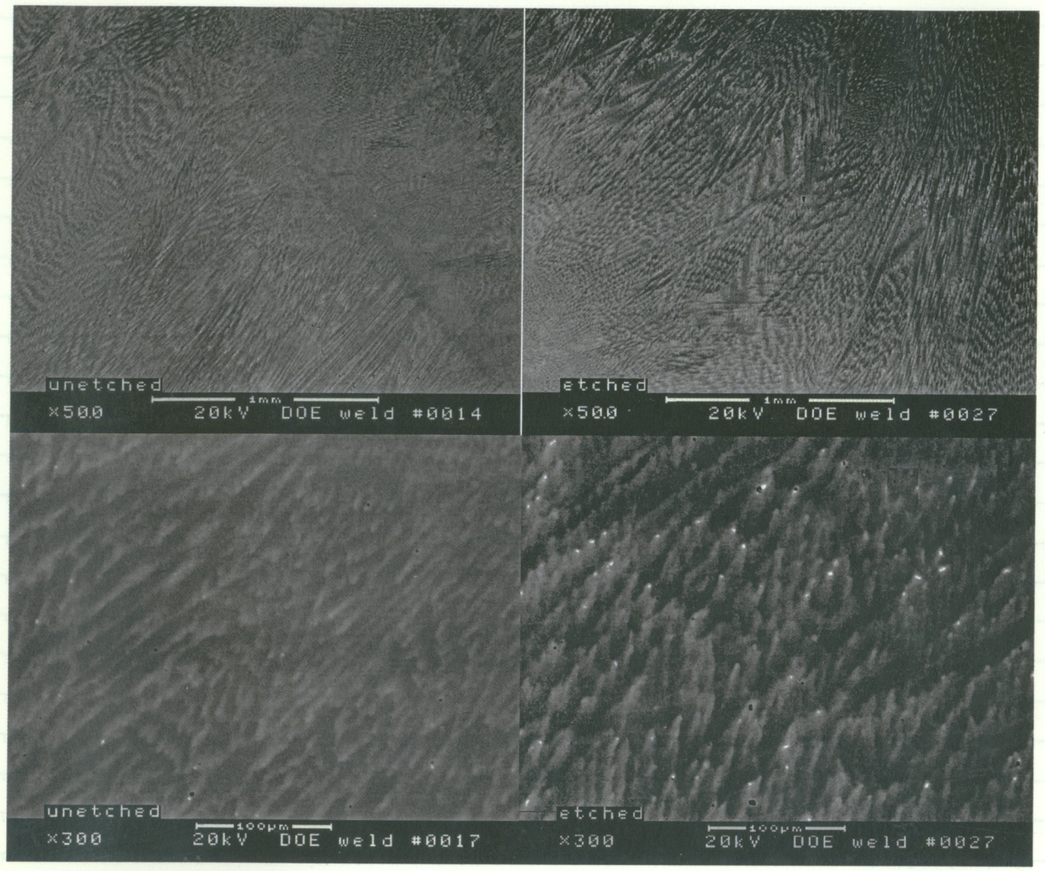
From Page No.

1. SEM micrographs of the DOE welded Alloy 22 (Heat 05990 2LL02)

Fusion zone

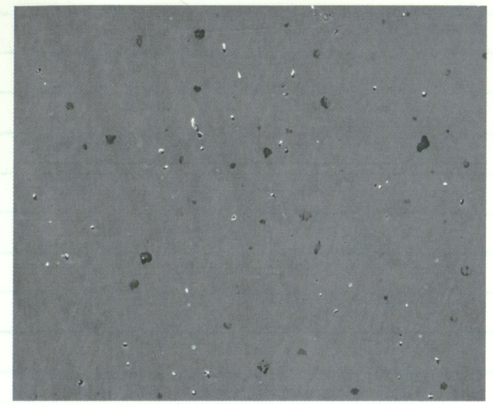


To Page No.

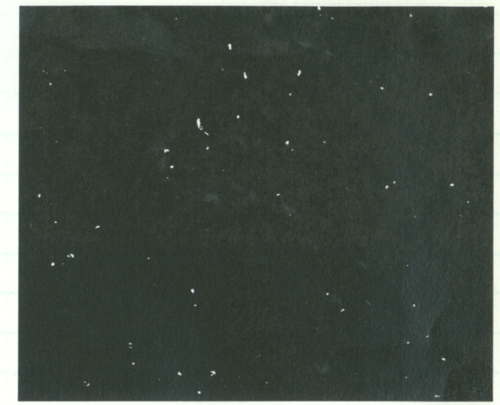
Witnessed & Understood by me, | Date | Invented by | Date 3/18/02
Recorded by

From Page No.

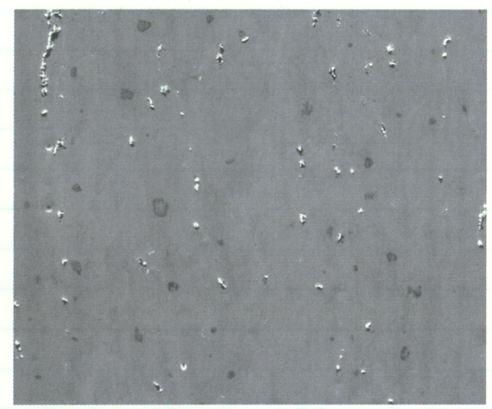
2. Representative precipitate distribution in the fusion zone of the DOE welded Alloy 22



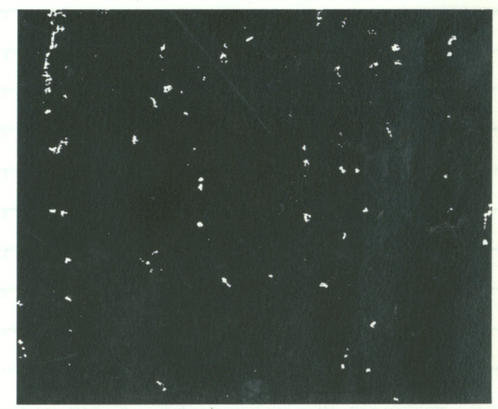
Frame 15 - SED image 500x



Binary image 500x



Frame 36 - SED image 500x



Binary image 500x

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 3/19/02
Recorded by

Project No.
Book No.
TITLE

From Page No.

3. Volume fraction of precipitate measurements by image analysis
(40 frames are taken equally spaced from top to center of the weld fusion zone that cover about half of the path.)

Frame No	% Precipitates
1	0.16
2	0.10
3	0.20
4	0.27
5	0.03
6	0.35
7	0.12
8	0.02
9	0.30
10	0.13
11	0.04
12	0.28
13	0.30
14	0.04
15	0.17
16	0.23
17	0.37
18	0.07
19	0.14
20	0.15
21	0.16
22	0.16
23	0.06
24	0.38
25	0.14
26	0.21
27	0.10
28	0.09
29	0.04
30	0.12
31	0.26
32	0.04
33	0.19
34	0.10
35	0.27
36	0.79
37	0.16
38	0.03
39	0.01
40	0.13

Summary Statistics	
Mean	0.17
Standard Deviation	0.14
Minimum	0.01
Maximum	0.79
Sum	6.91
Count	40

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by | 3/19/02

TITLE Intergranular Corrosion Test of the As-weld Alloy 22 Fusion Zone
Project No.
Book No.

From Page No.

Intergranular Corrosion Test

Specimen: welded Alloy 22 AW Heat 2277-8-3235
specimen cut from weld (fusion zone)

Solution: ASTM G-28 using Test method B for 24 hrs.

specimen
start wt: 13.76427g Sartorious Genius SN# 12809099 cal 12/21/01
End wt: 13.75921g

Dimensions: .96 x .54 In²
.54 x .18 In²
.18 x .96 In² Fowler Calipers SN# 20-8C-1 cal 12/31/01

Solution = 476 mls DI H_2O
10.032g $FeCl_3 \cdot 6H_2O$ Lot# 005437
7.201g $CuCl_2 \cdot 2H_2O$ Lot# 897713
90 mls H_2SO_4 (96.3%) Lot# 990715
18 mls HCl (37.6%) Lot# 002545

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by | 3/21/02

Project No.
Book No.
TITLE

From Page No.

