Project No.
Book No.

TITLE P-Phase Solvus Temperature Calculations in the Alloy 22 Weld of Cieslak

From Page No.

obj.: To calculate the P-Phase solvus temperature in the Alloy22 weld as a result of element segregation

Software: THERMOCALC Version N
Database: Thermotech Ni-Data Version 5

Results:

Calculated P-Phase Solvus Temperatures Using the Compositions in the Alloy 22 Weld*

Location	Chemical Content (wt%)					P-Phase Solvus Temperature (°C)
	Ni	Cr	Mo	Fe	W	
Dendrite Core	62.2	19.6	10.9	3.13	2.75	872
Interdendritic Region	52.4	21.1	18.8	2.90	3.43	1,319
Bulk Composition	57.0	21.2	13.4	3.17	3.29	1,067

*The compositions are abstracted from Figure 7 in M.J. Cieslak et al., The Welding Metallurgy of Hastelloy Alloys C-4, C-22, and C-276, Metallurgical Transactions, Vol. 17A, pp. 2035-2047, 1986.

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 1/29/03
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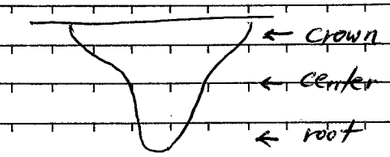
Project No.
Book No.

TITLE SEM-EDS Spot Analysis in the Fusion Zone of the DOE Weld

From Page No.

specimen: DOE weld under As-Welded condition

Locations of analysis (see morphology of the fusion zone on page 5)



Chemical Compositions in the Weld Fusion Zone (in wt%)

Dendrite Core Location	Ni	Cr	Mo	Fe	W
1-Crown1	60.89	21.43	12.34	2.77	2.58
2-Crown2	60.72	21.56	12.1	2.93	2.68
3-Center1	57.73	22.33	14.8	2.68	2.46
4-Center2	60.41	21.68	12.53	2.71	2.67
5-Root1	59.29	22.2	13.22	2.68	2.61
6-Root2	62.2	20.61	11.93	2.81	2.45

Interdendritic Region	Ni	Cr	Mo	Fe	W
1-Crown1	53.81	22.58	18.74	2.48	2.4
2-Crown2	52.12	22.89	20.05	2.37	2.57
3-Center1	56.17	22.64	16.18	2.56	2.45
4-Center2	54.1	22.75	18.18	2.46	2.51
5-Root1	55.86	22.36	16.78	2.53	2.47
6-Root2	59.02	21.59	14.35	2.68	2.36

	Ni	Cr	Mo	Fe	W
Mean	59.81	21.84	13.00	2.75	2.60
Std Dev	1.32	0.40	1.09	0.11	0.09
Minimum	57.73	21.43	12.10	2.68	2.46
Maximum	60.89	22.33	14.80	2.93	2.68
Count	5	5	5	5	5

Note: Exclude location 6 from the statistical analysis

	Ni	Cr	Mo	Fe	W
Mean	54.41	22.64	17.99	2.48	2.48
Std Dev	1.65	0.20	1.55	0.07	0.06
Minimum	52.12	22.36	16.18	2.37	2.40
Maximum	56.17	22.89	20.05	2.56	2.57
Count	5	5	5	5	5

Note: Exclude location 6 from the statistical analysis

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 3/26/03
Recorded by

Project No.
Book No.
TITLE P-Phase Solvus Temperature Calculations in the DOE Alloy 22 Weld

From Page No.

Software: THERMOCALC Version N
Database: Thermotech Ni-Data version 5

Results:

Location	Chemical Content (wt%)					P-Phase Solvus Temperature (°C)
	Ni	Cr	Mo	Fe	W	
Dendrite Core	59.8±1.3	21.8±0.4	13.0±1.1	2.75±0.11	2.60±0.09	1,024
Interdendritic Region	54.4±1.6	22.6±0.2	18.0±1.6	2.48±0.07	2.48±0.06	1,271
Alloy 22 Base Metal	60.64	20.44	12.80	2.63	3.08	1,026
Alloy 622 Filler Metal	59.89	20.41	13.99	2.46	2.96	1,066

To Page No.

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Project No.
Book No.
TITLE Precipitate Volume Fraction Measurements for the DOE Welds after Various Aging Treatments

From Page No.

1. The specimens were etched in a solution of 5g oxalic acid in 95 ml of 37% HCl solution at 6 V for a very short period so that removal of precipitates during etching can be minimized.

2. Measurements were conducted along the centerline of fusion zone for all aging treatments. Additional analysis was carried out through lengthwise of the 760°C/6hr specimen. Amount of precipitates was determined through multiple measurements using imaging analysis software loaded in an optical microscope

3. Results:

DOE	as welded	
image	% Precip.	% Matrix
M714	0.7	99.3
M715	0.8	99.2
M716	0.3	99.7
M717	0.2	99.8
M718	0.2	99.8
M719	0.3	99.7
M720	0.1	99.9
M721	0.3	99.7
M722	0.3	99.7
M723	0.4	99.6
M724	0.2	99.8
M725	0.7	99.3
M726	0.6	99.4
M727	0.9	99.1
M728	0.1	99.9
M729	0.3	99.7
M730	0.4	99.6
M731	0.4	99.6
M732	0.3	99.7
M733	0.6	99.4
M734	0.9	99.1
M735	0.2	99.8
M736	0.5	99.5

Summary Statistics	
Mean	0.42
Standard Deviation	0.24
Minimum	0.10
Maximum	0.90
Sum	9.70
Count	23.00

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by [signature] | 3/28/03

Project No.
Book No. TITLE

From Page No.

