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for
CRADA Number C/ORNL 01-0621

Between

UT-Battelle, LLC

and

Special Metals Corp.
(Participant)

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For the Participant:

Gaylord Smith
(Name)

Tech Manager – New Products
(Title)

October 15, 2007
(Date)

ORNL [illegible]

CRADA No. ORNL01-0629

OAK RIDGE
NATIONAL LABORATORY

MANAGED BY UT-BATTELLE
FOR THE DEPARTMENT OF ENERGY

CRADA FINAL REPORT
THERMO-MECHANICAL PROCESSING PARAMETERS FOR THE INCONEL® ALLOY 740

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CRADA FINAL REPORT
FOR CRADA NO. ORNL**01-0629**

THERMO-MECHANICAL PROCESSING PARAMETERS FOR THE INCONEL® ALLOY 740

Gail Mackiewicz Ludtka

ORNL

Materials Science and Technology Division

AND

Gaylord Smith

Special Metals Corporation

ABSTRACT

In 2000, a Cooperative Research and Development Agreement (CRADA) was undertaken between the Oak Ridge National Laboratory (ORNL) and the Special Metals Corporation (SMC) to determine the mechanical property response of the IN740 alloy to help establish thermo-mechanical processing parameters for the use of this alloy in supercritical and ultra-critical boiler tubes with the potential for other end uses. SMC had developed an alloy, commercially known as INCONEL alloy 740, which exhibited various beneficial physical, mechanical, and chemical properties. As part of SMC's on-going efforts to optimize this alloy for targeted boiler applications there was a need to develop an understanding of the thermo-mechanical response of the material, characterize the resulting microstructure from this processing, and possibly, utilize models to develop the appropriate processing scheme for this product.

STATEMENT OF OBJECTIVES

The overall technical objective of this Cooperative Research and Development Agreement (CRADA) between UT-Battelle, LLC, and SMC was to develop thermo-mechanical processing parameters for INCONEL alloy 740. The mechanical property data generated in this effort would enable a better understanding of the thermo-mechanical response of this material and enable SMC to develop the deformation and heat treatment processing parameters necessary to yield a product suitable for boiler tubes. The work performed to accomplish this goal included elevated and room temperature mechanical testing, thermal treatments, welding processing, the development and testing of filler materials; and microstructural characterization. A preliminary evaluation of the starting product was included in this goal to provide a comparison with baseline properties.

SCOPE OF WORK

To achieve the technical goals of this CRADA, the scope of this work included conducting elevated and room temperature mechanical tests on as-processed material and materials cold-worked to varying degrees of deformation; and, preparing, welding, testing and analyzing these welded test samples. These data are necessary in order for boiler tube manufacturers to develop the deformation and heat treatment parameters and thermal processing cycles needed for thermo-mechanical processing. The following tasks summarize the efforts completed in order to accomplish the overall objective of this project:

Task 1: Define Thermo-Mechanical Processing and Weldability of Boiler Tube Materials.
Task 2:Simulate Boiler Tube Fabrication and Test the Final Product (*eliminated due to project changes)*.
Task 3: Final Report

BENEFITS TO THE FUNDING DOE OFFICE'S MISSION

The key mission of the US DOE, Office of Energy Efficiency and Renewable Energy (EERE), Industrial Materials for the Future Program is to improve energy efficiency and energy utilization in energy intensive US industries. The overall goal of the parent program, of which this project is a part, is focused at developing materials and improving their metallurgical and mechanical performance so that these materials effectively operate at higher temperatures and/or pressures thereby, improving the (heat rate) energy efficiency of the working medium (steam) in power plants. Historically, problems (high thermal stresses and fatigue cracking) have occurred in the austenitic stainless steels often used for heavy section components exposed to high temperatures due, in part, to the low thermal conductivity and high thermal expansion of these steels. Consequently, alternate materials are being evaluated. IN740 is one of these candidate materials. The goal of this project (under this CRADA) was to characterize the performance of IN740 to enable a better understanding of the thermo-mechanical response of this material and to enable SMC to develop the deformation and heat treatment processing parameters necessary to yield a product suitable for boiler tubes.

TECHNICAL DISCUSSION OF WORK PERFORMED BY ALL PARTIES

Introduction

IN740 is an age hardenable nickel-chromium-cobalt alloy developed by Special Metals Corporation. Its nominal chemical composition is shown in Table 1. IN740 is age hardenable by the precipitation of a second phase, gamma prime (γ'). It is a nickel-based alloy, solid solution strengthened by cobalt. Chromium imparts high temperature corrosion resistance, and during heat treatment, niobium, aluminum, and titanium form the gamma prime precipitates required for strengthening. The optimum precipitation heat treating temperature for the alloy is 1400°F (760°C). Some physical properties for this alloy are listed in Table 2.

Table 1. – Nominal Composition of INCONEL alloy 740

ALLOY	Ni	Cr	Co	Nb	Ti	Al	Fe	Mo	Si	Mn	Zr	C	Ta
IN740	Bal.	25	20	2.0	1.8	0.9	1.0	0.6	0.5	0.3	0.04	0.03	--

Table 2. – Physical Constants of INCONEL alloy 740

Density, g/cm^3	8.109
lb/in^3	0.293
Melting Range, °C	1288 – 1362
°F	2350 – 2484

Objectives

The overall technical objective of this CRADA was to develop thermo-mechanical processing parameters for INCONEL alloy 740. The mechanical property data generated in this effort were intended to enable a better understanding of the thermo-mechanical response of this material and enable SMC to develop the deformation and heat treatment processing parameters necessary to yield a product suitable for boiler tubes.

Benefits

Since the development of materials properties at elevated operating temperatures has a major impact on the projection of capital and operating costs for the advanced steam cycle power plants, an accurate assessment based on knowledge of material performance was judged to be essential to the development process. This CRADA was expected to provide the data necessary to develop an understanding of the thermo-mechanical response of the material, characterize the resulting microstructure from this processing, and provide these data ultimately for use in computer models to develop the appropriate processing scheme for this product.

Tasks and Work Performed

The technical objective of this CRADA was to develop thermo-mechanical processing parameters for INCONEL alloy 740. The major tasks defined for this project were to *Define Thermo-Mechanical Processing and Weldability of Boiler Tube Materials* (Task 1); *Simulate Boiler Tube Fabrication and Test the Final Product*; (Task 2); and to document these results in a *Final Report* (Task 3). A redirection of program goals resulted in ORNL completing Task 1 (the thermomechanical property portion) and Task 3 (Final Report) of this CRADA. This report documents these results. In addition, included in this report (in Appendix 2), are some results provided by SMC on the welding performance of IN740 based on work that they conducted during the course of this CRADA. SMC conducted a full penetration butt weld on ¼" thick INCONEL 740 (complete chemistry can be seen in Table A2-1 of Appendix 2.) utilizing the GTAW process with matching composition filler. Parameters used to achieve these welds are summarized in Appendix 2 (Table A2-2). Pre-weld and post-weld conditions along with mechanical testing data are documented in Table A2-3. Additional mechanical testing was conducted and these results are presented in Figures A2-1 through A2-7.

Task 1: Define Thermo-Mechanical Processing and Weldability of Boiler Tube Materials.

SMC provided the alloy IN740 as rod stock in several conditions (heat number #574582). One of these conditions was provided in the standard bar annealed condition (SA - 2050F (1120C) for 30 minutes followed by a water quench or rapid air cool) that has a typical hardness of Rockwell B 90. This condition is *equivalent to* the hot worked and annealed condition and acted as a baseline for the subsequently provided cold worked material. Several cold-worked conditions were also provided. Each piece varied in the amount of cold work, ranging from 0% to 40% cold work. The details for these pieces (as-received) are described below in Table 3. Oak Ridge National Laboratory (ORNL) fabricated, prepared and tested full size tensile specimens (for room temperature testing) and Gleeble™ compression specimens (for elevated temperature testing) from the materials provided; and determined and characterized both tensile and compressive properties for IN740.