Test Results

Corrosion Rate Calculation After ASTM G28B Testing

	Fusion Zone
K	8.76E+04
W(g)	0.00506
A(cm^2)	10.17
T(h)	24
D(g/cm^3)	8.69
R(mm/y)	0.2089
R(mpy)	8.2280
Depth(um)	0.57
Test Surface Inspection	Two Pitting Areas, Attack in Interdentritic Regions

R(mm/y)=(K*W)/(A*T*D)
R(mpy)=R(mm/y)*(3.45E6/8.76E4)
Depth(um)=(W*10000)/(A*D)

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To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 3/22/02
Recorded by [illegible]

Project No.
Book No.
TITLE Scanning Transmission Electron Microscopy Analysis of Alloy 22 at ASU

From Page No.

STEM analysis of Alloy 22 was performed at the Center for Solid State Science, Arizona State University. The objective of this investigation is to determine the overall microstructure and grain boundary chemistry of Alloy 22 specimens under mill-annealed, welded, and heat-treated conditions.

1. Microscope : Philips CM 200 STEM, 200kV

2. Alloy 22 specimens
   A. Heat 2277-8-3175
      specimen #1 – mill-annealed condition
      specimen #3 – heat-treated at 870°C / 5 min
      specimen #4 – heat-treated at 870°C / 30 min
   B. Heat 05990ZLL2
      specimen #2 – as-welded plate from Allegheny Ludlum (DOE)

3. Thin foil preparation
   3-mm-diameter cylinders were cut out from the bulk samples using EDM, followed by cutting slices about 500-μm thick from the cylinders. The 3-mm disks were polished from both sides to about 100μm, dimpled down to 10μm, and then ion milled until a small perforation was detected.

4. STEM analysis
   The microscope was operated at 200 kV. All images were recorded as digital files using Digital Micrograph system. For x-ray microanalyses (EDS) a 2nm electron beam diameter was used. Elemental concentrations were calculated using the known bulk chemical composition of mill-annealed Alloy 22 as an internal standard, according to the Cliff-Lorimer procedure.

ref: J. I. Goldstein and D. B. Williams; Quantitative Microanalysis with High Spatial Resolution, The Metals Society, London, 1981, pp. 5-14.

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 4/25/02
Recorded by [illegible]

From Page No.

5. Results

A. Specimen #1 – Mill-annealed condition

EDS spot analysis for k factor calculations

Mill-Annealed	Ni	Mo	Cr	W	Fe
Bulk(wt%)	57.80	13.60	21.40	3.00	3.80
Matrix1					
I	31491	6185	13466	1171	2264
C(rat)	1.00	0.24	0.37	0.05	0.07
C(wt%)	57.80	13.60	21.40	3.00	3.80
k1	1.00	1.20	0.87	1.40	0.91
Matrix2					
I	37067	7024	15994	1389	2615
C(rat)	1.00	0.24	0.37	0.05	0.07
C(wt%)	57.80	13.60	21.40	3.00	3.80
k2	1.00	1.24	0.86	1.39	0.93
Matrix3*					
I	33499	6144	14382	1207	2499
C(rat)	1.00	0.24	0.37	0.05	0.07
C(wt%)	57.80	13.60	21.40	3.00	3.80
k3	1.00	1.28	0.86	1.44	0.88
k(ave)	1.00	1.24	0.86	1.41	0.91

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date

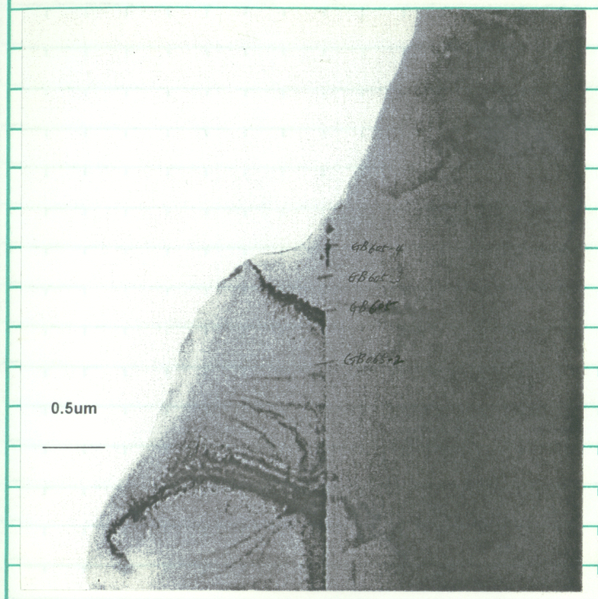
Recorded by M. Pan | 5/7/02

From Page No.

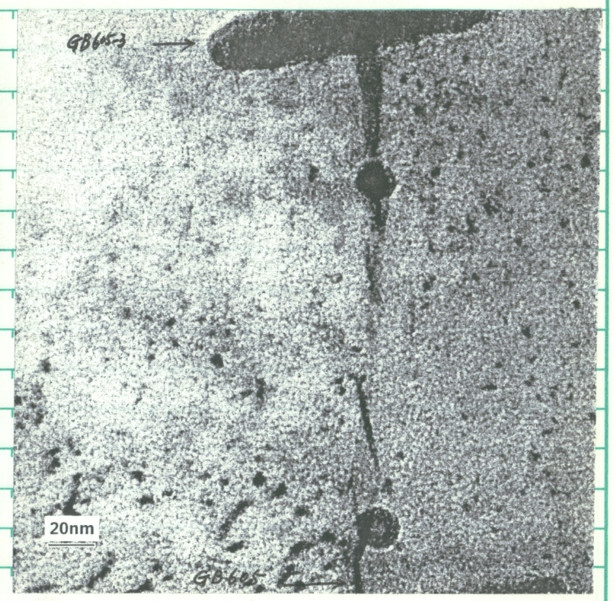
B. Specimen #3 – 870°C/5min

a. micrographs of a grain boundary region with EDS line scan positions

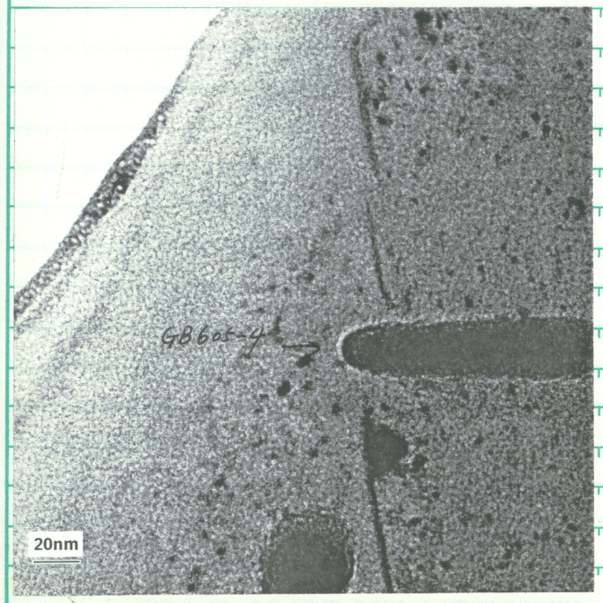
#3gb6-4000



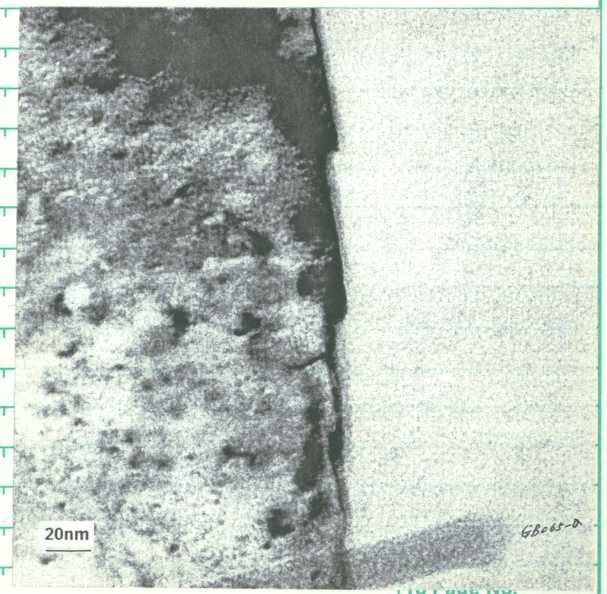
#3gb6-2-72000



#3gb6-6-72000



#3gb6-4-72000



To Page No.