760C/6hrs (weld centerline)

image	% Precip.	% Matrix
M422	5.4	94.6
M423	4.7	95.3
M424	3.9	96.1
M425	3.3	96.7
M426	3.5	96.5
M427	4.5	95.5
M428	4.2	95.8
M429	4.1	95.9
M430	4.2	95.8
M431	3.6	96.4
M432	4.4	95.6
M433	4.4	95.6
M434	5.6	94.4
M435	5	95
M436	5.1	94.9
M437	5	95
M438	4.4	95.6

Summary Statistics	
Mean	4.43
Standard Deviation	0.65
Minimum	3.30
Maximum	5.60
Sum	75.30
Count	17.00

760C/60hrs (weld centerline)

image	% Precip.	% Matrix
M439	2.9	97.1
M440	1.8	98.2
M441	1.8	98.2
M442	3.8	96.2
M443	2.5	97.5
M444	2.3	97.7
M445	3.5	96.5
M446	1.8	98.2
M447	2.6	97.4

Summary Statistics	
Mean	2.56
Standard Deviation	0.74
Minimum	1.80
Maximum	3.80
Sum	23.00
Count	9.00

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by [illegible] | 3/28/03

TITLE
Project No.
Book No.

From Page No.

760C/6hrs (lengthwise)

image	% Precip.	% Matrix	
M448	1.6	98.4	
M449	1.7	98.3	
M450	1.2	98.8	
M451	1.3	98.7	
M452	1.5	98.5	
M453	1.2	98.8	
M454	1.3	98.7	
M455	1.3	98.7	
M456	1.2	98.8	
M457	3.8	96.2	weld
M458	2.9	97.1	weld
M459	3.5	96.5	weld
M460	3.8	96.2	weld
M461	3.6	96.4	weld
M462	3.6	96.4	weld
M463	4.6	95.4	weld
M464	4.4	95.6	weld
M465	3.4	96.6	weld
M466	4	96	weld
M467	4.6	95.4	weld
M468	3.5	96.5	weld
M469	3.9	96.1	weld
M470	3.8	96.2	weld
M471	2.4	97.6	weld
~~M472~~	~~4~~	~~96~~	
M473	1.8	98.2	
M474	2.3	97.7	
M475	2.1	97.9	
M476	1.9	98.1	
M477	1.5	98.5	
M478	1.5	98.5	
M479	1.5	98.5	
M480	1.4	98.6	
M482	1.5	98.5	
M483	1.1	98.9	
M484	1.3	98.7	
M485	1.5	98.5	
M486	0.2	99.8	
M487	1.6	98.4	
M488	0.5	99.5	

* excluded from the statistical analysis
mp 3/28/03

*Summary Statistics (Outside Weld)**	
Mean	1.42
Standard Deviation	0.44
Minimum	0.20
Maximum	2.30
Sum	34.00
Count	24.00

Summary Statistics (Weld)	
Mean	3.72
Standard Deviation	0.58
Minimum	2.40
Maximum	4.60
Sum	55.80
Count	15.00

To Page No.

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Recorded by [illegible] | 3/28/03

From Page No.

870C/5min (weld centerline)

image	% Precip.	% Matrix
M691	1.3	98.7
M692	1.3	98.7
M693	1.1	98.9
M694	1.4	98.6
M695	1	99
M696	1	99
M697	1.4	98.6
M698	1.3	98.7
M699	1	99
M700	1.3	98.8
M701	0.9	99.1
M702	1.2	98.8
M703	0.9	99.1
M704	1	99
M705	1.1	98.9
M706	1	99
M707	1.3	98.7
M708	1.4	98.6
M709	1.1	98.9
M710	1.2	98.8
M711	1	99
M712	1.1	98.9
M713	1.2	98.8

Summary Statistics	
Mean	1.15
Standard Deviation	0.16
Minimum	0.90
Maximum	1.40
Sum	26.50
Count	23.00

870C/30min (weld centerline)

image	% Precip.	% Matrix
M370	1.7	98.3
M371	5.8	94.2
M372	4.8	95.2
M373	3.6	96.4
M374	3.9	96.1
M375	3.7	96.3
M376	3.7	96.3
M377	4	96
M378	5.4	94.6
M379	3.9	96.1
M380	3.9	96.1
M381	3.4	96.6
M382	3.3	96.7
M383	3.6	96.4
M384	5.1	94.9
M385	4	96
M386	5.2	94.8
M387	3.9	96.1

Summary Statistics	
Mean	4.05
Standard Deviation	0.94
Minimum	1.70
Maximum	5.80
Sum	72.90
Count	18.00

No.

Witnessed & Understood by me, | Date | Invented by | Date 3/28/03
Recorded by

From Page No.

