x167.64	0.400x66	3.72	8.2	3819	Rx
9	1.02x167.64	0.400x66	10.84	23.9	3819	UnRx
3	0.64x167.00	0.250x65-3/4	1.63	3.6	3819	Rx
6	0.64x167.00	0.250x65-3/4	2.99	6.6	3819	UnRx
SUB-TOTAL	21 pieces		19.19	42.3	3819	
8	1.02x182.24	0.400x71-3/4	10.52	23.2	3820	Rx
1	0.64x179.07	0.250x70-1/2	0.59	1.3	3820	Rx
1	0.64x111.76	0.250x44	0.36	0.8	3820	Rx
6	0.64x181.29	0.250x71-3/8	3.45	7.6	3820	UnRx
1	0.68x179.39	0.265x70-5/8	0.54	1.2	3820	UnRx
1	0.68x67.31	0.265x26-1/2	0.27	0.6	3820	UnRx
1	1.02x182.88	0.400x72	1.36	3.0	3820	UnRx
SUB-TOTAL	19 pieces		17.10	37.7	3820	
1	0.99x156.84	0.390x61-3/4	1.27	2.8	3821	Rx
1	.099x193.67	0.390x76-1/4	1.00	2.2	3821	Rx
4	0.99x198.06	0.390x78	5.22	11.5	3821	Rx
1	0.64x152.40	0.250x60	0.45	1.0	3821	Rx
3	0.64x193.04	0.250x76	1.81	4.0	3821	Rx
2	0.64x196.82	0.250x77-1/2	1.54	3.4	3821	Rx
9	0.64x196.82	0.250x77-1/2	5.76	12.7	3821	UnRx
SUB-TOTAL	21 pieces		17.06	37.6	3821	
1	2.86x152.40	1.125x60	9.57	21.1	3834	UnRx
1	3.05x139.70	1.200x55	8.75	19.3	3834	UnRx
1	2.54x30.48	1.000x12	1.86	4.1	3834	Rx
SUB-TOTAL	3 pieces		20.18	44.5	3834	

TABLE G-1 (CONT'D.)

FASTENER STOCK SHIPMENTS - TDNiCr

Quantity	Nominal		Weight		Heat	
Pcs.	Size(cm.)	Size(in.)	Kgs.	Lbs.	No.	Condition
1	1.27x121.92	0.500x48	1.54	3.4	3844	Rx
1	1.27x92.07	0.500x36-1/4	1.18	2.6	3844	Rx
1	1.27x104.77	0.500x41-1/4	1.32	2.9	3844	Rx
1	1.27x111.76	0.500x44	1.41	3.1	3844	Rx
1	1.27x113.03	0.500x44-1/2	1.41	3.1	3844	Rx
1	1.27x135.89	0.500x53-1/2	1.72	3.8	3844	Rx
1	1.27x152.40	0.500x60	1.91	4.2	3844	Rx
3	1.27x182.88	0.500x72	6.94	15.3	3844	Rx
1	1.27x252.09	0.500x99-1/4	3.18	7.0	3844	Rx
1	0.84x207.00	0.330x81-1/2	1.00	2.2	3844	Rx
2	0.84x183.52	0.330x72-1/4	1.81	4.0	3844	Rx
1	0.84x212.72	0.330x83-3/4	1.00	2.2	3844	UnRx
2	0.84x243.84	0.330x96	2.27	5.0	3844	UnRx
SUB-TOTAL	17 pieces		26.67	58.8	3844	
2	1.27x95.88	0.500x37-3/4	2.49	5.5	3845	Rx
2	1.27x113.03	0.500x44-1/2	2.86	6.3	3845	Rx
4	1.27x182.88	0.500x72	9.30	20.5	3845	Rx
1	1.27x215.26	0.500x84-3/4	2.72	6.0	3845	Rx
1	1.27x238.12	0.500x93-3/4	2.99	6.6	3845	Rx
2	0.84x186.69	0.330x73-1/2	1.81	4.0	3845	Rx
1	0.84x205.74	0.330x81	1.00	2.2	3845	Rx
1	0.84x215.90	0.330x85	1.04	2.3	3845	UnRx
2	0.84x241.30	0.330x95	2.27	5.0	3845	UnRx
SUB-TOTAL	16 pieces		26.49	58.4	3845	
GRAND TOTAL	124 pieces		166.4	366.4		