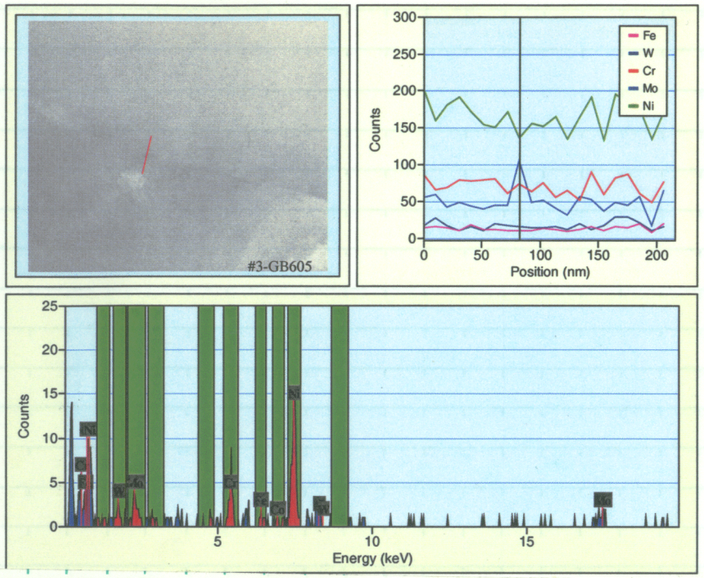
Witnessed & Understood by me, | Date | Invented by | Date

Recorded by M. Pan | 5/7/02

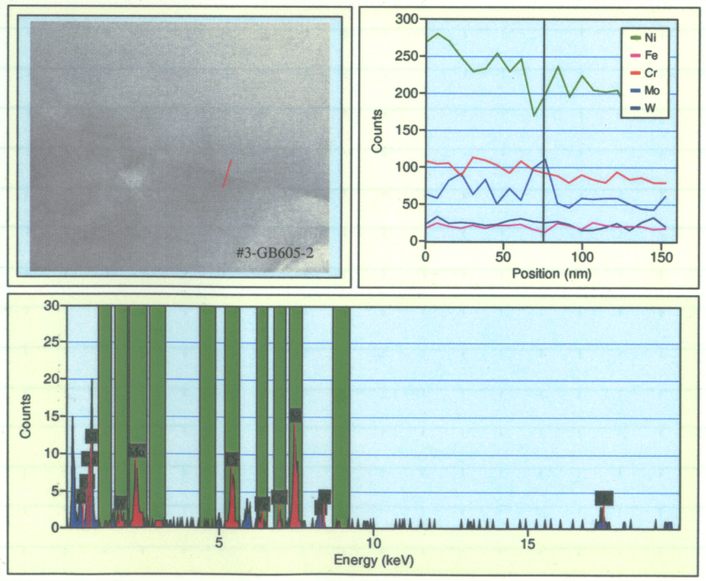
From Page No.

B.6 EDS line scan to show the presence of nanostructured Mo-rich films at grain boundaries. (see micrographs on page 29)

#3-GB605 Across GB Precipitate Position 1



#3-GB605-2 Across GB precipitate Position 2

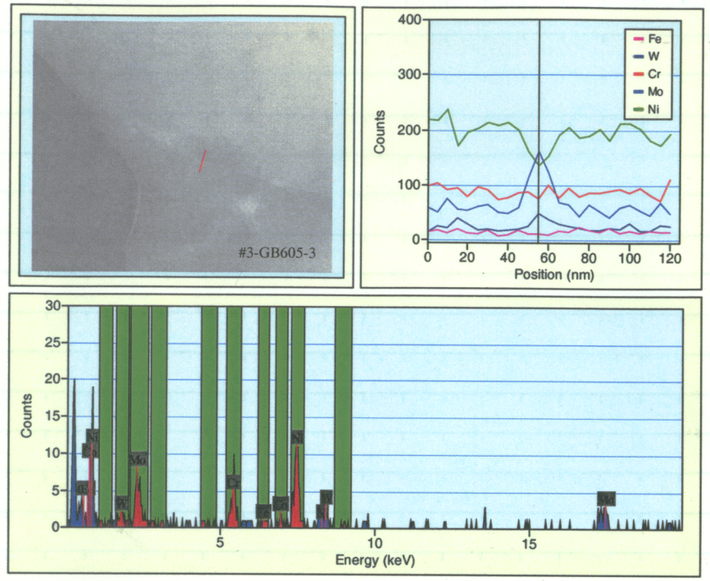


To Page No.

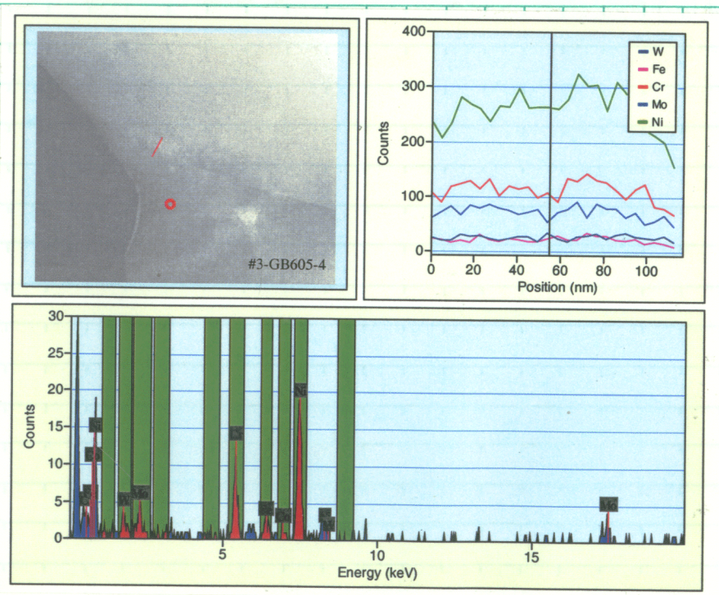
Witnessed & Understood by me, | Date | Invented by | Date
Recorded by 5/7/02

From Page No.

#3-GB605-3 Across GB Precipitate Position 3



#3-GB605-4 Across GB without precipitate



To Page No.