870C/1hr (weld centerline)

image	% Precip.	% Matrix
M388	8.1	91.9
M389	6.9	93.1
M390	7.4	92.6
M391	9	91
M392	5.5	94.5
M393	8.4	91.6
M394	7.6	92.4
M395	10.4	89.6
M396	8	92
M397	8.3	91.7
M398	11.3	88.7
M399	7.5	92.5
M400	8.4	91.6
M401	9.7	90.3
M402	8.2	91.8
M403	8.2	91.8
M404	4.7	95.3

Summary Statistics	
Mean	8.09
Standard Deviation	1.58
Minimum	4.70
Maximum	11.30
Sum	137.60
Count	17.00

870C/4hrs (weld centerline)

image	% Precip.	% Matrix
M405	12.9	87.1
M406	10.4	89.6
M407	9.9	90.1
M408	11.7	88.3
M409	12	88
M410	8.8	91.2
M411	11	89
M412	11.6	88.4
M413	9.1	90.9
M414	11.7	88.3
M415	12.3	87.7
M416	12.7	87.3
M417	8.5	91.5
M418	11.7	88.3
M419	11.5	88.5
M420	10.5	89.5
M421	8.4	91.6

Summary Statistics	
Mean	10.86
Standard Deviation	1.46
Minimum	8.40
Maximum	12.90
Sum	184.70
Count	17.00

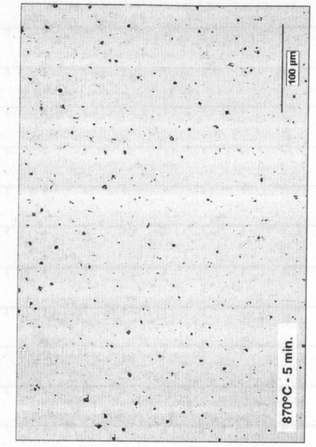
To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 3/28/03
Recorded by

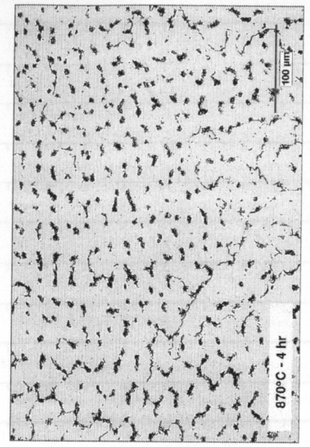
Project No.
Book No.
TITLE

From Page No.

4. Representative microstructure for various aging treatments



870 °C/5 min



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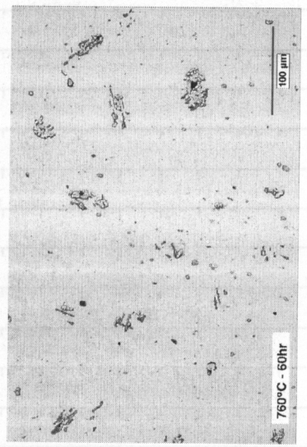

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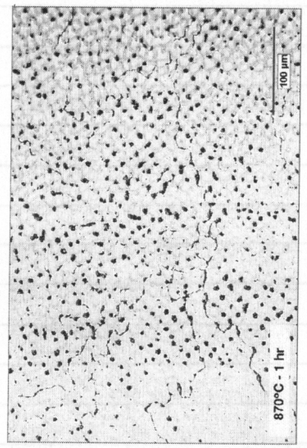
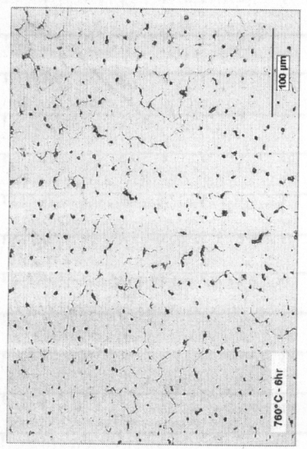
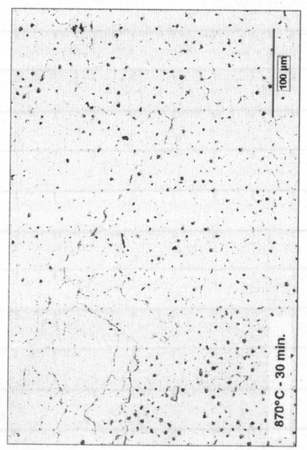


870 °C/1 hr



760 °C/6 hr



870 °C/30 min

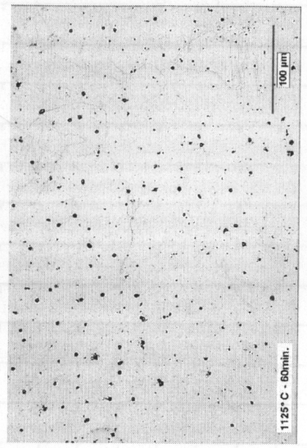
To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by [signature]	3/28/03

Project No.
Book No.
TITLE Microstructure of the DOE welds after various Solution Annealing Treatments

From Page No.