TABLE G-2

FASTENER STOCK SHIPMENTS - TDNiCrAl

Quantity	Nominal		Weight		Heat	
Pcs.	Size(cm.)	Size(in.)	Kgs.	Lbs.	No.	Condition
1	1.27x 30.48	0.500x12	0.36	0.8	3863	UnRx
1	1.27x182.88	0.500x72	2.27	5.0	3863	UnRx
1	1.27x225.42	0.500x88-3/4	2.77	6.1	3863	UnRx
1	1.27x248.92	0.500x98	3.08	6.8	3863	UnRx
1	1.27x370.81	0.500x146	4.35	9.6	3863	UnRx
1	1.27x349.25	0.500x137-1/2	4.17	9.2	3863	UnRx
1	1.27x372.11	0.500x146-1/2	4.40	9.7	3863	UnRx
1	1.27x416.56	0.500x164	5.08	11.2	3863	UnRx
SUB-TOTAL	8 pieces		26.49	58.4	3863	
1	1.27x 30.48	0.500x12	0.36	0.8	3864	UnRx
1	1.27x290.83	0.500x114-1/2	3.36	7.4	3864	UnRx
1	1.27x293.37	0.500x115-1/2	3.36	7.4	3864	UnRx
1	1.27x274.32	0.500x108	3.27	7.2	3864	UnRx
1	1.27x190.50	0.500x 75	2.27	5.0	3864	UnRx
1	1.27x152.40	0.500x 60	1.77	3.9	3864	UnRx
1	1.27x152.40	0.500x 60	1.77	3.9	3864	UnRx
1	1.27x140.97	0.500x 55-1/2	1.63	3.6	3864	UnRx
1	1.27x138.43	0.500x 54-1/2	1.59	3.5	3864	UnRx
1	0.76x243.84	0.300x 96	0.91	2.0	3864	UnRx
1	0.76x223.52	0.300x 88	0.82	1.8	3864	UnRx
2	0.76x214.63	0.300x 84-1/2	1.72	3.8	3864	UnRx
2	0.76x185.42	0.300x 73	1.36	3.0	3864	UnRx
SUB-TOTAL	15 pieces		24.18	53.3	3864	
1	1.27x 30.48	0.500x 12	0.36	0.8	3865	UnRx
1	1.27x266.70	0.500x105	3.18	7.0	3865	UnRx
1	1.27x265.43	0.500x104-1/2	3.18	7.0	3865	UnRx
1	1.27x264.16	0.500x104	3.18	7.0	3865	UnRx
1	1.27x262.89	0.500x103-1/2	3.18	7.0	3865	UnRx
1	1.27x254.00	0.500x100	3.08	6.8	3865	UnRx
1	1.27x152.40	0.500x 60	1.91	4.2	3865	UnRx
1	0.76x245.11	0.300x 96-1/2	1.13	2.5	3865	UnRx
1	0.76x231.14	0.300x 91	1.04	2.3	3865	UnRx
1	0.76x212.72	0.300x 83-3/4	1.00	2.2	3865	UnRx
1	0.76x200.03	0.300x 78-3/4	1.00	2.0	3865	UnRx
2	0.76x182.88	0.300x 72	1.63	3.6	3865	UnRx
SUB-TOTAL	13 pieces		23.77	52.4	3865	

TABLE G-2 (CONT'D.)

FASTENER STOCK SHIPMENTS - TDNiCrAl

Quantity	Nominal		Weight		Heat	
Pcs.	Size(cm.)	Size(in.)	Kgs.	Lbs.	No.	Condition
1	1.27x300.35	0.500x118-1/4	3.45	7.6	3867	UnRx
1	1.27x299.72	0.500x118	3.40	7.5	3867	UnRx
2	1.27x297.18	0.500x117	6.71	14.8	3867	UnRx
1	1.27x260.34	0.500x102-1/2	2.95	6.5	3867	UnRx
1	1.27x238.76	0.500x 94	2.45	5.4	3867	UnRx
1	0.76x236.22	0.300x 93	0.86	1.9	3867	UnRx
1	0.76x217.16	0.300x 85-1/2	0.82	1.8	3867	UnRx
1	0.76x203.83	0.300x 80-1/4	0.77	1.7	3867	UnRx
1	0.76x127.00	0.300x 50	0.50	1.1	3867	UnRx
SUB-TOTAL	10 pieces		21.91	48.3	3867	
1	1.27x 30.48	0.500x 12	0.36	0.8	3866	UnRx
1	1.27x306.00	0.500x120-1/2	3.45	7.6	3866	UnRx
1	1.27x279.40	0.500x110	3.08	6.8	3866	UnRx
1	1.27x273.05	0.500x107-1/2	2.95	6.5	3866	UnRx
1	1.27x162.56	0.500x 64	1.86	4.1	3866	UnRx
2	1.27x152.40	0.500x 60	3.36	7.4	3866	UnRx
1	1.27x128.27	0.500x 50-1/2	1.45	3.2	3866	UnRx
1	1.27x120.65	0.500x 47-1/2	1.32	2.9	3866	UnRx
1	0.76x238.76	0.300x 94	0.82	1.8	3866	UnRx
1	0.76x228.60	0.300x 90	0.68	1.5	3866	UnRx
1	0.76x213.36	0.300x 84	0.82	1.8	3866	UnRx
1	0.76x195.58	0.300x 77	0.68	1.5	3866	UnRx
1	0.76x182.88	0.300x 72	0.73	1.6	3866	UnRx
1	0.76x151.13	0.300x 59-1/2	0.54	1.2	3866	UnRx
SUB-TOTAL	15 pieces		22.09	48.7	3866	
1	1.27x287.02	0.500x113	3.54	7.8	3868	UnRx
1	1.27x285.75	0.500x112-1/2	3.49	7.7	3868	UnRx
1	1.27x273.04	0.500x107-1/2	3.27	7.2	3868	UnRx
2	1.27x266.70	0.500x105	6.53	14.4	3868	UnRx
1	1.27x251.46	0.500x 99	3.04	6.7	3868	UnRx
1	0.76x414.02	0.300x163	2.00	4.4	3868	UnRx
2	0.76x412.75	0.300x162-1/2	3.95	8.7	3868	UnRx
SUB-TOTAL	9 pieces		25.81	56.9	3868	
GRAND TOTAL	70 pieces		144.25	318.0		