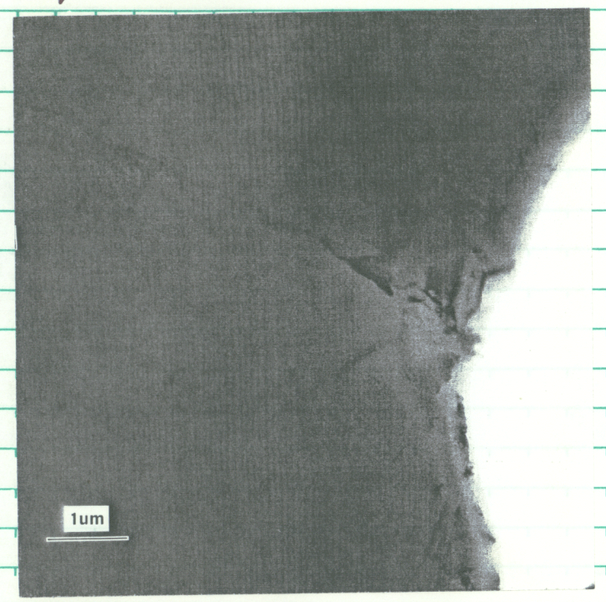
Witnessed & Understood by me, | Date | Invented by | Date
Recorded by 5/7/02

From Page No.____

C. Specimen #4 – 870°C/30min

a. micrographs of a grain boundary region (GB3)

#4gb3-01-2550



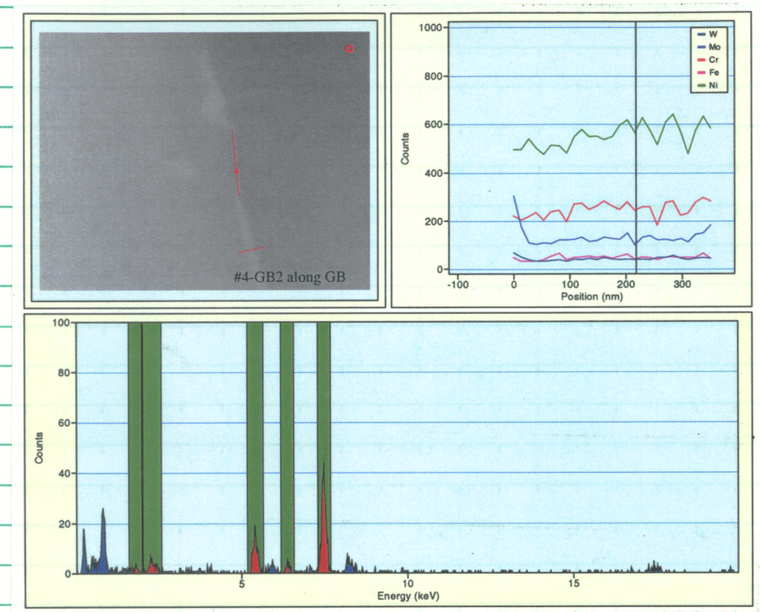
#4gb3-03-20000

To Page No._____

From Page No.____

b. EDS line scan along grain boundary and across precipitate.
No measurable depletion of chromium and molybdenum was detected in the matrix adjacent to the precipitate nor in the grain boundary region between precipitates.

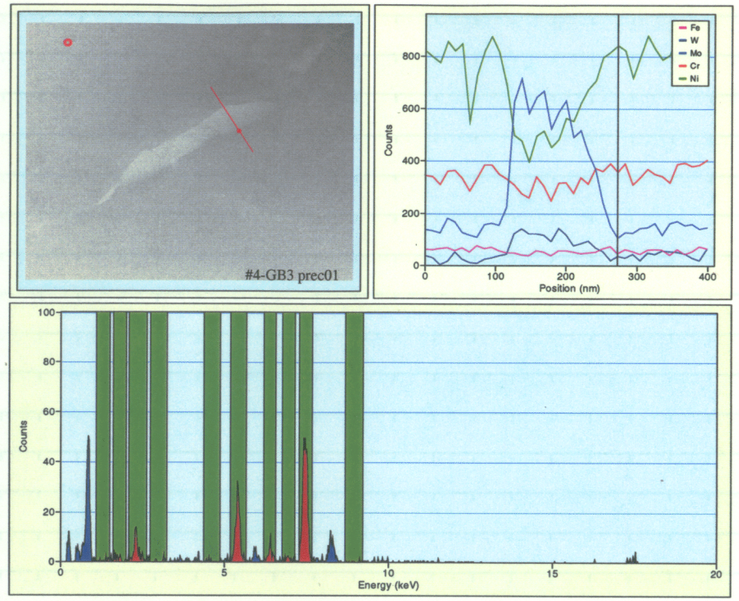
Along Grain Boundary



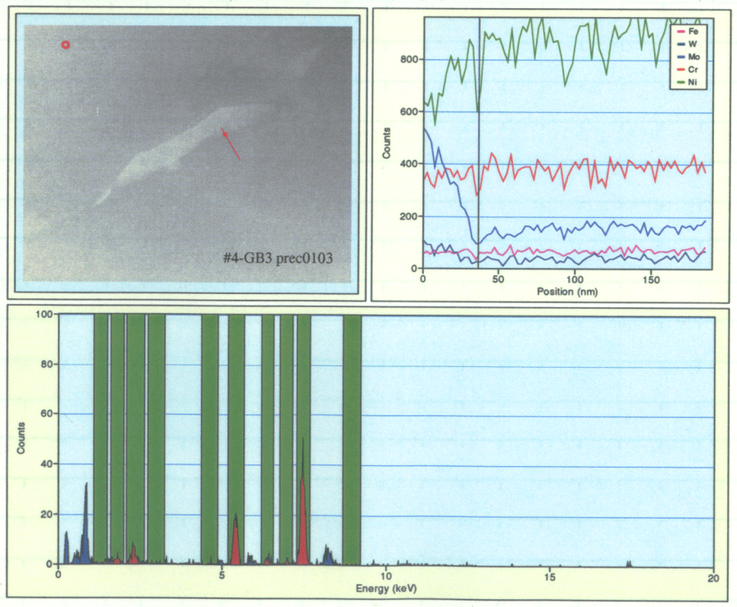
To Page No._____

From Page No.

Across Precipitate



Adjacent to precipitate



To Page No.

From Page No.

C1. EDS spot analysis of GB2

870C/30min-GB2	Ni	Mo	Cr	W	Fe
Bulk(wt%)	57.80	13.60	21.40	3.00	3.80
Matrix1					
I	32671	5935	14476	1087	2332
C(rat)	1.00	0.24	0.37	0.05	0.07
C(wt%)	57.80	13.60	21.40	3.00	3.80
k1	1.00	1.30	0.84	1.56	0.92
Matrix2					
I	32509	5912	14111	1124	2317
C(rat)	1.00	0.24	0.37	0.05	0.07
C(wt%)	57.80	13.60	21.40	3.00	3.80
k2	1.00	1.29	0.85	1.50	0.92
Matrix3					
I	31273	5752	13623	985	2191
C(rat)	1.00	0.24	0.37	0.05	0.07
C(wt%)	57.80	13.60	21.40	3.00	3.80
k3	1.00	1.28	0.85	1.65	0.94
k(ave)	1.00	1.29	0.85	1.57	0.93
GB1					
I	8394	1424	3621	268	652
C(rat)	1.00	0.22	0.37	0.05	0.07
C(wt%)	58.62	12.82	21.40	2.94	4.22
GB2					
I	8273	1453	3662	181	648
C(rat)	1.00	0.23	0.37	0.03	0.07
C(wt%)	58.55	13.26	21.93	2.01	4.25
PPT1					
I	7886	12227	8462	1970	771
C(rat)	1.00	2.00	0.91	0.39	0.09
C(wt%)	22.78	45.54	20.68	8.93	2.07
PPT2					
I	8051	9826	5222	1628	810
C(rat)	1.00	1.57	0.55	0.32	0.09
C(wt%)	28.30	44.54	15.53	8.98	2.64
PPT3					
I	7928	8839	6812	1339	681
C(rat)	1.00	1.44	0.73	0.27	0.08
C(wt%)	28.49	40.97	20.72	7.55	2.27

To Page No.

Project No.
Book No.
TITLE

From Page No.

C2. EDS spot analysis of GB3

870C/30min-GB3	Ni	Mo	Cr	W	Fe
Bulk(wt%)	57.80	13.60	21.40	3.00	3.80
k(ave)	1.00	1.24	0.86	1.41	0.91
Prec1-Matrix					
I	32546	6826	14172	1332	2577
C(rat)	1.00	0.26	0.38	0.06	0.07
C(wt%)	56.42	14.68	21.18	3.25	4.06
Prec1					
I	13902	19539	8697	3745	1386
C(rat)	1.00	1.74	0.54	0.38	0.09
C(wt%)	26.54	46.29	14.31	10.06	2.41
AdjPrec1-Top1					
I	16531	2753	7328	478	1413
C(rat)	1.00	0.21	0.38	0.04	0.08
C(wt%)	58.34	12.06	22.29	2.37	4.53
AdjPrec1-Top2					
I	16222	2631	7285	399	1383
C(rat)	1.00	0.20	0.39	0.03	0.08
C(wt%)	58.57	11.79	22.68	2.03	4.54
AdjPrec1-Bottom1					
I	16633	2617	7381	654	1352
C(rat)	1.00	0.20	0.38	0.06	0.07
C(wt%)	58.35	11.39	22.32	3.23	4.31
AdjPrec1-Bottom2					
I	17181	4557	7688	838	1293
C(rat)	1.00	0.33	0.39	0.07	0.07
C(wt%)	53.78	17.70	20.75	3.69	3.68

To Page No.