Representative photographs showing residual precipitates in the Alloy 22 welds



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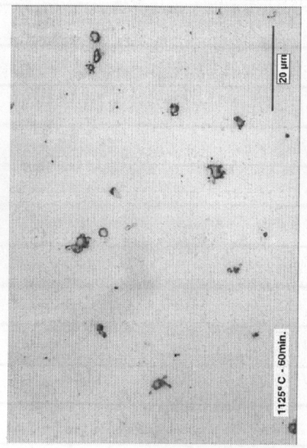

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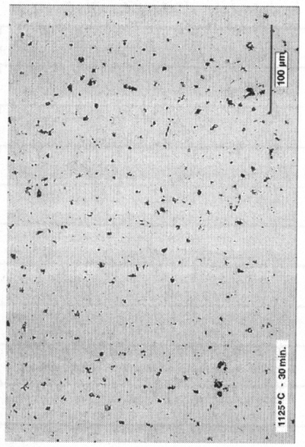

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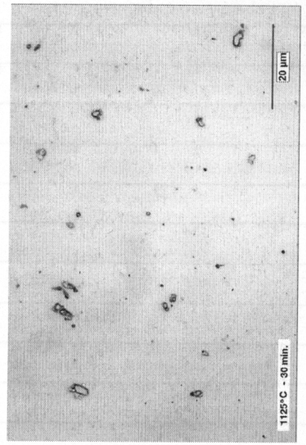

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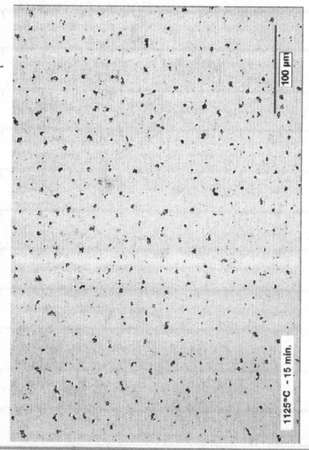

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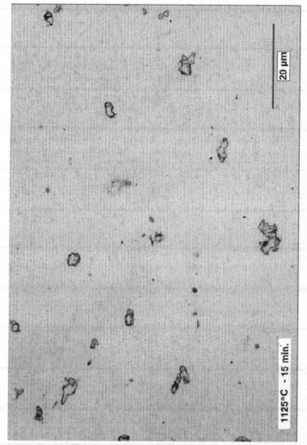

1125 °C/15 min

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To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by [signature]	4/1/03

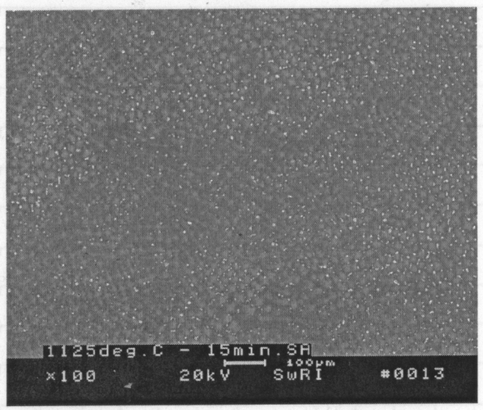
Project No.
Book No.
TITLE Influence of Solution Annealing Temperature on the Microstructure of the DOE welds

From Page No.

Specimens: sections from the DOE welded plate from Framatome ANP, Inc. (Alloy 22 Heat 059902LL2)

Solution heat treatments:
temperatures — 1200, 1250, and 1300 °C
duration — 15 minutes for all temperatures

Results:
1. SEM photographs showing microstructural changes in Alloy 22 welds



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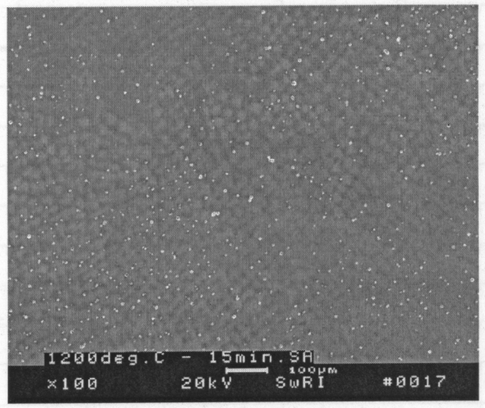
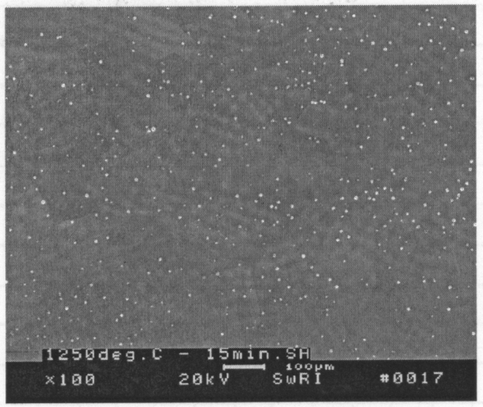
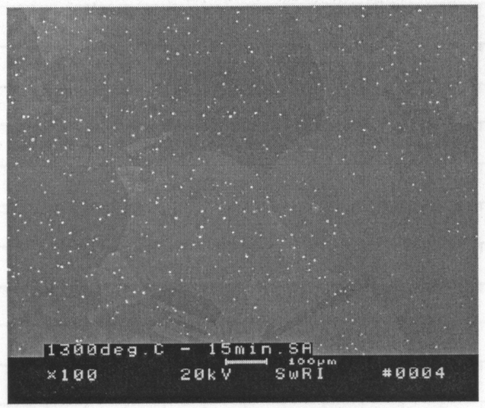


1200 °C/15 min



1250 °C/15 min



1300 °C/15 min

To Page No.