Table 3: Condition of tensile specimen blanks received

Initial Thickness [inch]	As-rolled Thickness[inch]	% Cold Work	Solution Treatment	Hot Worked & Annealed Condition
0.591[1]	--	--	2050°F (1121°C)	2050°F (1121°C)/1h/Water Quench
0.591[2]	0.532	10%	"	"
0.532[3]	0.473	20%	"	"
0.473[4]	0.414	30%	"	"
0.414[5]	0.355	40%	"	"

1) Piece of 0.591" plate annealed at 2050°F / 1 hour / Water Quench
2) Piece of 0.591" annealed plate cold rolled down to 0.532" (10% CW)
3) Piece of 0.532" annealed plate cold rolled down to 0.473" (20% CW)
4) Piece of 0.473" annealed plate cold rolled down to 0.414" (30% CW)
5) Piece of 0.414" annealed plate cold rolled down to 0.355" (40% CW)

Compression tests were conducted on a Gleeble testing machine at ORNL for elevated temperatures and strain rates selected to simulate possible extrusion processing temperatures. The testing parameters evaluated are summarized in the test matrix (Table 4) below for the specific testing temperatures, hold times, quench rates, and strain rates as indicated. A range of test temperatures and strain rates were identified and prioritized by SMC as primary (P -most important) and secondary (S- less important) due to the limited funding available for this testing. All "P" samples were tested with a type-S thermocouple attached (spot welded) in the middle of the samples, where the diametral strain gage was also placed. The diameter and length of each specimen were measured prior to and after testing to monitor the strain. During testing, isothermal conditions were maintained along the test specimen as much as possible.

Table 4. Gleeble Test Priority Schedule for the 740 Alloy for the as-cast and hot-worked & annealed conditions.

Test Temperatures	1000°C	1100°C	1200°C
Strain Rates [s^{-1}]			
1	S	S	S
5	P	P	P
10	P	P	P
20	S	S	S

NOTE: P indicates the highest priority conditions to be run 1st; and S indicates the conditions that were of secondary importance (and were not run due to limited funding availability.)

The results from this testing are summarized below in Figure 1. These data demonstrate that the forging force required to thermomechanically work IN740 can be significantly reduced (by ~60%) if the working temperature is increased by 200°C to 1200°C (2192°F) or by ~40% if the working temperature is increased by 100°C to 1100°C (2012°F). Further, these data also indicate that at strains required for forming IN740, varying the strain rate from 5 to 10 per second has a little effect on the stress-strain response for this alloy.

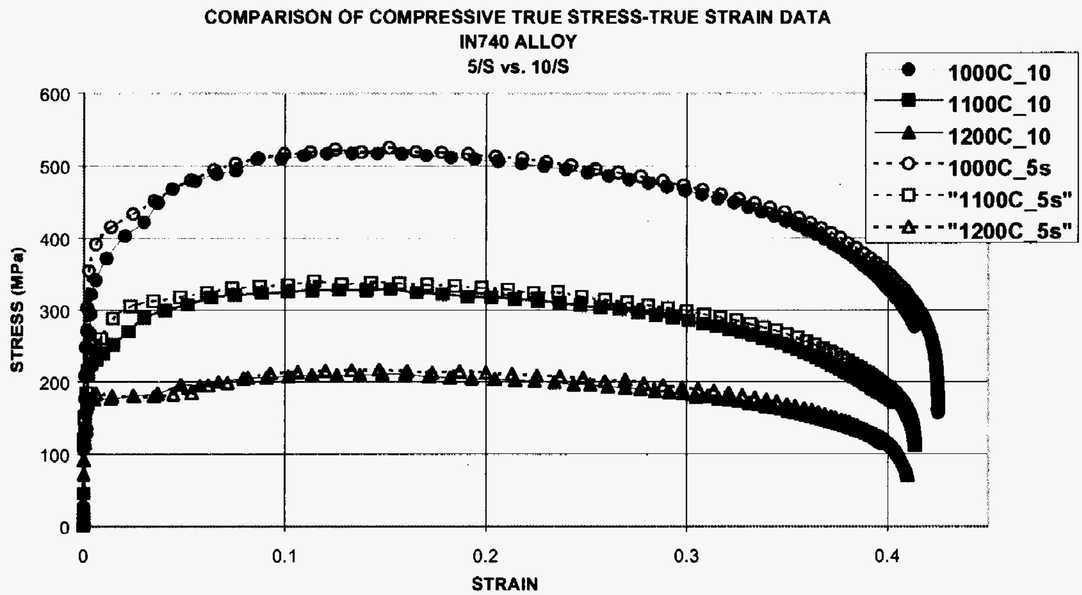


Figure 1 Summary of elevated temperature and strain rates effects on IN740 results generated by conducting compression tests using a Gleeble testing machine.

Table 5. Summary Table of Room Temperature Tensile Properties of INCONEL alloy 740.
(*Cross Head Speed = 0.0074"/s.; 0.444 in/min; 1" G. L. extensometer; 0.252" dia tensile)*

Testing Temperature (°C)	Yield Strength, .02% (0.2%) (ksi)	Ultimate Tensile Strength, σ_{UTS} (ksi)	Elongation, % Plastic at Break	Reduction of Area, %	Elastic Modulus, Msi	Material Condition
23	40.6 (47.3)	120.3	63.2	69.8	29.3	Solution Annealed (SA)
23	64.5 (79.5)	121.5	52.5	64.0	33.1	SA + 10% Cold Work (CW)
23	89.1 (114.4)	138.	31.1	57.8	28.7	SA + 20% CW
23	113.3 (146.8)	162.5	17.3	53.0	28.9	SA + 30% CW
23	116.1 (152.3)	170.5	13.4	50.8	29.2	SA + 40% CW

The room temperature tensile properties determined for Heat # D574582 of Inconel alloy 740 listed in Table 5 are plotted in Figure 2 below. During this testing, the cross head speed was controlled to be maintained at 0.0074"/s.; 0.444 in/min using a 1" G. L. extensometer on a 0.252" diameter tensile specimen. These tests were conducted at room temperature and hardness values were determined for materials that were cold worked 0, 10, 20, 30, 40, and 50%. The .02%, 0.2% yield strength, ultimate tensile strength, elongation (% plastic at break), % reduction of area, and elastic modulus were determined from these tests. These data are fundamental to determining the tube reducing parameters for the alloy. The results from these tests demonstrated that the yield strength of IN740 increased significantly up to 30% cold work, and then leveled off. In comparing the UTS values for all material conditions indicated (Table 5 and Figure 2), these data indicate that the UTS increases with the level of cold work applied to IN740. These unexpected results suggest that, perhaps, either a preferred texture developed with increasing amounts of cold work, or, more likely, some aging (due to sample heating) occurred during the cold working operation in this IN740 alloy.

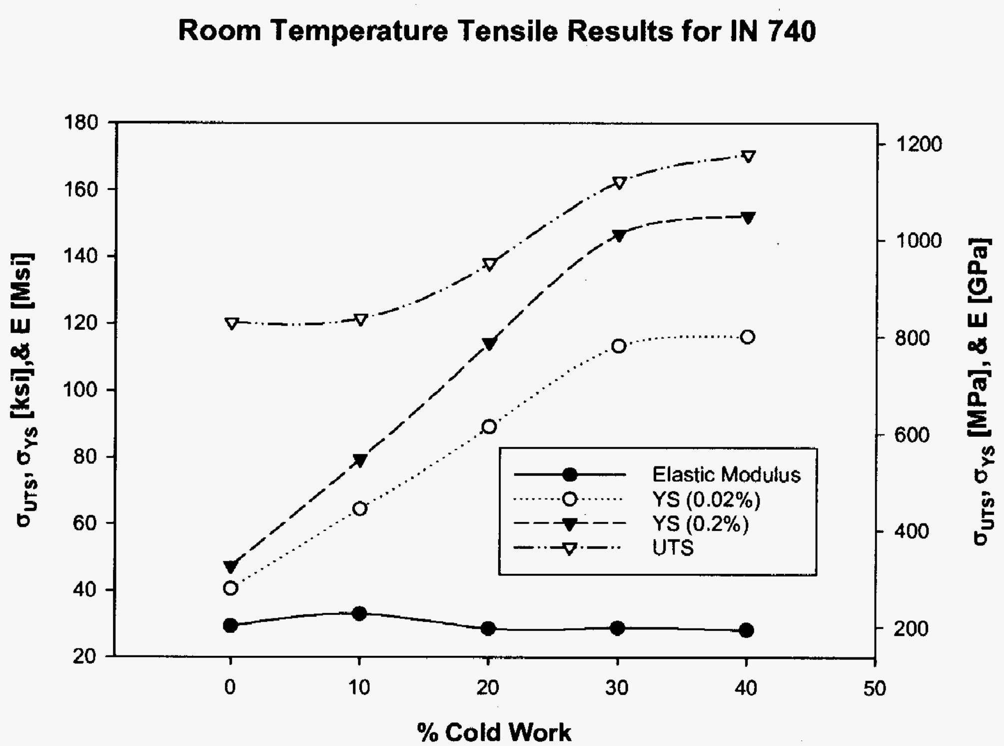


Figure 2 Plots summarizing stress-strain tensile data determined for IN740 at room temperature on full size tensile specimens tested following the ASTM E-8 standard testing method.