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Project No.
TITLE STEM Results for As-Welded Alloy22 from DOE
Book No.

From Page No.

D. Specimen #2 – DOE As-Welded

a. EDS spot analysis

DOE As-Weld	Ni	Mo	Cr	W	Fe
Bulk(wt%)	59.58	13.85	20.35	2.63	2.85
Matrix1					
I	32671	5935	14476	1087	2332
C(rat)	1.00	0.23	0.34	0.04	0.05
C(wt%)	59.58	13.85	20.35	2.63	2.85
k1	1.00	1.28	0.77	1.33	0.67
Matrix2					
I	32509	5912	14111	1124	2317
C(rat)	1.00	0.23	0.34	0.04	0.05
C(wt%)	59.58	13.85	20.35	2.63	2.85
k2	1.00	1.28	0.79	1.28	0.67
Matrix3					
I	31273	5752	13623	985	2191
C(rat)	1.00	0.23	0.34	0.04	0.05
C(wt%)	59.58	13.85	20.35	2.63	2.85
k3	1.00	1.26	0.78	1.40	0.68
k(ave)	1.00	1.27	0.78	1.33	0.67
PPT1					
I	18167	19580	12269	2327	867
C(rat)	1.00	1.37	0.53	0.17	0.03
C(wt%)	31.98	43.91	16.86	5.47	1.03
Next to PPT1					
I	15734	3158	6197	490	786
C(rat)	1.00	0.26	0.31	0.04	0.03
C(wt%)	60.58	15.49	18.63	2.52	2.04
PPT2					
I	26023	20627	17402	1890	1017
C(rat)	1.00	1.01	0.52	0.10	0.03
C(wt%)	37.38	37.75	19.51	3.62	0.99
Next to PPT2					
I	46864	9978	20134	1343	1853
C(rat)	1.00	0.27	0.34	0.04	0.03
C(wt%)	59.38	16.11	19.91	2.27	1.58

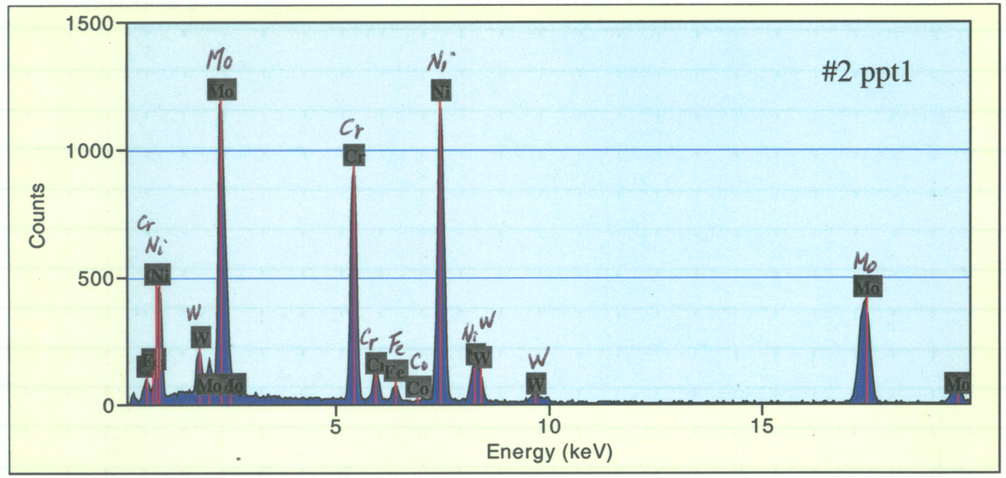
To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by [signature] | 5/7/02

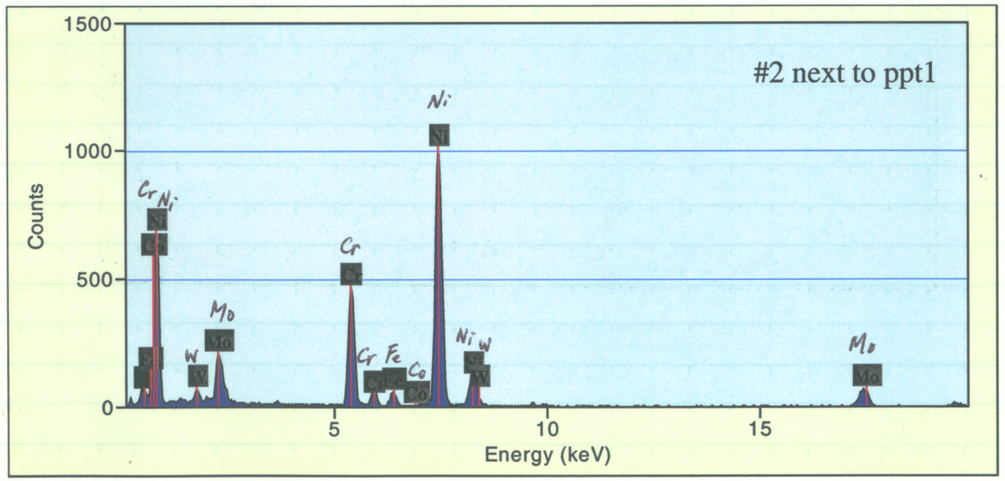
From Page No.

b. EDS spectra on the precipitate and adjacent to the precipitate

On Precipitate (ppt1)



Adjacent to Precipitate ppt 1



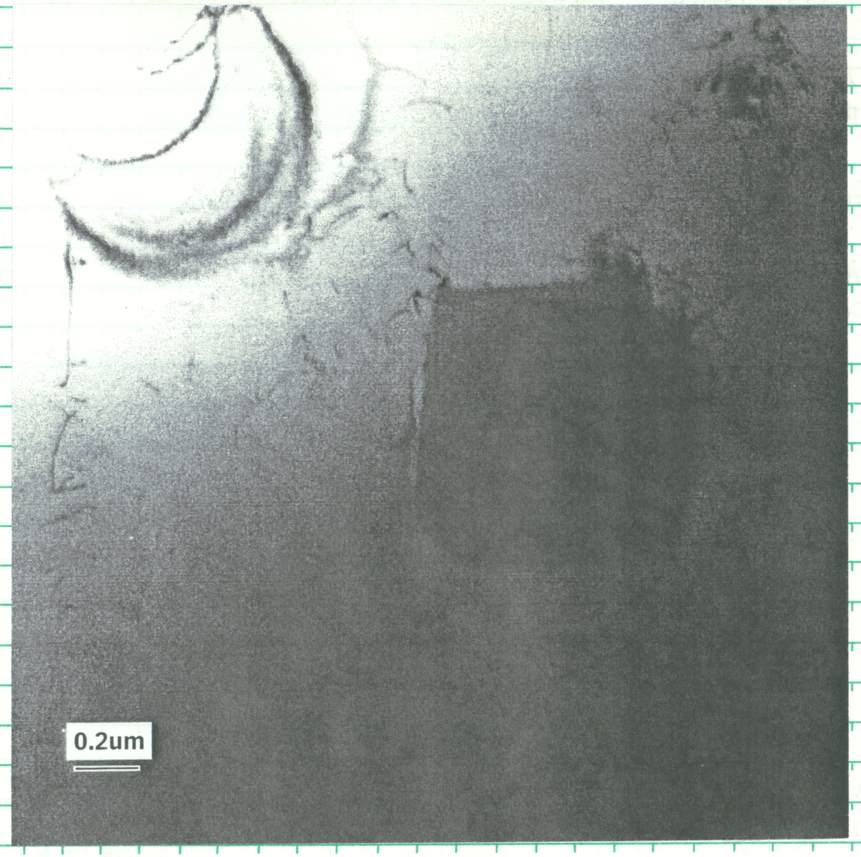
To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by 5/7/02

From Page No.

c. precipitates in the fusion zone

#2-gb1-01-7000 (Precipitate ppt 1 with composition on page 37)



To Page No.