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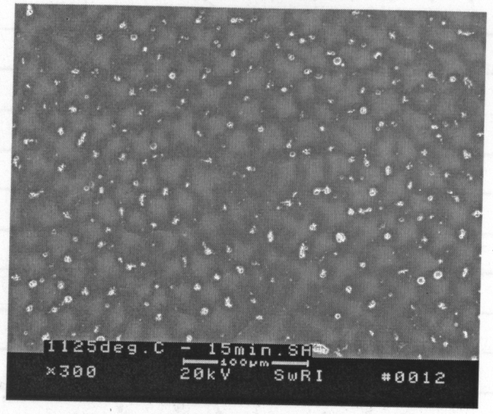
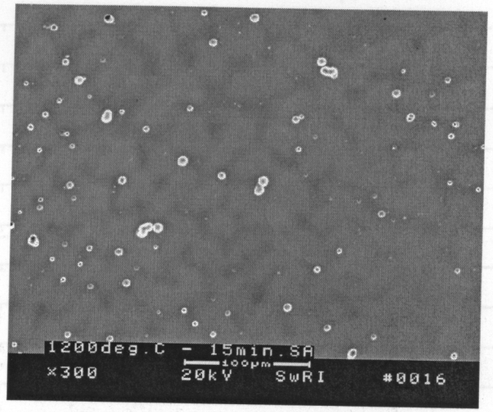
TITLE
Project No.
Book No.

From Page No.

2. Enlarged SEM photographs showing the detailed microstructures in the fusion zone



1125 °C/15 min



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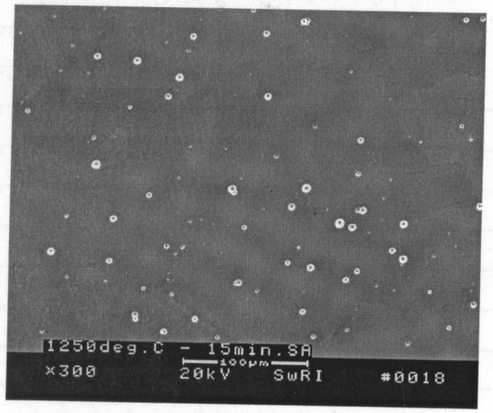

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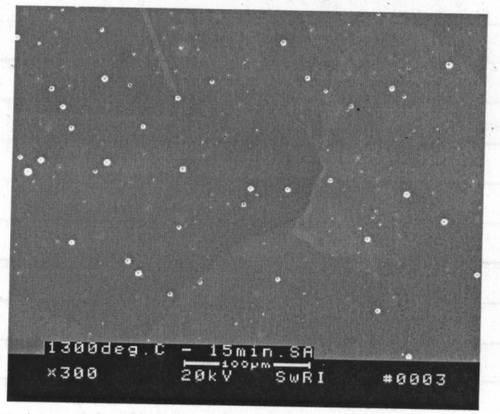


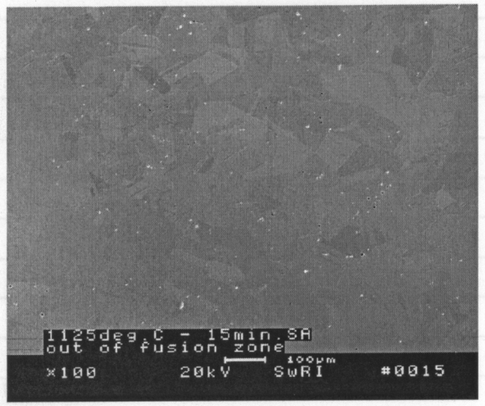
1300 °C/15 min

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by Zi Pan | 4/30/03

From Page No.

3. SEM photographs showing microstructural changes outside the fusion zone



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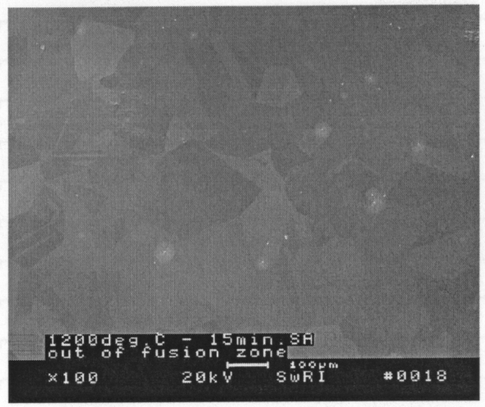
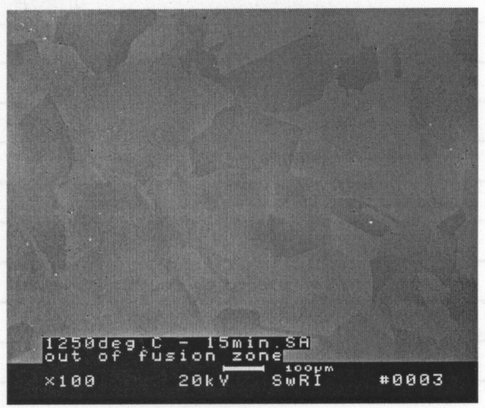
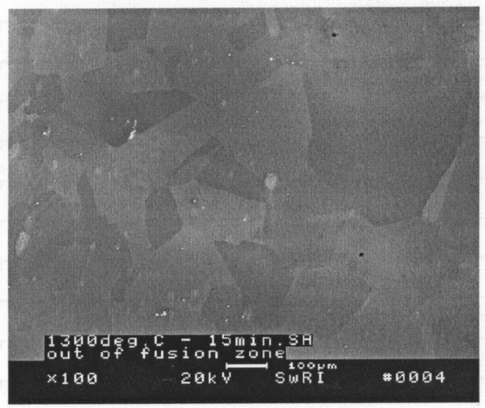


1200 °C/15 min



1250 °C/15 min



1300 °C/15 min

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Zi Pan	4/30/03

From Page No.