SUBJECT INVENTIONS (As defined in CRADA)
None

COMMERCIALIZATION POSSIBILITIES
Unknown

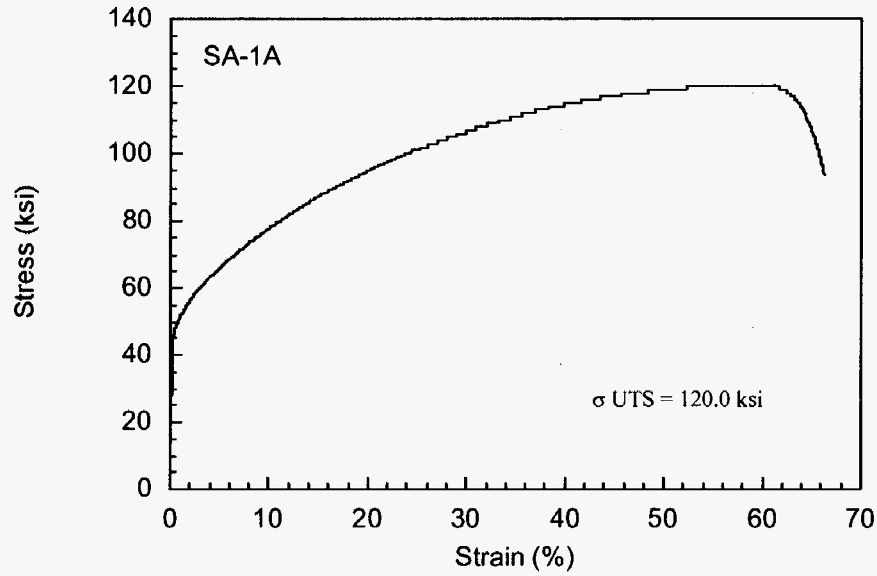
PLANS FOR FUTURE COLLABORATION

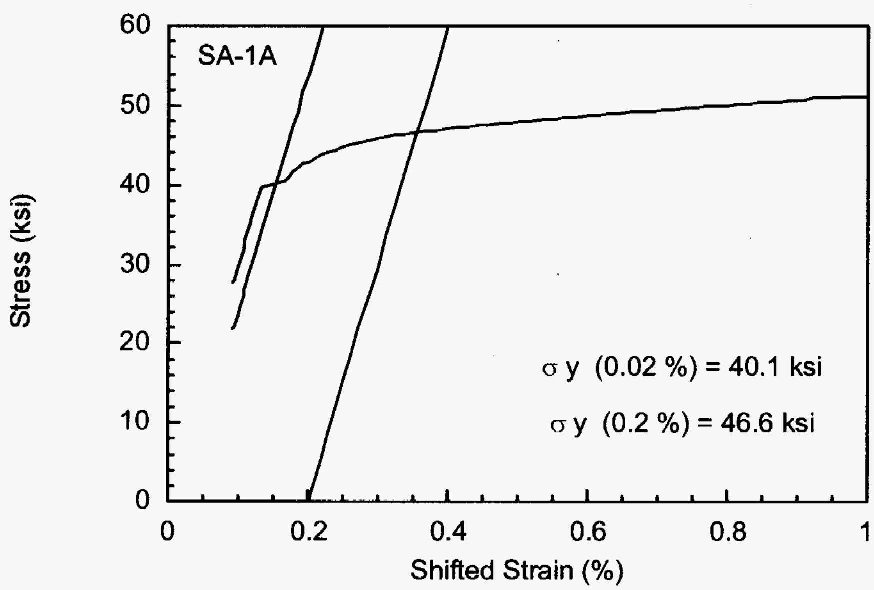
Several attempts were made to continue collaborations with SMC, but no additional projects were mutually identified that were not covered under other areas of this program.

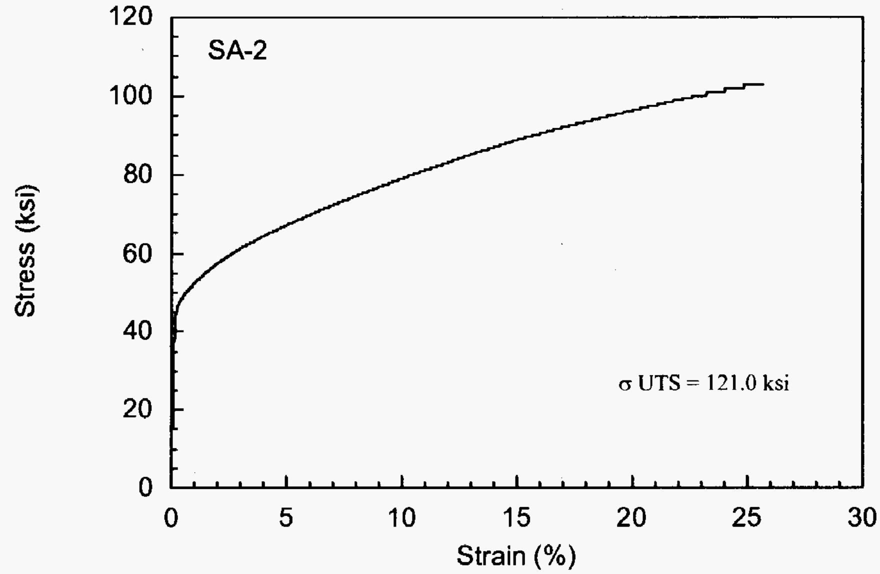
CONCLUSIONS

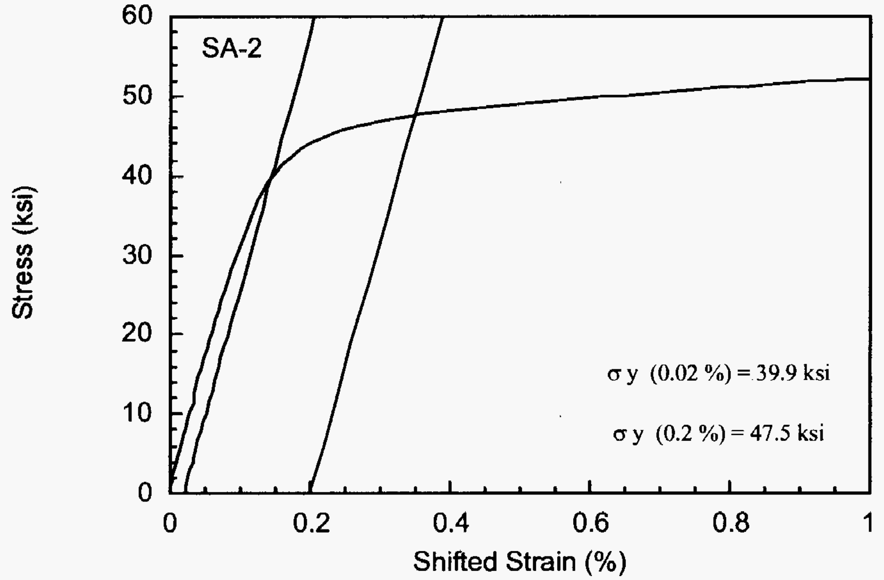
Room temperature tensile and elevated temperature compression flow stress data were generated for the IN740 alloy to develop an understanding of the thermo-mechanical response of this material and enable SMC to develop the deformation and heat treatment processing parameters necessary to yield a product suitable for boiler tubes. A preliminary evaluation of the starting product was included in this goal to provide a comparison with baseline properties. These data are important for SMC to evaluate and develop the deformation and thermal processing parameters necessary to yield an IN740 alloy product suitable for boiler tube applications. These data also could be incorporated into computer models to further assist SMC and their customers in evaluating and developing appropriate processing schemes for this product. These results, in conjunction with the welding processing evaluation; the development and testing of filler materials; and microstructural characterization (conducted by SMC and not included as part of this report), provide the information for SMC to evaluate the use of IN740 for boiler tube applications.