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by 5/7/02

Project No.
Book No.
TITLE Intergranular Corrosion Tests of the 870°C Heat-Treated Alloy22 Specimens in 2.5% HCl

From Page No.

Intergranular Corrosion Tests

Specimens: 4 C-22 Heat 2277-8-3175 Specimens
#1 specimen: Mill-Annealed · Polished To 120 Grit Finish - 1 corner Ground
#2 specimen: Thermally Aged 870°C for 3 min · Polish To 120 Grit Finish · 2 corners Ground
#3 specimen: Thermally Aged 870°C for 5 min · Polished To 120 Grit Finish · 3 corners Ground
#4 specimen: Thermally Aged 870°C for 30 min · Polished To 120 Grit Finish - 4 corners Ground

See pg #12 for Drawing of Specimens for Identification Purposes (Corner Grounding)

Solution: All Tests will Be Run In A 2.5% HCl Solution for 24hrs. prepared As Follows

2.5% HCl
500 mls DI H_2O
28.20 mls HCl Lot # 002562 (37.5%)

#1 specimen - 1 corner Ground
Start wt: 8.76576g Santorious Genius
End wt: 8.72393g SN# 12809099 Cal 12/21/01

Dimensions:
.52 x .455 In²
.455 x .263 In²
.52 x .263 In²

#2 specimen - 2 Corner Ground
Start wt: 7.62431g
End wt: 7.58724g

Dimensions
.50 x .455 In²
.455 x .246 In²
.50 x .246 In²

#3 Specimen - 3 Corner Ground
Start wt: 6.73885g
End wt: 6.70559g

Dimensions
.495 x .39 In²
.39 x .240 In²
.495 x .240 In²

#4 Specimen - 4 Corner Ground
Start wt: 7.24106g
End wt: 7.20026g

Dimensions
.487 x .480 In²
.480 x .225 In²
.487 x .225 In²

All Dimension Measurements Taken with Fowler Calipers SN# 20-8C-1 Cal 12/3/01

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 5/16/02
Recorded by

TITLE
Project No.
Book No.

From Page No.

Test Results

Corrosion Rate Calculations for Tests in 2.5% HCl According to ASTM G28 Section 8

	Mill-Annealed	870 C/3 min	870 C/5 min	870 C/30 min
K	8.76E+04	8.76E+04	8.76E+04	8.76E+04
W(g)	0.04183	0.03707	0.03326	0.0408
A(cm^2)	6.36	5.97	5.23	5.82
T(h)	24	24	24	24
D(g/cm^3)	8.69	8.69	8.69	8.69
R(mm/y)	2.7618	2.6095	2.6703	2.9426
R(mpy)	108.7701	102.7702	105.1661	115.8916
Depth(um)	7.57	7.15	7.32	8.06
Test Surface Inspection	No Pit Shallow GB Attack	No Pit Shallow GB Attack	No Pit Shallow GB Attack	No Pit Deep GB Attack

R(mm/y)=(K*W)/(A*T*D)
R(mpy)=R(mm/y)*(3.45E6/8.76E4)
Depth(um)=(W*10000)/(A*D)

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 5/17/02
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Project No.
Book No.
TITLE STEM-EDS Chemical Composition Data Analysis

From Page No.

Statistical ananlysis of chemical composition

Precipitate Data(wt%)	Ni	Mo	Cr	W	Fe
	26.54	46.29	14.31	10.06	2.41
	22.78	45.54	20.68	8.93	2.07
	28.30	44.54	15.53	8.98	2.64
	28.49	40.97	20.72	7.55	2.27
Mean	26.53	44.33	17.81	8.88	2.34
Std Deviation	2.65	2.35	3.37	1.03	0.24
95% Conf Interval	3.00	2.66	3.81	1.17	0.27

Adjacent to ppt Data(wt%)	Ni	Mo	Cr	W	Fe
	58.34	12.06	22.29	2.37	4.53
	58.57	11.79	22.68	2.03	4.54
	58.35	11.39	22.32	3.23	4.31
Mean	58.42	11.75	22.43	2.54	4.46
Std Deviation	0.13	0.34	0.22	0.62	0.13
95% Conf Interval	0.15	0.38	0.25	0.70	0.15

To Page No.

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Project No.
Book No.
TITLE Acceptance Tests of Thermo-Calc and DICTRA Software Packages

From Page No.

1. Thermo-Calc for Windows (TCW) Version 2, Released in 2002.

Test Case: Prediction of equilibrium phases in UDIMAT 720, a high strength nickel base superalloy for turbine blade applications, as a function of temperature using Ni-DATA database.

Initial Equilibrium Conditions:

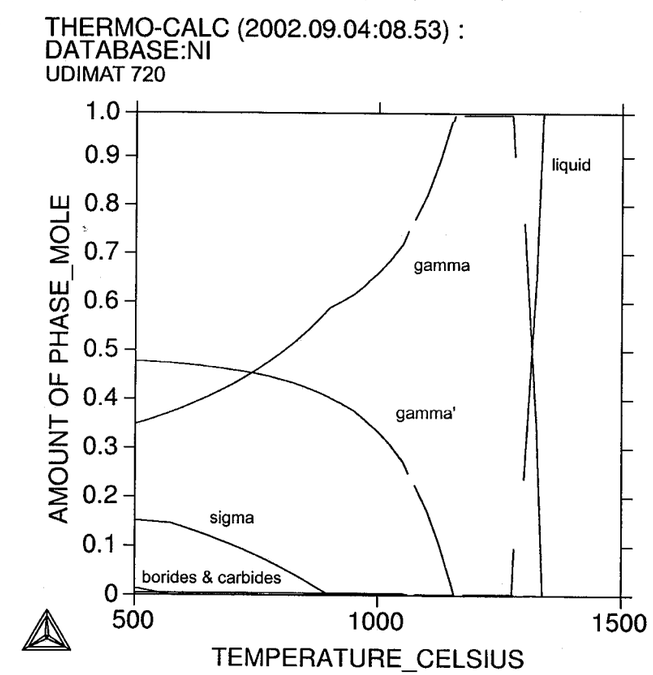
a. Material Ni-2.49 Al-0.032 B-0.02 C-14.8 Co-18.0 Cr-0.03 Fe-3.04 Mo-0.003 N-4.98 Ti-1.25 W (in wt.%)

b. Conditions temperature 1,000°C, pressure 100,000 Pa, system size 1 mole

Map/Step Definition:

Temperature as the independent variable, from 500 to 1500°C, 100 steps.

Results: The calculated equilibrium phases in mole fraction vs. temperature in °C for U720 are given below. The results are consistent with Figure 3 in Ni-DATA promotion sheet and Figure 11a reported by Reed, et al. in Metallurgical and Materials Transactions 30A, pp. 521-533, 1999. The TCW software and Ni-DATA database are acceptable.



To Page No.