See the detailed procedures of the measurements recorded on page 55

1125C ~~1120~~/15min (weld centerline) Zip 5/1/03

image	% Precip.	% Matrix
M737	2	98
M738	1.3	98.7
M739	2	98
M740	2.8	97.2
M741	1.7	98.3
M742	1.7	98.3
M743	1.3	98.7
M744	1.5	98.5
M745	1.4	98.6
M746	1.5	98.5
M747	1.7	98.3
M748	1.4	98.6
M749	1.5	98.5
M750	1.6	98.6
M751	2.2	97.8
M752	1.6	98.4
M753	1.8	98.2
M755	1.4	98.6
M756	2.3	97.7
M757	1.6	98.4
M758	2.5	97.5
M759	2.1	97.9
M760	1.7	98.3

Summary Statistics	
Mean	1.77
Standard Deviation	0.40
Minimum	1.30
Maximum	2.80
Sum	40.60
Count	23

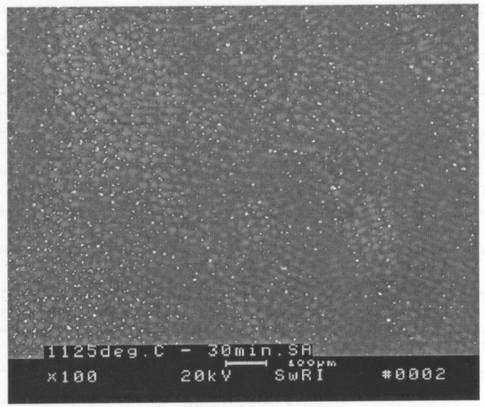
To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Zi Pan	5/1/03

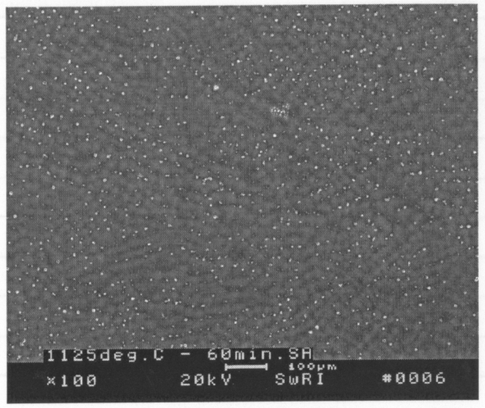
Project No.
Book No.
TITLE Microstructure of the DOE welds after Solution Annealing at 1125°C for 30 and 60min.

From Page No.

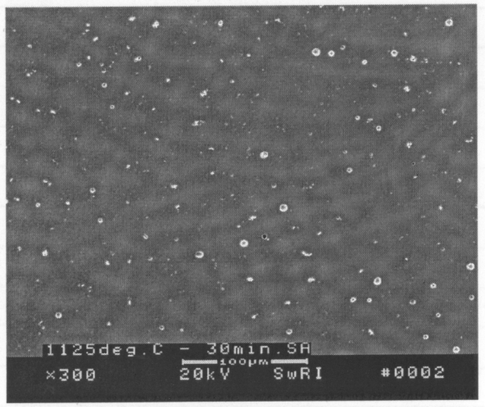
SEM photographs showing microstructural changes in Alloy 22 welds (Compare the photographs with those for 1125°C/15min on pages 62 & 63).



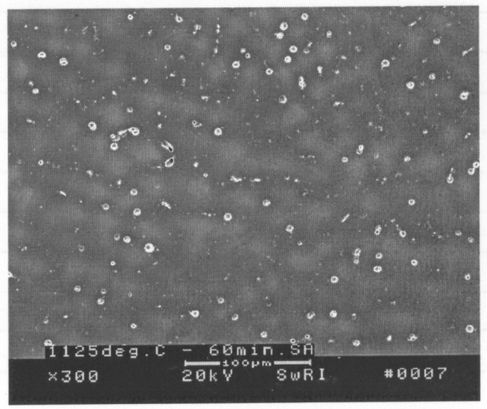
1125 °C/30 min



1125 °C/60 min



1125 °C/30 min



1125 °C/60 min

To Page No.

Witnessed & Understood by me, Date ~~5/7/03~~ 5/7/03 Invented by Recorded by Date 5/7/03

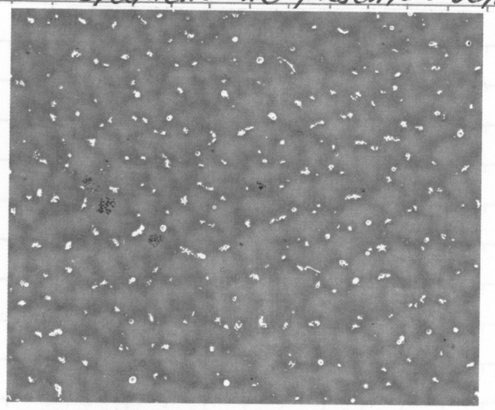
TITLE Element Dot Maps on the DOE welds after Various Solution Annealing Treatments

Project No.
Book No.