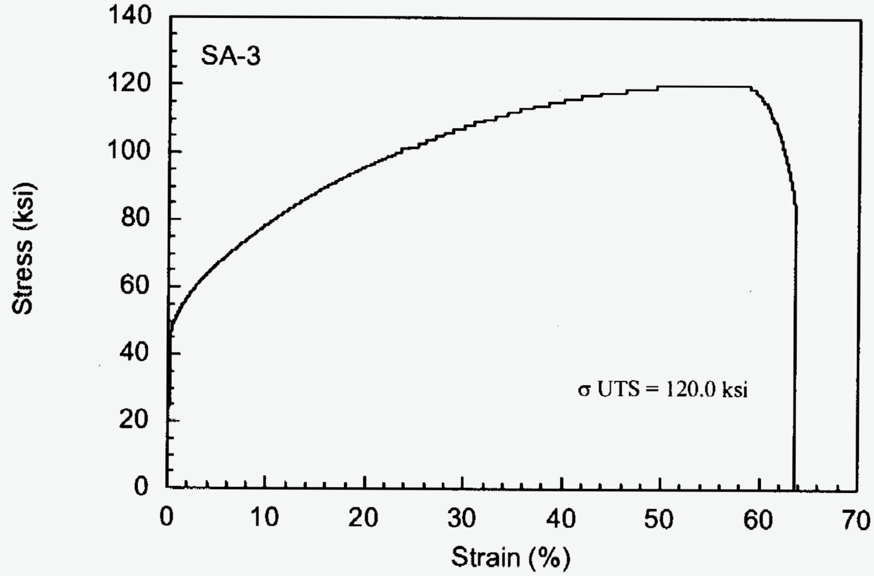
APPENDIX 1 – ROOM TEMPERATURE TENSILE TESTING RESULTS FOR ALLOY IN740

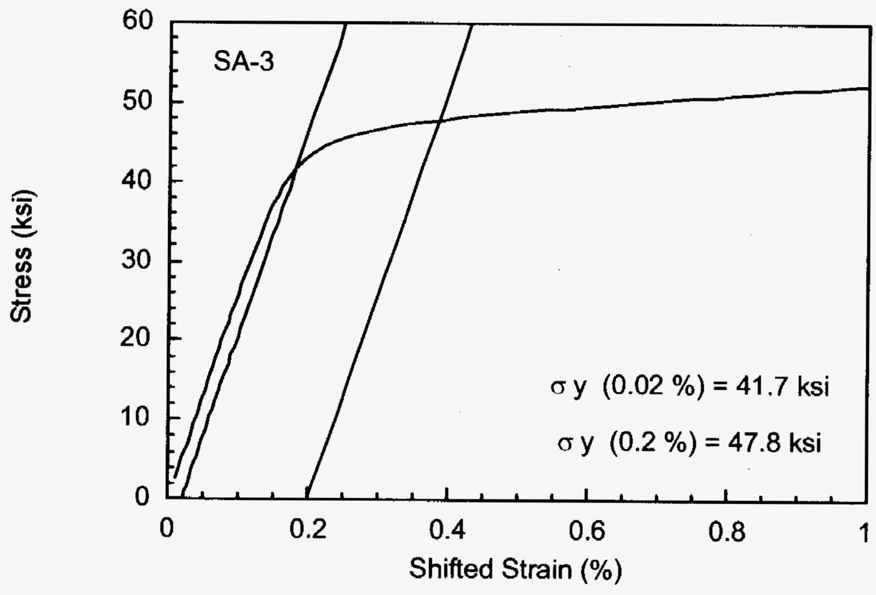


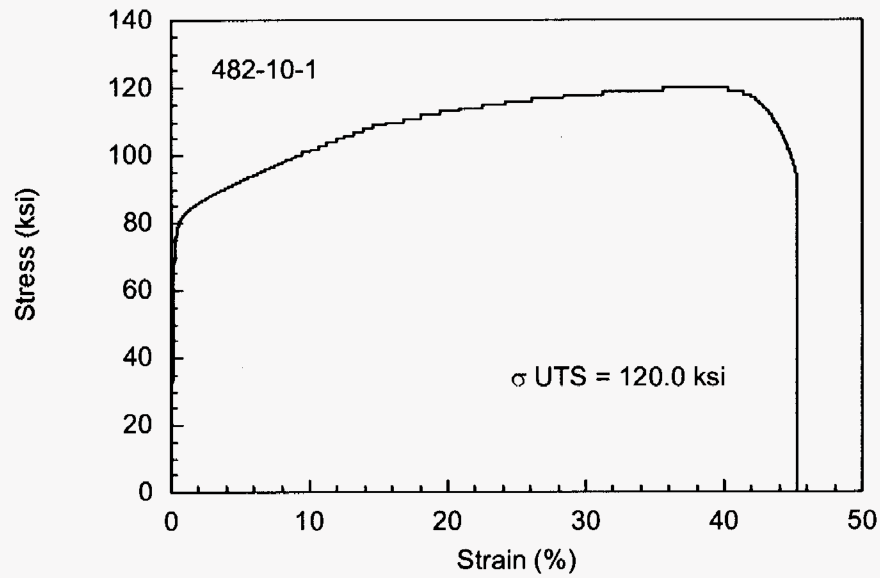


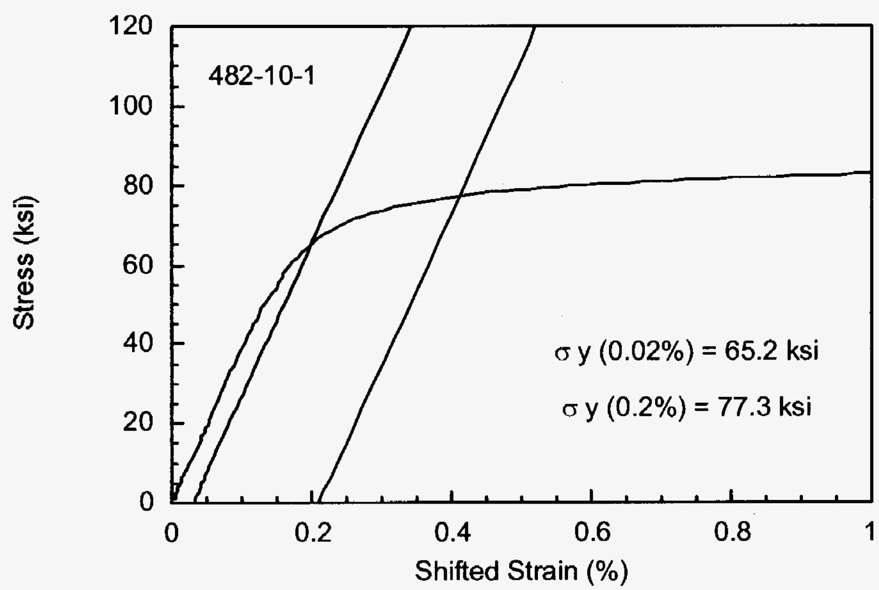

SA-2
σ UTS = 121.0 ksi
Stress (ksi)
Strain (%)
120
100
80
60
40
20
0
0
5
10
15
20
25
30

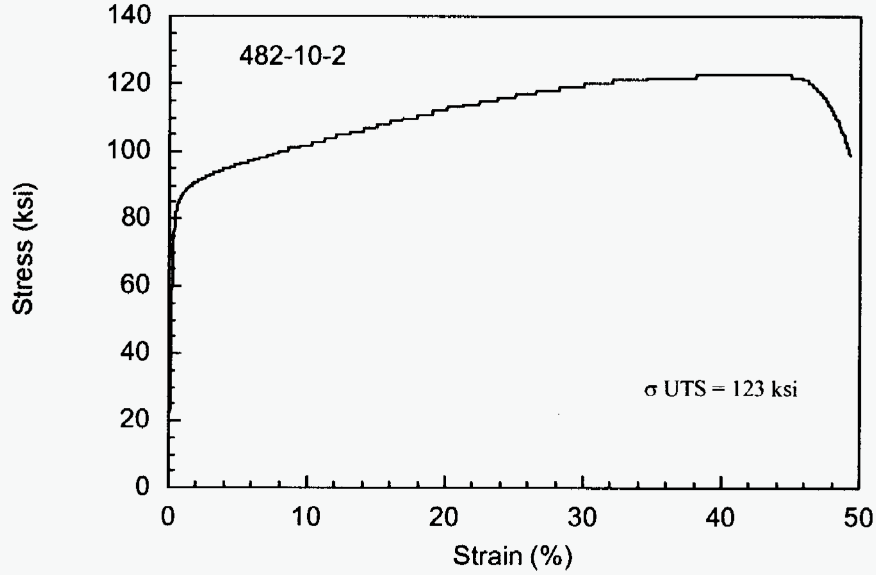

SA-2
σ y (0.02 %) = 39.9 ksi
σ y (0.2 %) = 47.5 ksi
Stress (ksi)
Shifted Strain (%)
60
50
40
30
20
10
0
0
0.2
0.4
0.6
0.8
1