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Ni-DATA, a multi-component database for Ni-based Superalloys

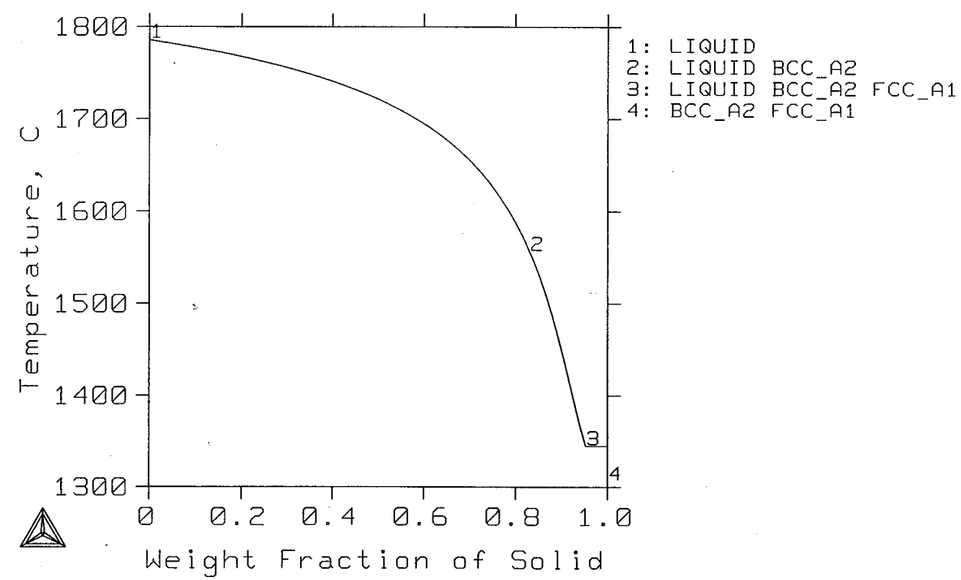
Information potentially subject to copyright protection was redacted from this location. The redacted material was from a user manual for the database listed above.

From Page No.

2. Thermo-Calc Classic (TCC) Version P, Released in 2002.
Test Case: Solidification simulation of Cr-10 mole% Ni alloy using the Scheil module and Ni-DATA (tcex15 in Thermo-Calc Examples)

Initial Conditions:
Phases included liquid, FCC, BCC
Calculated liquidus temperature 1,786 °C
Temperature range 1,300 – 1,800 °C, temperature step 5 °C

Results: The calculated diagram shows sections of the solification curve where different phases are stable. The results are consistent with that documented in Thermo-Calc Examples Manual.
The TCC software is acceptable.



To Page No.

From Page No.

3. DICTRA Version 21, Released in 2001.

Test Case: Carburization of Ni-25 wt% Cr alloy using Ni-DATA and MOB2 (Example d1 in DICTRA Examples)

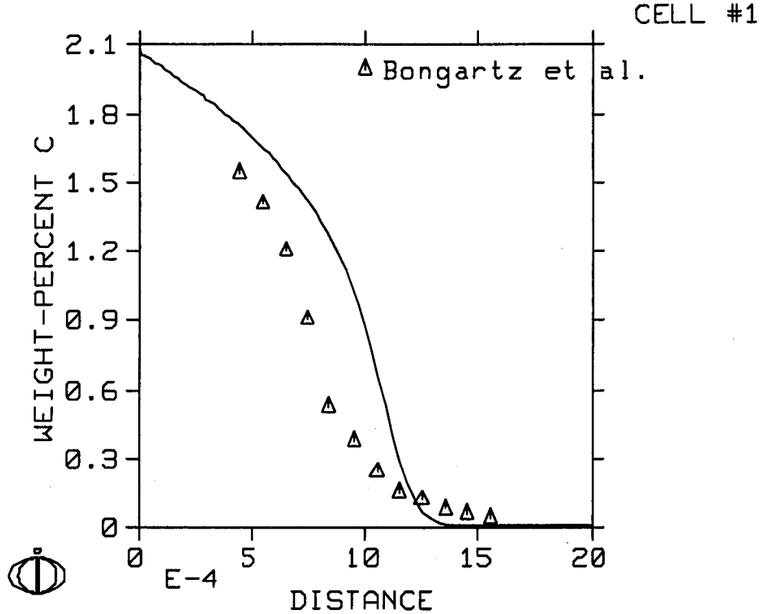
Initial conditions:

Phases included FCC, M_7C_3, M_3C_2, graphite

Simulation was conducted at 850 °C for 1,000 hr with the assumption of a constant surface C activity, $a_C = 1$ relative to graphite.

Results: The calculated diagrams, d1.1 and d1.2, are given below.

a. Figure d1.1 shows total C concentration in wt% vs. penetration distance in m after carburization for 1,000 h at 850 °C. Experimental data from Bongartz et al. in Corrosion Vol. 42, pp. 390-397, 1986 are included for comparison. The results are consistent with that documented in DICTRA Examples Manual and Figure 1a reported by Engström et al. in Metallurgical and Materials Transactions 25A, pp. 1127-1134, 1994. Note the difference, ~~probably~~ probably as a result of use of SSOL database in the DICTRA example.



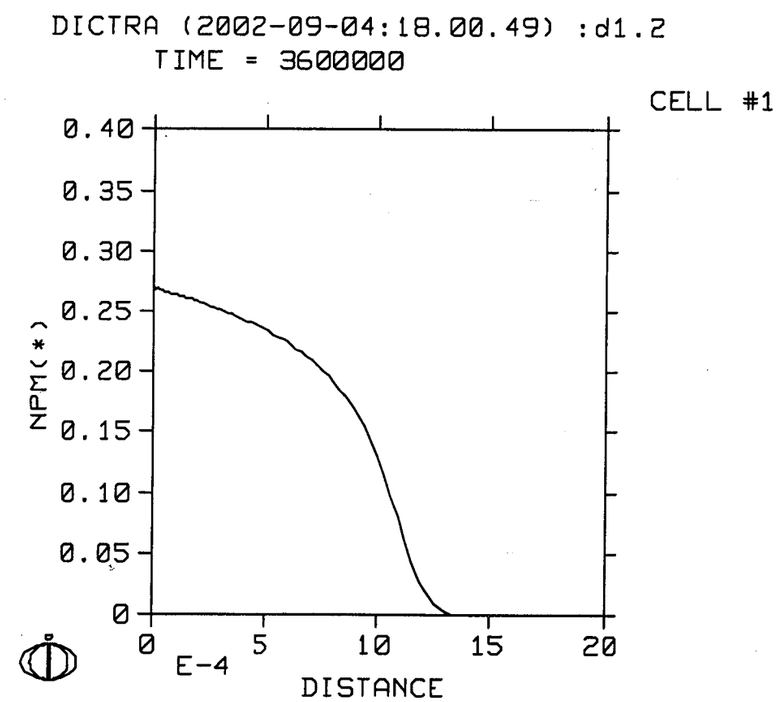
To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 9/5/02

Recorded by

From Page No.

Results (b): Figure d1.2 shows mole fraction of the precipitated carbides vs. penetration distance in m for the Ni-25 wt% Cr alloy. The results are consistent with that documented in DICTRA Examples Manual and Figure 1b reported by Engström et al. in Metallurgical and Materials Transactions 25A, pp. 1127-1134, 1994. Note the difference, probably as a result of use of SSOL database in the DICTRA example. The DICTRA software is acceptable. MOB2 database is also acceptable.



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To Page No.

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Project No.
Book No.

TITLE Sensitivity Analysis of P-Phase Solvus Temperature for Alloy 22

From Page No.

Obj: To calculate the P-Phase solvus temperatures (the upper stability temperature) by varying the specified composition limits for each element

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

Element	Content (wt%) Minimum	Maximum*	Baseline#
C	0.002+	0.015	0.01
Co	0.21+	2.5	2.0
Cr	20.0*	22.5	21.2
Fe	2.0*	6.0	4.0
Mo	12.5*	14.5	13.5
Si	0.022+	0.08	0.08
W	2.5*	3.5	3.0

* Composition requirements specified in ASTM standard B575
\+ Composition of C22 heats used in the DOE aging study (T.S.E. Summers, et al., Influence of thermal aging on the mechanical and corrosion properties of C-22 alloy welds, LLNL report No. UCRL-JC-137727, 2000)
\# Composition of Alloy 22 assumed in FY01 Supplemental Science and Performance Analyses, Volume 1, Fig. 7.3.2-2, TDR-MGR-MD-000007 Rev 00, 2001.