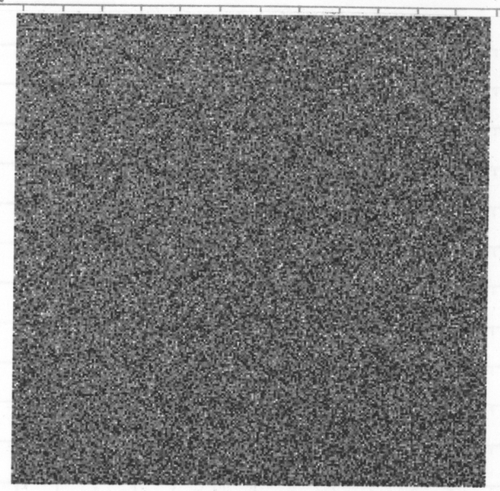
From Page No.

SEM element mapping on the DOE welded material solution annealed at various temperatures (1125, 1200, 1250, and 1300°C) for 15 minutes was performed to determine the chemistry in the fusion zone. The microstructural changes in the fusion zone are not discernible in the Ni, Cr, and Mo maps for all solution annealing treatments.

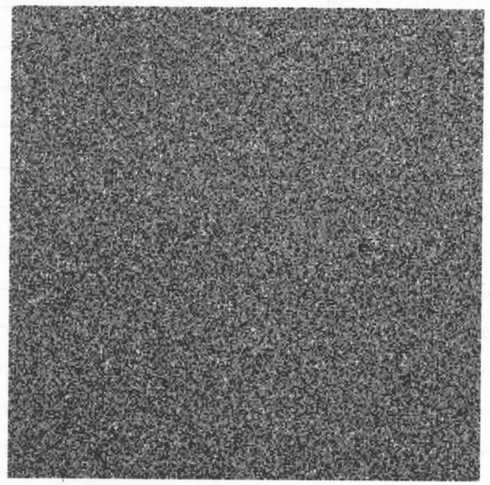
Only the element maps recorded in the 1125°C/15min and 1300°C/15min specimens are presented below.



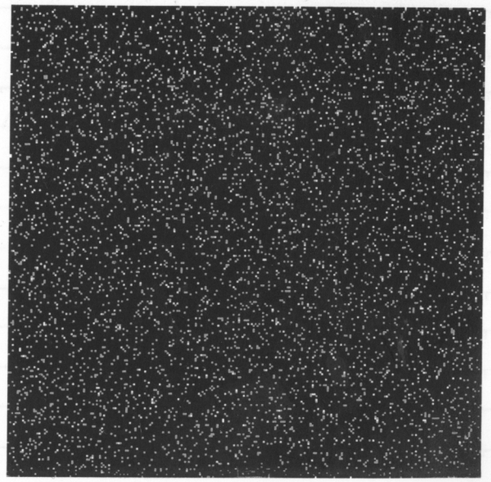
1125°C/15min



Ni Map



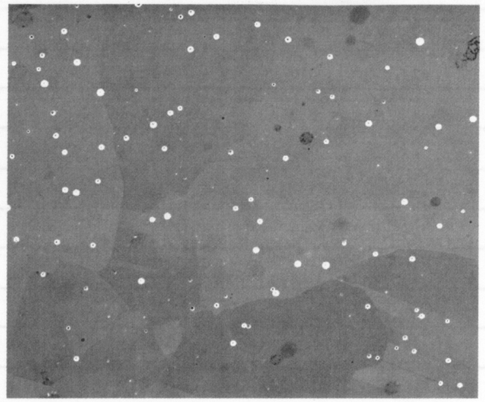
Cr Map



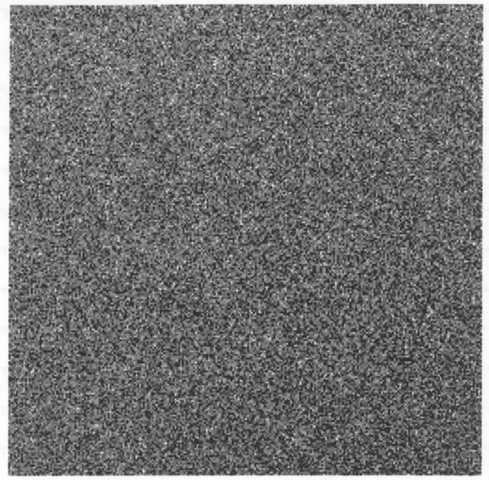
Mo Map

Witnessed & Understood by me, Date Invented by Recorded by Date 5/14/03

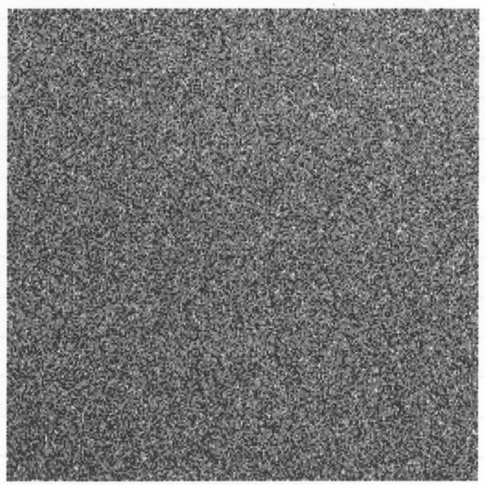
From Page No.