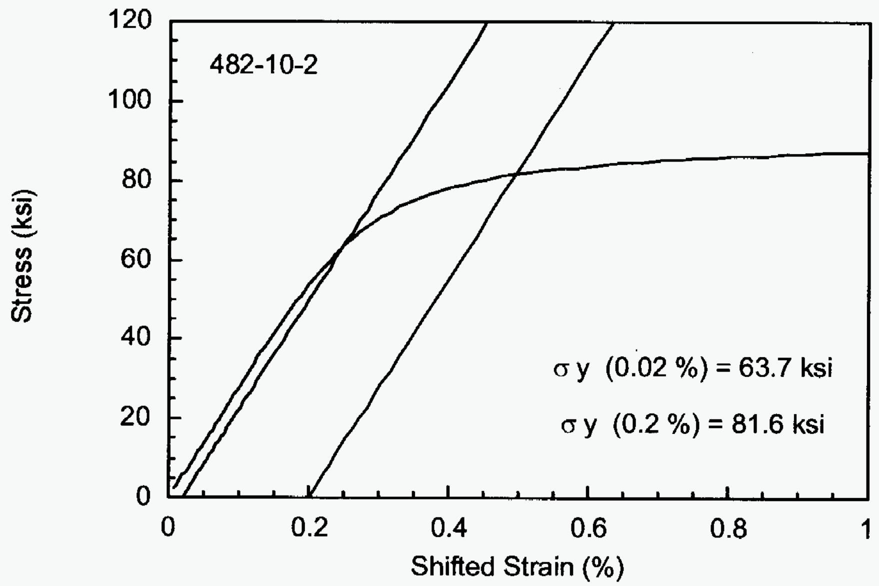

SA-3
Stress (ksi)
Strain (%)
σ UTS = 120.0 ksi
0
20
40
60
80
100
120
140
0
10
20
30
40
50
60
70

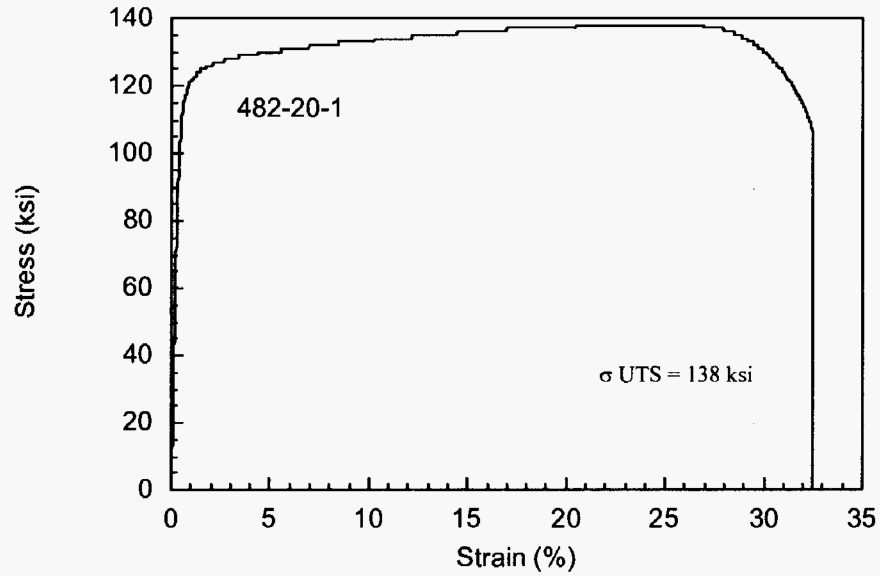

SA-3
Stress (ksi)
Shifted Strain (%)
σ y (0.02 %) = 41.7 ksi
σ y (0.2 %) = 47.8 ksi
0
10
20
30
40
50
60
0
0.2
0.4
0.6
0.8
1

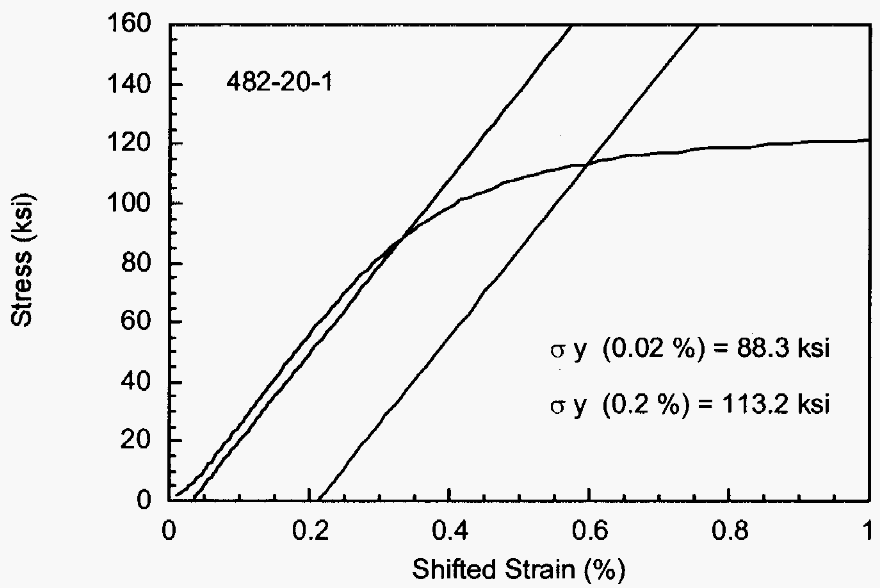

482-10-1
σ UTS = 120.0 ksi
Stress (ksi)
Strain (%)
0
20
40
60
80
100
120
140
0
10
20
30
40
50

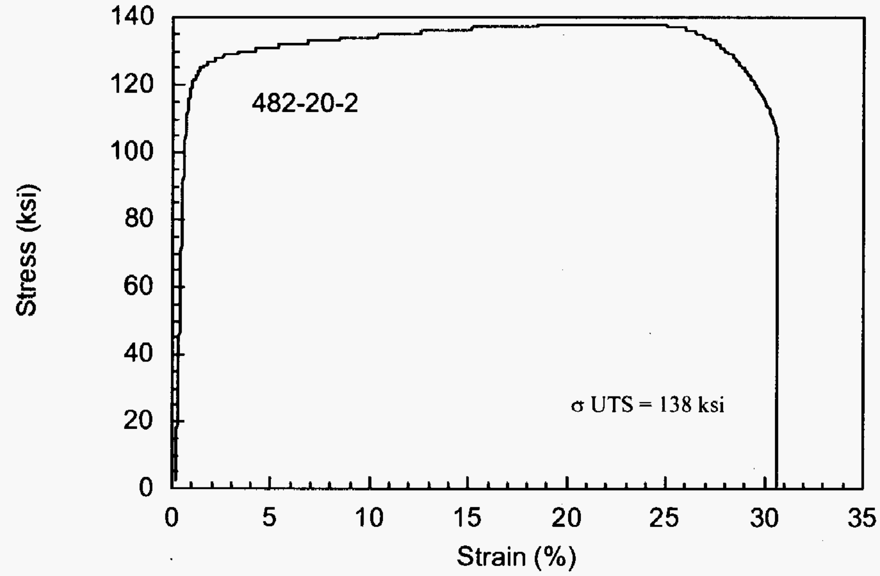

482-10-1
σ y (0.02%) = 65.2 ksi
σ y (0.2%) = 77.3 ksi
Stress (ksi)
Shifted Strain (%)
0
20
40
60
80
100
120
0
0.2
0.4
0.6
0.8
1

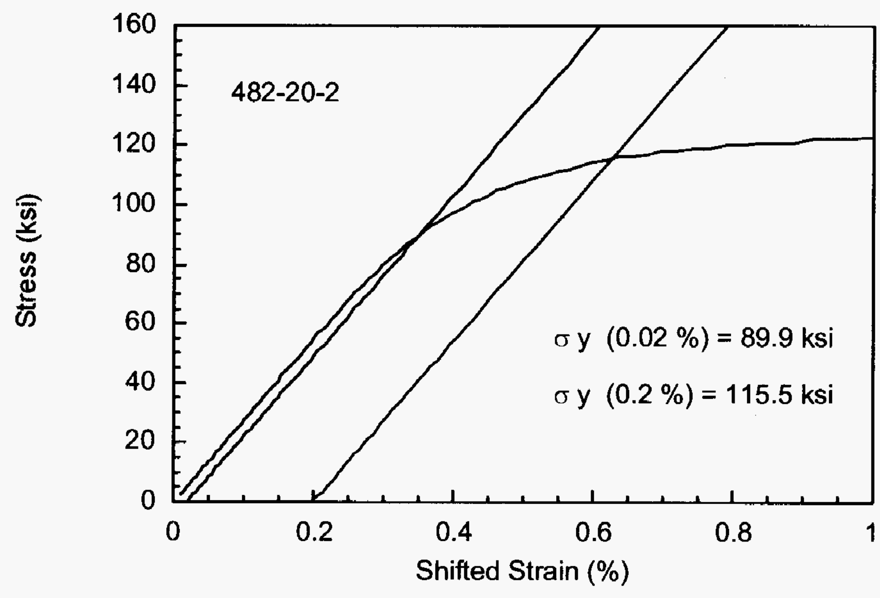

482-10-2
σ UTS = 123 ksi
Stress (ksi)
Strain (%)
0
20
40
60
80
100
120
140
10
30
50