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 1/10/03
Recorded by

Project No.
Book No.

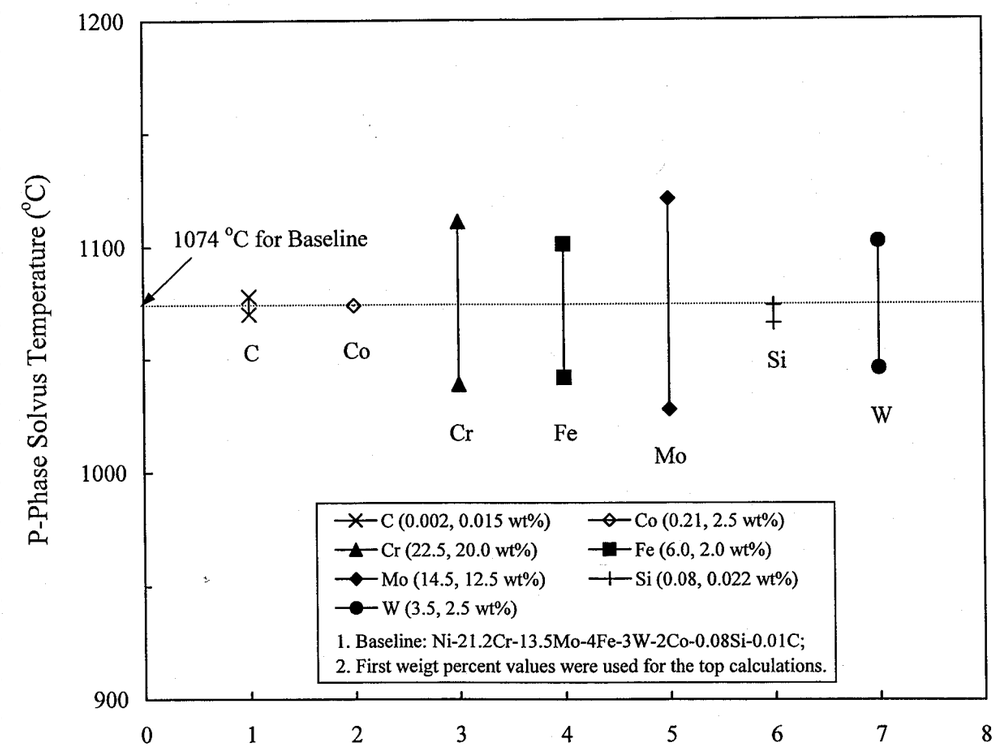
TITLE

From Page No.

Results:

Calculated P-Phase Solvus Temperatures Using the Composition Limits of Each Element

Solvus Temperature (°C)	Element						
	C	Co	Cr	Fe	Mo	Si	W
High	1,078	1,074	1,111	1,101	1,121	1,074	1,102
Low	1,070	1,074	1,039	1,042	1,028	1,066	1,046



To Page No.

Witnessed & Understood by me, | Date | Invented by | Date 1/13/03
Recorded by

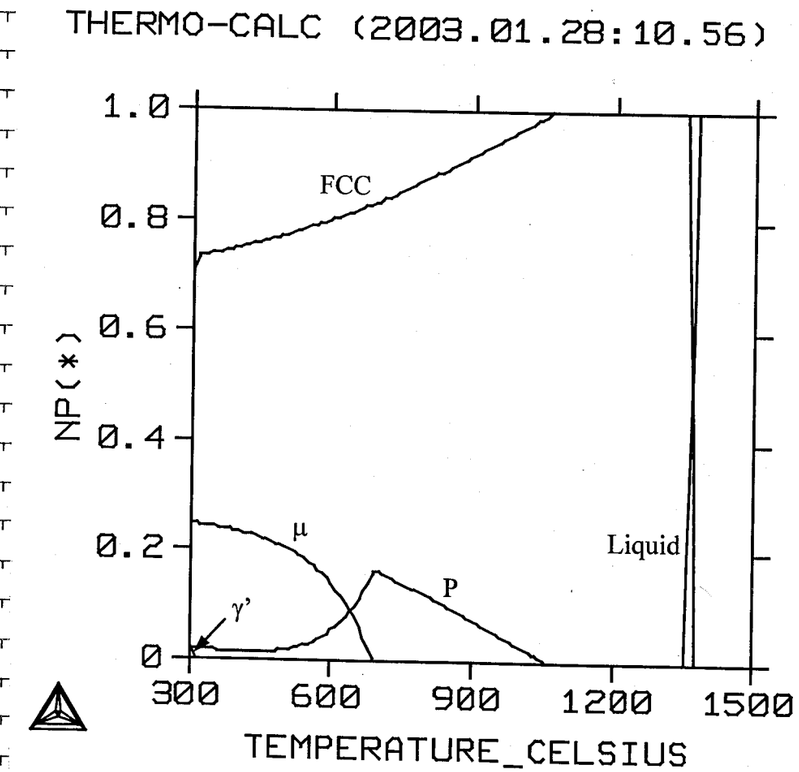
Project No.
Book No.
TITLE Phase Diagram and Phase Composition Calculations for Alloy 22

From Page No.

Objective: To calculate the phase diagram under equilibrium conditions and phase compositions at 870°C for one Alloy22 heat used at CNWRA

Software: THERMOCALC Version N
Database: Thermotech Ni-Data Version 5
Alloy composition (wt%): Ni – 21.4Cr – 13.6Mo – 3.08Fe – 3W – 0.09Co – 0.12Mn – 0.15V – 0.03Si – 0.004C

Results: The calculated diagrams in phase mole fraction vs. temperature are given below. The calculated phase contents and compositions at 870°C are provided in a table.



To Page No.

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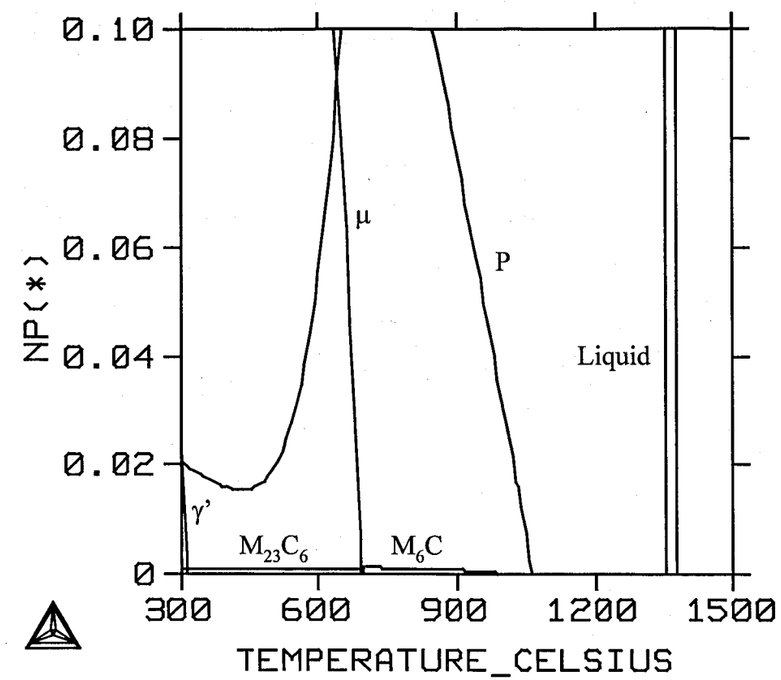
TITLE
Project No.
Book No.

From Page No.



Calculated Phase Compositions of Alloy 22 at 870 °C

Phase	Amount		Chemical Content (wt%)							
	wt%	mole%	Ni	Cr	Mo	W	Fe	Co	Si	C
γ	88.88	90.83	62.60	21.69	10.34	1.85	3.39	0.095	0.034	0.001
P	11.01	9.07	28.47	19.10	39.50	12.29	0.59	0.045	—	—
M_6C	0.11	0.10	25.08	14.47	52.12	4.80	0.93	0.016	0.001	2.58

To Page No.

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