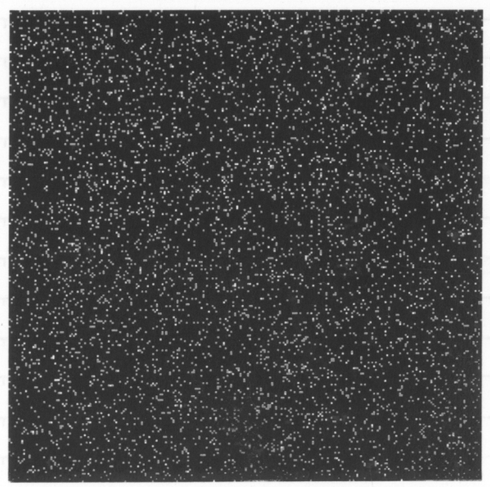
1300°C/15min



Ni Map



Cr Map



Mo Map

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Yi-Ming Pan	5/14/03

From Page No.

Scientific notebook initial entry for passive film surface analysis of Alloy 22

Title: Passive film surface analysis of Alloy 22

Tests Performed by: Yi-Ming Pan and Darrell Dunn

Objectives: Evaluate changes in the oxide composition and structure as a function of the applied potential, time, and temperature of electrochemical treatments

Equipments: X-ray photoelectron spectroscopy and Auger electron spectroscopy

Materials: Alloy 22 specimens under various electrochemical treatment conditions

Measurement Parameters: thickness, composition, and chemical state of the oxide films

Uncertainty and Sources of Error: The presence of oxide film that formed after air exposure may influence characterization of the passive film arising from electrochemical treatments.

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Yi-Ming Pan	7/18/03

Project No.
Book No.
TITLE Alloy 22 specimens for Surface Analysis

From Page No.

Requisition: 03002082
Requisitioner: Dunn, Darrell S.
Req Organization: 1.20.05.04
Phone: (210) 522-6090

SOUTHWEST RESEARCH INSTITUTE

Suggested Supplier: Texas Toolmakers
City/State: San Antonio, TX
Contact: Mike Ridgway
Phone: 494-3651 Fax: 494-6139

Page 1 of 1
Date Printed: 08/05/2003
Requisition Date: 08/05/2003

Line #	Item / Description	U/M	Need By Date	Requested Qty	Est Unit Cost	Estimated Costs
1	XPS test specimens CNWRA drawing 20-06002-0	EA	8/13/2003	10.00	85.85	858.50

1-081-005 machined from Alloy 22 plate heat 2277-8-3175. Attached quote and CNWRA drawing 20-06002-01-081-005
Deliver To: Darrell Dunn/bldg. 57
Account: 704-000 Organization: 1.20 Project: 06002.01.081 Allocation Pct: 100.00
Total Estimated Cost: $858.50

Special Instructions: Quality & Technical Requirements: Dimensional inspection per dimensions and tolerances identified in CNWRA drawing
20-06002-081-005 is required.

Government Project?: YES Property Type: G1 Is Govt. Property being sent to supplier?: YES

Quality Assurance?: YES ASL Required: YES

SUPPLIER SHALL FURNISH DIMENSIONAL INSPECTION DATA VERIFYING COMPLIANCE WITH THE REQUIREMENTS OF THE FABRICATION DRAWINGS.

Requisition for XPS specimens from
Alloy 22 heat 2277-8-3175
Drawing Page 71
Documentation from Texas Toolmakers P 72 & 73

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by Darrell Dunn 8/17/03

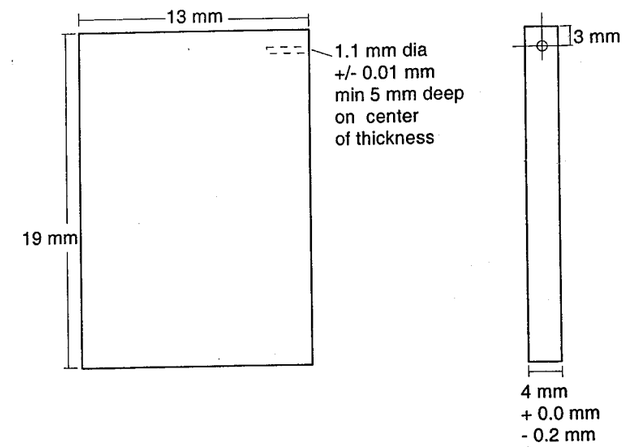
TITLE
Project No.
Book No.

From Page No.

Darrell S. Dunn
SwRI-CNWRA
Phone: (210) 522-6090
Fax: (210) 522-5184
e-mail: ddunn@swri.org

XPS Specimen
CNWRA Drawing 20-06002-01-081-005
Dimensional tolerances 0.5 mm
unless otherwise specified

To be completed at time of order:
Material: Alloy C-22
Heat: 2277-8-3175
Specimen Orientation: N/A
Other:



Darrell Dunn 8/4/03
Initiated by. D. Dunn Date

V. Jain 8/4/03
Reviewed by V. Jain Date

R D Brient 8/5/03
QA Approval B. Brient Date

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by Darrell Dunn 8/17/03