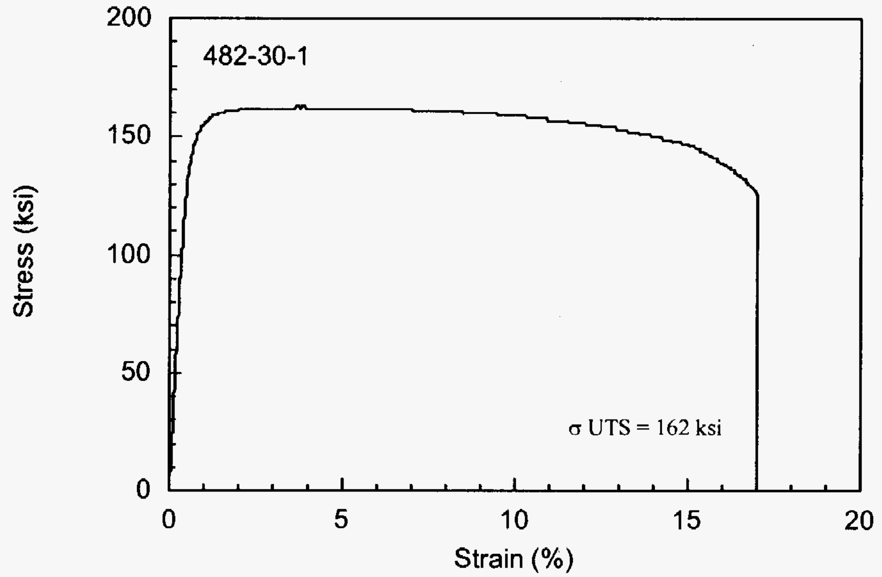

482-10-2
σ y (0.02 %) = 63.7 ksi
σ y (0.2 %) = 81.6 ksi
Stress (ksi)
Shifted Strain (%)
0
0.2
0.4
0.6
0.8
1
20
40
60
80
100
120

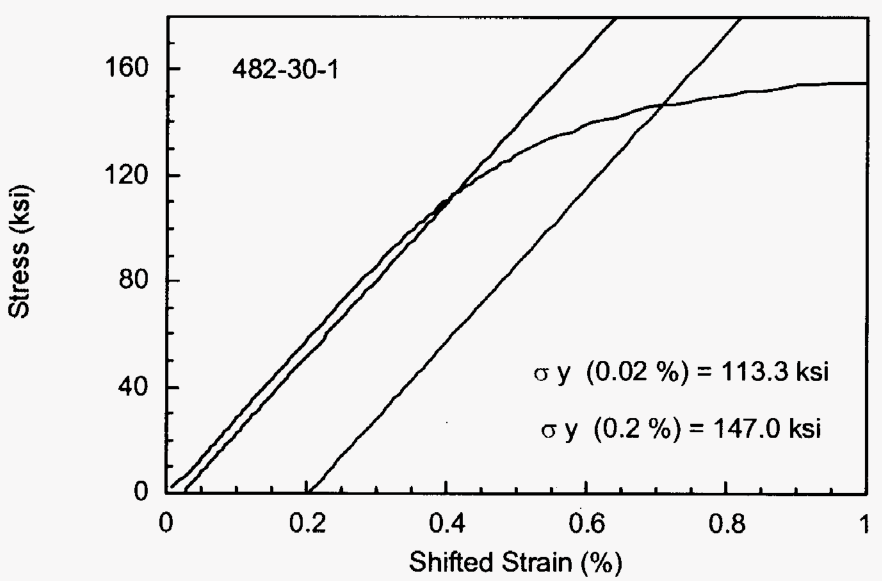

140
120
100
80
60
40
20
0
Stress (ksi)
482-20-1
σ UTS = 138 ksi
0
5
10
15
20
25
30
35
Strain (%)

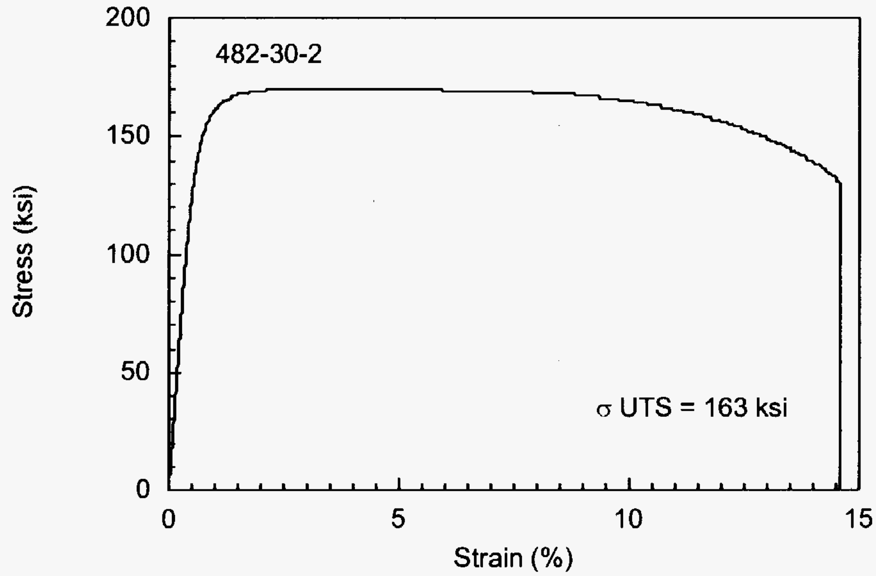

160
140
120
100
80
60
40
20
0
Stress (ksi)
482-20-1
σ y (0.02 %) = 88.3 ksi
σ y (0.2 %) = 113.2 ksi
0
0.2
0.4
0.6
0.8
1
Shifted Strain (%)


140
120
100
80
60
40
20
0
Stress (ksi)
482-20-2
σ UTS = 138 ksi
0
5
10
15
20
25
30
35
Strain (%)


160
140
120
100
80
60
40
20
0
Stress (ksi)
482-20-2
σ y (0.02 %) = 89.9 ksi
σ y (0.2 %) = 115.5 ksi
0
0.2
0.4
0.6
0.8
1
Shifted Strain (%)