TABLE G-3

RECTANGULAR EXTRUSION SHIPMENTS - TDNiCrAl

Quanity Pcs.	Nominal Size(cm.)	Nominal Size(in.)	Weight Kgs.	Weight Lbs.	Heat No.	Alloy
1	4.44x10.79x70.48	1-3/4x4$\frac{1}{4}$x27-3/4	26.0	57.3	3915	3.5 Al
1	4.44x10.79x50.80	1-3/4x4$\frac{1}{4}$x20	18.5	41.0	3915	3.5 Al
1	3.49x15.24x152.4	1-3/8x6 x60	64.9	143.0	3916	3.5 Al
1	4.44x10.79x120.6	1-3/4x4$\frac{1}{4}$x47$\frac{1}{2}$	44.8	98.8	3924	5.0 Al
1	4.44x10.79x73.66	1-3/4x4$\frac{1}{4}$x29	27.1	59.8	3925	5.0 Al
1	3.49x15.24x123.2	1-3/8x6 x48$\frac{1}{2}$	51.3	113.0	3934	5.0 Al
1	3.17x15.24x19.05	1-1/4x6 x7$\frac{1}{2}$	7.3	16.0	3934	5.0 Al
1	2.86x15.24x17.14	1-1/8x6 x6-3/4	6.4	14.0	3934	5.0 Al
1	3.17x11.43x44.45	1-1/4x4$\frac{1}{2}$x17$\frac{1}{2}$	12.9	28.5	3936	5.0 Al
1	3.17x11.43x40.64	1-1/4x4$\frac{1}{2}$x16	11.6	25.5	3936	5.0 Al
1	3.17x11.43x40.64	1-1/4x4$\frac{1}{2}$x16	11.6	25.5	3936	5.0 Al
1	3.17x11.43x40.64	1-1/4x4$\frac{1}{2}$x16	11.6	25.5	3936	5.0 Al
1	3.17x11.43x40.64	1-1/4x4$\frac{1}{2}$x16	11.6	25.5	3936	5.0 Al
1	2.54x11.43x19.05	1 x4$\frac{1}{2}$x7$\frac{1}{2}$	4.7	10.4	3936	5.0 Al
1	3.49x15.24x21.59	1-3/8x6 x8$\frac{1}{2}$	8.8	19.5	3938	5.0 Al
1	3.49x15.24x69.94	1-3/8x6 x26-3/4	28.8	63.7	3938	5.0 Al
GRAND TOTAL	16 pieces		347.9	767.0		

TABLE G-4

DENSIFIED BILLET SHIPMENTS - TDNiCrAl

Quantity Pcs.	Nominal Size(cm.)	Nominal Size(in.)	Weight Kgs.	Weight Lbs.	Heat No.	Alloy
1	19.050Øx36.51	7½Øx14-3/8	83.0	183.0	3931	3.5 Al
1	19.050Øx34.29	7½Øx13-1/2	79.6	175.5	3932	3.5 Al
1	19.050Øx31.75	7½Øx12-1/2	73.5	162.0	3933	5.0 Al
1	19.050Øx34.29	7½Øx13-1/2	78.9	174.0	3935	5.0 Al
1	19.050Øx35.56	7½Øx14	81.5	179.5	3939	5.0 Al
GRAND TOTAL 5 pieces			396.5	874.0		