482-30-1
Stress (ksi)
Strain (%)
σ UTS = 162 ksi
0
50
100
150
200
0
5
10
15
20


482-30-1
Stress (ksi)
Shifted Strain (%)
σ y (0.02 %) = 113.3 ksi
σ y (0.2 %) = 147.0 ksi
0
40
80
120
160
0
0.2
0.4
0.6
0.8
1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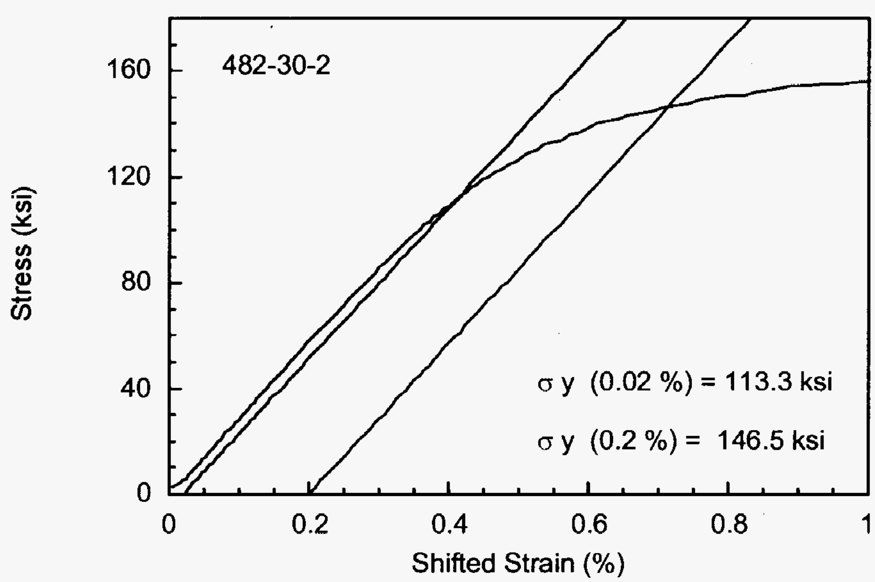
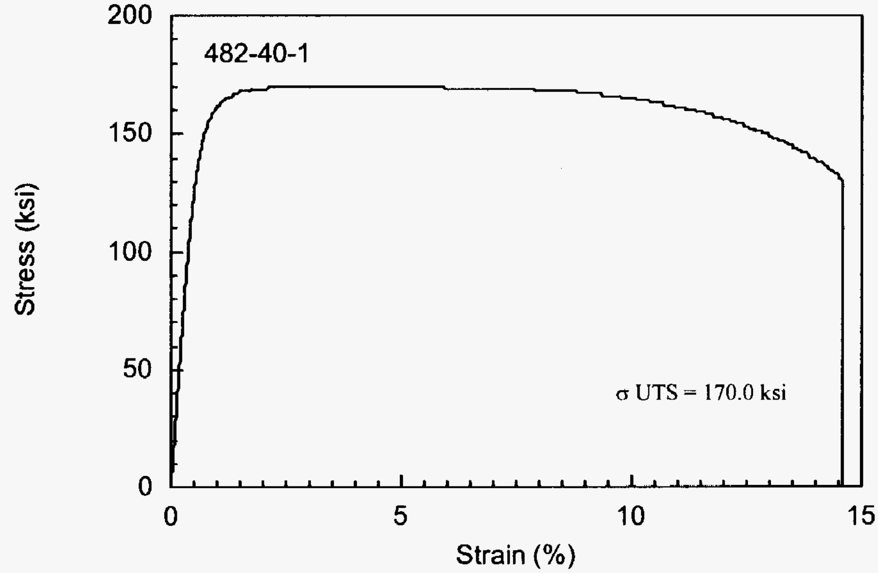
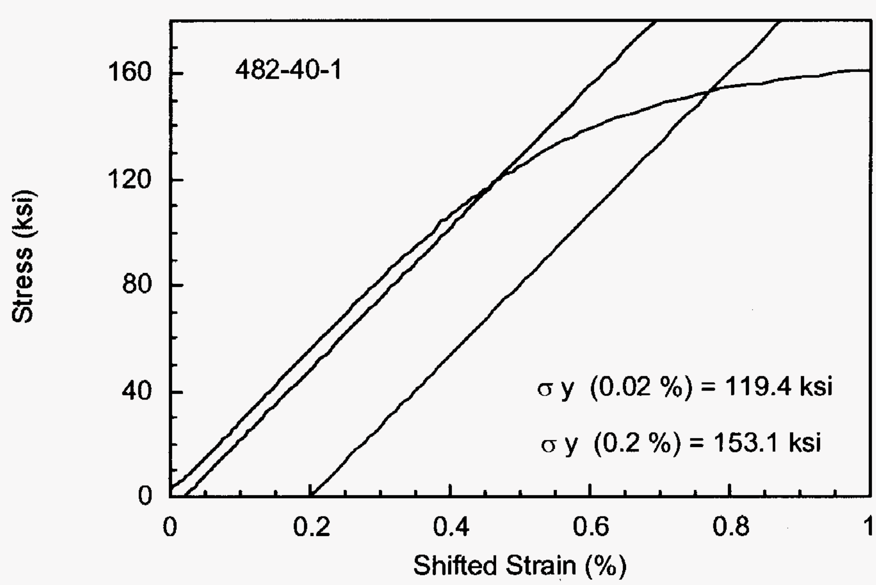
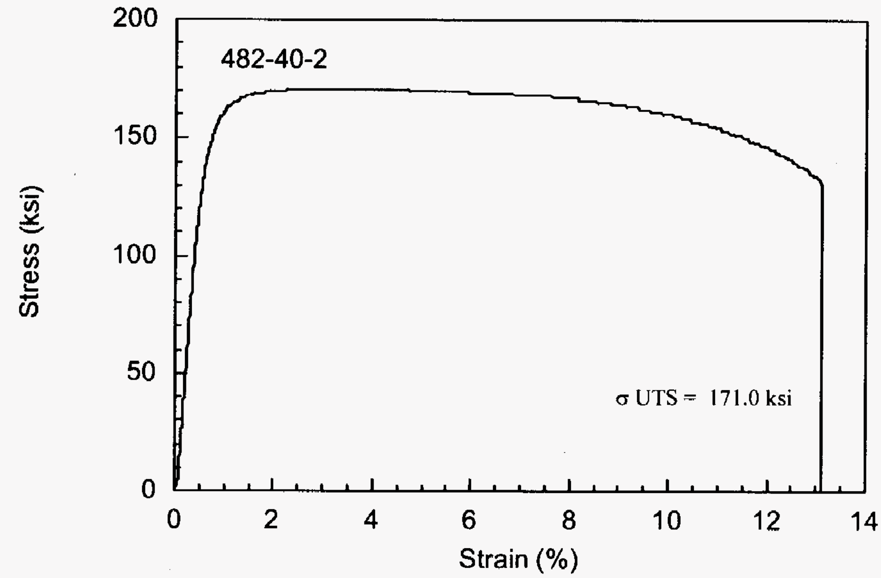
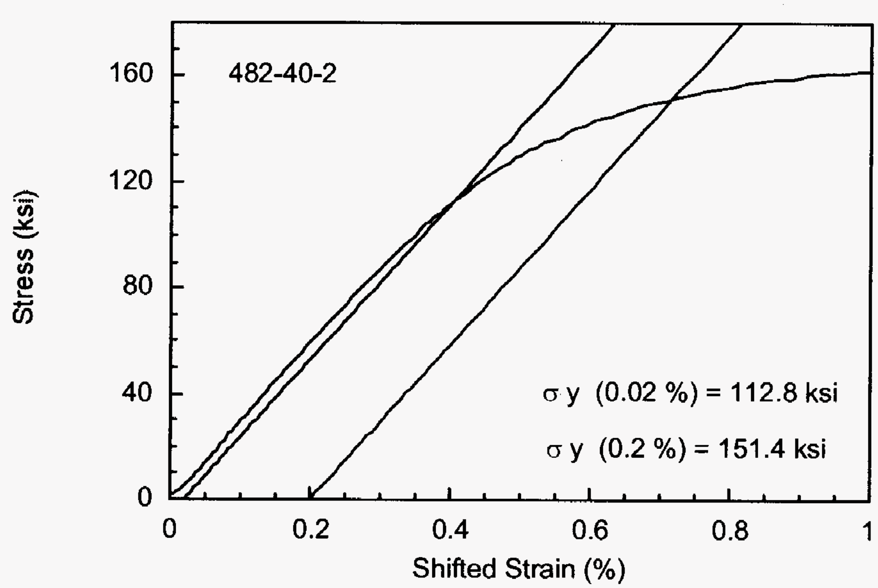
Stress (ksi)
Strain (%)
σ UTS = 163 ksi
200
150
100
50
0
0
5
10
15


482-30-2
Stress (ksi)
Shifted Strain (%)
σ y (0.02 %) = 113.3 ksi
σ y (0.2 %) = 146.5 ksi
160
120
80
40
0
0
0.2
0.4
0.6
0.8
1


482-40-1
Stress (ksi)
Strain (%)
σ UTS = 170.0 ksi
0
50
100
150
200
0
5
10
15


482-40-1
Stress (ksi)
Shifted Strain (%)
σ y (0.02 %) = 119.4 ksi
σ y (0.2 %) = 153.1 ksi
0
40
80
120
160
0
0.2
0.4
0.6
0.8
1


482-40-2
Stress (ksi)
Strain (%)
σ UTS = 171.0 ksi
200
150
100
50
0
0
2
4
6
8
10
12
14


482-40-2
Stress (ksi)
Shifted Strain (%)
σ y (0.02 %) = 112.8 ksi
σ y (0.2 %) = 151.4 ksi
160
120
80
40
0
0
0.2
0.4
0.6
0.8
1

APPENDIX 2 –SMC RESULTS FOR GTAW PROCESSED IN740 ALLOY WITH MATCHING COMPOSITIONFILLER FOR PRE-WELD AND POST-WELD CONDITIONS

Table A2-1. Chemical Analysis of INCONEL Alloy 740

Element	INCONEL Alloy 740 BLT2820B (Base material)	INCONEL Alloy 740 HV 0175 (Filler material)
C	.030	.043
Mn	.26	.28
Fe	.46	1.06
S	<.001	.002
Si	.51	.53
Cu	.02	.05
Ni	48.73	48.84
Cr	24.34	23.90
Al	.97	1.09
Ti	1.78	1.75
Mg	.007	.003
Co	19.8	19.93
Mo	.50	.51
Nb	1.99	1.92
P	<.005	.002
B	.003	.002
Ca	.002	.003
V	<.01	.013
W	<.05	.001
Zr	.025	.012

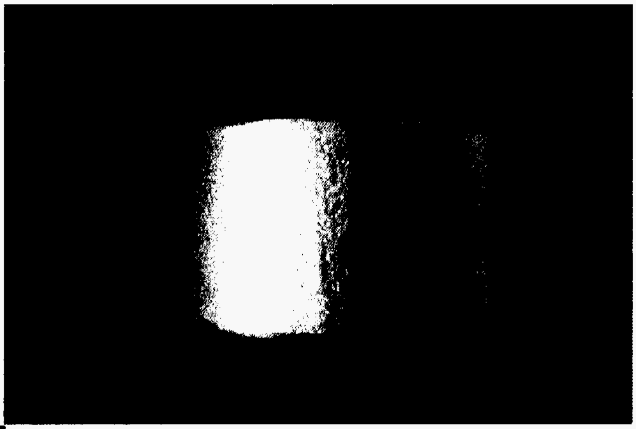


Figure A2-1. Photograph Illustrating Acceptable 4-T Face Bend.

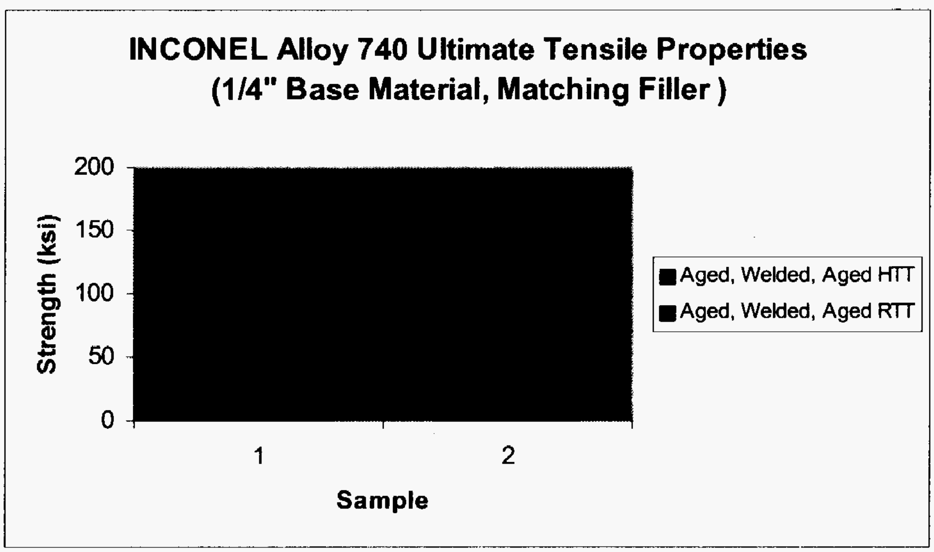


Figure A2-2. Ultimate Tensile Strength Values for GTAW INCONEL Alloy 740 Fabricated with INCONEL FILLER METAL 740 in the Aged Condition.

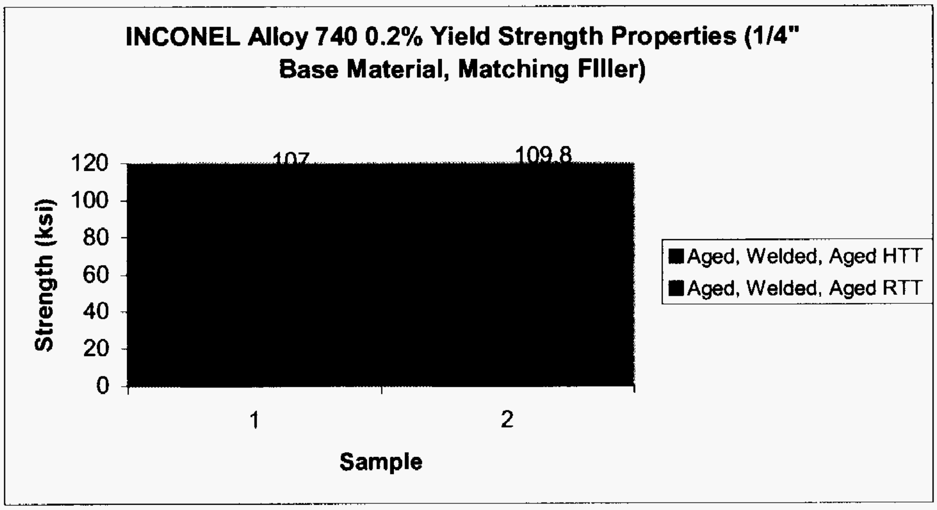


Figure A2-3. Yield Strength Values for GTAW INCONEL Alloy 740 Fabricated with INCONEL FILLER METAL 740 in the Aged Condition.

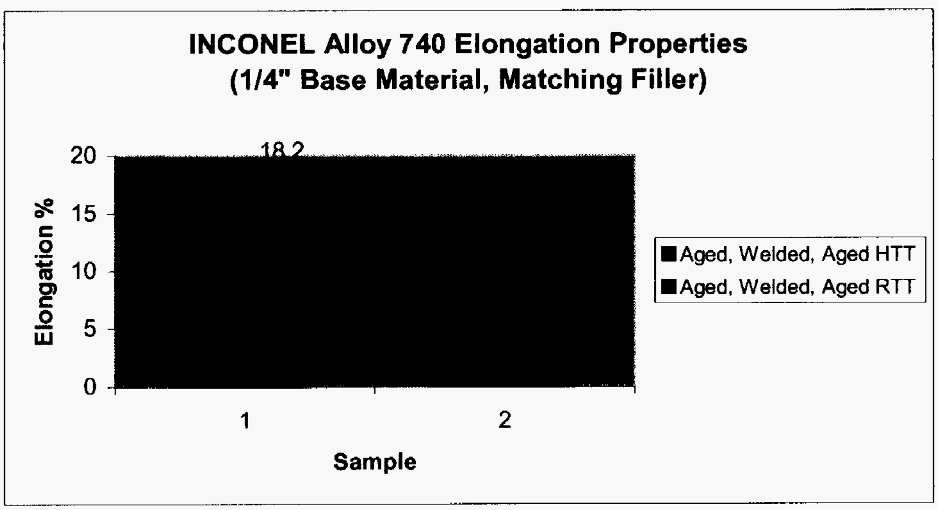


Figure A2-4. Elongation Values for GTAW INCONEL Alloy 740 Fabricated with INCONEL FILLER METAL 740 in the Aged Condition.

Figure A2-5 Microphotograph Illustrating the Fusion and Adjacent Heat Affect Zone Integrity of Gas Tungsten Arc Welded INCONEL Alloy 740 (aged condition) with a Matching Composition Filler Material. Etchant : Bromine Magnification: 50 X

Figure A2– 6 Microphotograph Illustrating the Fusion and Adjacent Heat Affect Zone Integrity of Gas Tungsten Arc Welded INCONEL Alloy 740 (aged condition) with a Matching Composition Filler Material. Etchant : Bromine; Magnification: 20 X

Figure A2– 7. Microphotograph Illustrating the Weld Metal Integrity of Gas Tungsten Arc Welded INCONEL Alloy 740 (aged condition) with a Matching Composition Filler Material. Etchant – Bromine; Magnification: 100 X

Table A2-2. Welding Parameters for Manual GTAW of INCONEL Alloy 740 with a Matching Composition Filler Material

Gas Tungsten Arc Welding (Manual)	
Parameter	**Value**
Power Supply	**Miller Dynasty DX 300**
Base Material	**INCONEL Alloy 740 (BLT2820B)**
Filler Material	**.093" Dia. INCONEL Filler Metal 740 (HV0175)**
Amperage	**185 Amp**
Voltage	**14Volt**
Shielding Gas/Flow Rate	**100% Argon @ 30 cfh**
Electrode	**2% Thoriated Tungsten 1/8" Dia.**

Table A2-3. Mechanical Test Results for Hign Temperature Test and Room Temperature Test Samples

Pre-weld Condition	Post-weld Condition	Test Temp.	Ultimate Tensile ksi	0.2% Yield ksi	Elongation %	Failure Location
Aged*	Aged*	1382° F	110.2	90.3	5.2	WM
Aged*	Aged*	1382° F	99	94.5	4.9	WM
Aged*	Aged*	RT	159.5	107	18.2	WM
Aged*	Aged*	RT	156.5	109.8	14.8	WM

* 1472°F/4hr/AC